Original Article

Influence of Different Finishing-Polishing Procedures and Thermocycle Aging on the Surface Roughness of Nano-Ceramic Hybrid CAD/CAM Material

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ABSTRACT

Aim: The aim of this study was to evaluate the effectiveness of different finishing-polishing (FP) procedures on reducing surface roughness of nanoceramic hybrid CAD/CAM material before and after thermocycle (TMC) aging. **Materials and Methods:** Nano-ceramic hybrid CAD/CAM specimens were subjected to 8 different (2-glaze and 6-mechanical) FP procedures as follows (n=20): 1. Optiglaze (OG) 2. Diamond glaze (DG), 3. Vita Enamic Polishing Set (VE), 4. VE+Gradia Diapolisher paste (VE-G) 5. VE+Super-Snap SuperBuff (VE-S) 6. Sof-Lex Disc kit (SL) 7. SL+Gradia Diapolisher paste (SLG) 8. SL+Super-Snap SuperBuff (SL-S). Surface roughness of each specimen was measured by using a contact profilometer. All specimens were artificially aged with TMC (5000 cycles, 5◦C/55◦C) and surface roughness measurements were repeated. One extra specimen from each group before and after TMC was examined with SEM. Surface roughness data were analyzed by repeated measures ANOVA, dependent t-test and Tukey test. **Results:** Significantly higher mean Ra values were found for groups OG and DG compared to other groups (P<0.05). Differences between glaze groups and differences between mechanical FP groups were not significant (P>0.05). Groups SL-S and DG demonstrated comparable results before and after TMC (P>0.05). SEM analysis revealed that surfaces of VE-S and SL-S groups were smooth and free of scratches. **Conclusions:** Mechanical FP procedures were more effective in reducing surface roughness of nanoceramic hybrid CAD/CAM material than glaze applications. Sof-Lex kit followed by Super-Snap SuperBuff disc application can be recommended as the mechanical FP procedure of choice considering that this method provided smooth surfaces that were maintained after TMC.

Keywords: *Glaze, mechanical finishing-polishing; nano-ceramic hybrid cad/cam material, surface roughness, thermocycle aging*

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Introduction

Computer-aided design and computer-aided manufacturing (CAD/CAM) is favored by clinicians due to several advantages including time efficiency in each step of the treatment from digital impression to production, simplified clinical and laboratory procedures, and better communication with both patient and dental technician.[1,2] The popularity of CAD/CAM technology has led to advances in material science as well.[3] Various types of blocks with different advantages and disadvantages have been introduced for use in CAD/CAM systems. Most recently, hybrid blocks that combine the advantages of ceramics and resin polymers have been developed. Favorable features of these materials included elasticity close to dentin,

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reduced abrasiveness to the antagonist dentition, good marginal adaptation, and ease of repair.[3-5] Cerasmart (GC Corporation, Tokyo, Japan) is a flexible nano-ceramic composite resin hybrid block that consists of a polymeric matrix reinforced by homogeneous and evenly distributed nano-ceramic fillers.[6] As this material does not require any crystallization or glaze firing after manufacturing, nano-ceramic hybrid block is a valid choice for chairside use in which the entire fabrication and finishing process is completed in the dental office within a single appointment.[5,7]

The milling process of CAD/CAM restorations results in a rough surface. Therefore, the surface should undergo a finishing-polishing (FP) process before the delivery of restoration.[5] Otherwise, the restoration may be compromised esthetically, and increased microbial retention may cause gingival inflammation and secondary caries.[5,8,9] To provide a smooth restoration surface in chairside use, various mechanical FP applications for hybrid CAD/CAM restorations are available including various types of two- or three-step rubber polishing sets with different grits, polishing pastes with diamond filler, and multi-step polishing strips.[9,10] Also, light-cured surface varnishes with methyl methacrylate (MMA) content have been introduced for resin containing CAD/CAM restorations as these materials cannot be glazed via firing due to resin content.[11] Several studies have compared the effectiveness of these FP applications and techniques in different combinations.[3,5,9,10] Yet, no consensus has been reached in the literature which is the best option for reducing the surface roughness of hybrid material. Also, the effect of aging is lacking considering that the long-term effectiveness of surface treatments was reported to change with the thermocycle process.[12]

According to these considerations, the aim of this study was to evaluate the effect of different FP procedures and thermocycle (TMC) aging on the surface roughness of nano-ceramic hybrid CAD/CAM material. The null hypotheses tested were as follows: 1. Surface roughness of nano-ceramic hybrid materials will not differ before and after FP procedures. 2. Surface roughness of nano-ceramic hybrid materials subjected to different FP procedures will not differ before and after TMC aging.

Methodology

This study investigated the effect of eight different FP procedures and TMC aging on the surface roughness of a nano-ceramic hybrid CAD/CAM material. The minimum specimen size required to be included in this study was calculated at 0.30 effect size, 90% power, and $\alpha = 0.05$ error level in terms of two time points (before–after TMC) as n = 20 per group.[3] Two more specimens from each group were prepared and used for scanning electron microscope (SEM) analysis before and after TMC (N = 176). The materials used in this study are presented in Table 1.

Nano-ceramic hybrid CAD/CAM blocks (Cerasmart, GC Corporation, Tokyo, Japan) were wet-sliced by a diamond saw (Micracut 201, Metkon, Turkey) to obtain 176 rectangular-shaped plates with 2 mm thickness. Silicon carbide papers with 600 and 800 grit were used to equalize the surface of all specimens. Then, specimens were randomly divided into eight groups according to the FP procedures applied as follows: 1. Optiglaze (OG), 2. Diamond glaze TD (DG), 3. Vita Enamic polishing set (VE), 4. VE + Gradia Diapolisher paste (VE-G), 5. VE + Super-Snap SuperBuff (VE-S), 6. Sof-Lex Disk

Table 1: Materials used, their compositions, and the manufacturers

Material	Type	Manufacturer	Composition
Cerasmart	Nano-ceramic hybrid CAD/CAM block	GC Corporation, Tokyo, Japan	Inorganic portion (71 wt%): silica (20 nm SiO_2) and barium glass (300 nm) nanoparticles polymers (29 wt%): *bis-MEPP, UDMA, DMA
Optiglaze	Light-cured glaze	GC Corporation, Tokyo, Japan	Methyl methacrylate (25–50%), silicon dioxide (5–10%), titanium dioxide (1–5%), photo initiator (1–5%), diphenyl (2,4,6-trimethylbenzoyl) phosphine oxide (1–5%)
Diamond glaze TD	Light-cured glaze	On-dent, Izmir, Turkey	Methyl methacrylate (30–60%), multifunctional acrylate (20–40%), silicium dioxide (10–15%), diphenylphosphine oxide (2–5%)
Vita Enamic polishing set technical	Rubber polishing kit	Vita Zahnfabrik Bad Sackingen, Germany	Two-step silicon carbide polishing drills with pre-polishing (pink) and high-gloss (grey) polishers
Gradia Diapolisher paste	Diamond polishing paste	GC Corporation, Tokyo, Japan	Pat varnish with diamond particles (~5 µm)
Super-Snap SuperBuff Set	Paste impregnated felt polishing buff disk	Shofu Inc, Kyoto, Japan	Aluminum oxide-coated abrasive buff disk from woolen cloth with mounting core of *PVC
Sof-Lex XT Finishing and Polishing System	Flexible extra-thin contouring and polishing disks	3M ESPE St Paul, MN, USA	Aluminum oxide-coated disks with four different abrasive grades: Brown (17.01 µm); orange (7.01 µm); light orange (5.72 µm); yellow (1.68 µm)

*UDMA : urethane dimethacrylate, DMA : dodesil dimetakrilat, BisMEPP : 2.2- Bis (4-Metakriloksi-polietoksifenil) propan, PVC : polyvinyl chloride kit (SL), 7. SL + Gradia Diapolisher paste (SL-G), and 8. SL + Super-Snap SuperBuff (SL-S). Test groups and details of application protocols are described in Table 2. All materials were applied following the manufacturers' instructions by the same calibrated operator. Afterward, all specimens were ultrasonically cleaned in distilled water for 10 min.[13]

Surface roughness (Ra) of each specimen was measured by using a contact profilometer (Perthometer; Mahr Gmbh, Ingolstadt, Germany). For each specimen, three measurements in micrometers (µm) were made in different directions, and the mean of these three measurements was used for statistical analysis.[13] All specimens were artificially aged with TMC (THE-1100, SD Mechatronik, Feldkirchen-Westerham, Germany) in distilled water (5000 cycles, 5°C/55°C thermal application, and dwell time of 30 s).[14] Then, surface roughness measurements were repeated with the protocol described before TMC. Two extra specimens from each group were coated with electrically conducting metal and examined under ×1000 magnification with SEM to visualize the surface irregularities (EVO 40 series, Carl Zeiss AG, Oberkochen, Germany). The SEM images were obtained from two separate specimens before and after TMC from each group that were subjected to FP procedures.[15]

The data were tested for normality with Kolmogorov–Smirnov test. Statistical analyses were done using repeated measures ANOVA, dependent t-test for paired specimens (intragroup comparisons regarding TMC), and post hoc Tukey test (intergroup comparisons regarding different FP procedures) ($\alpha = 0.05$). All statistical analyses were performed by R v. 3.5.3 (Microsoft Corporation, Redmond, WA, USA).[16]

Results

Repeated measures ANOVA results are presented in Table 3 which showed a statistical significance for FP procedures and TMC ($p < 0.001$), yet not for the interaction between them ($p = 0.077$). Therefore, intergroup comparisons were done irrespective of the TMC variable.

The descriptive statistics for test groups regarding TMC are listed in Table 4. Minimum and maximum Ra values ranged between 0.06 and 3.02 µm, respectively. Mean

Table 2: Application procedures and abbreviations of study groups

Polishing System	Group abbreviations	Application procedure
Optiglaze	OG	-Air blasting with 50 µm aluminum oxide (Al_2O_3) particles with a pressure of 2.5 bars for 10 sec. -Ultrasonic cleaning in distilled water for 5 min. Silanization with G-Multi Primer (GC Corporation; Tokyo, Japan) for 30 seconds, and air drying. -Optiglaze Clear application and LED curing with 430 nm wavelength for 40 sec (Bluephase 20i, Ivoclar Vivadent; Schaan, Liechtenstein), according to the manufacturer's recommendations
Diamond glaze TD	DG	Same application protocol with OG group was applied.
Vita Enamic polishing set	VE	-Pre-polishing (pink) drill was applied with a low-speed hand-piece (10.000 rpm) for 60 sec -High-gloss polishing drill was applied with a low-speed hand-piece (8.000 rpm) for 60 sec
Vita Enamic polishing set + Gradia Diapolisher paste	VE-G	-The specimens were treated as in the group VE. -Gradia Diapolisher paste was applied with a chamois buffing wheel using a low-speed hand-piece (10,000 rpm, for 20 sec)
Vita Enamic polishing set + Super-Snap SuperBuff	VE-S	-The specimens were treated as in the group VE. -The surfaces of specimens were wet, and SuperBuff disk was moisten with water for 30 sec. -Disks were applied with fleeting strokes using a low-speed hand-piece with 10.000 rpm for 20 sec. -A new disk was used for each specimen.
Sof-Lex Disk kit	SL	-Four different grades of abrasive aluminum oxide-coated disks were applied in decreasing order of grits. -The thick and medium grit disks were used at 30,000 rpm, and the thin and super fine grit disks were used at 10,000 rpm. -Each disk was applied in the same direction for 15–20 seconds. -Disks were disposed in every single use.
Sof-Lex Disk kit + Gradia Diapolisher paste with chamois wheel	SL-G	-The specimens were treated as in the group SL. -Gradia Diapolisher paste was applied as described in Group VE-G.
Sof-Lex Disk kit + Super-Snap SuperBuff	SL-S	-The specimens were treated as in the group SL. -Gradia Diapolisher paste was applied as described in Group VE-S.

Table 3: Repeated measures ANOVA results

Effect	Sum of squares	df	Mean squares	*F*	*P*
FP procedure	37,073	7	5,296	64,477	<0,001
TMC	0,441	1	0,441	22,289	<0,001
FP procedure* TMC	0,259	7	0,037	1,874	0,077

*FP: Finishing-polishing, TMC: Thermocycle

Table 4: Descriptive statistics of test groups (Ra: surface roughness, SD: standard deviation)

*FP procedure	*TMC	Ra (µm) Mean±SD	Minimum	Maximum	*P*
*OG	Before	1.21±0.43	0.39	1.92	0.015
	After	1.3±0.41	0.73	2.26	
*DG	Before	1.27±0.7	0.37	3.02	0.108
	After	1.39±0.67	0.39	2.86	
*VE	Before	0.21±0.04	0.14	0.31	0.004
	After	0.3±0.14	0.14	0.61	
*VE-G	Before	0.3±0.12	0.13	0.61	0.046
	After	0.32±0.11	0.15	0.6	
*VE-S	Before	0.17±0.07	0.06	0.34	0.034
	After	0.2±0.09	0.06	0.43	
*SL	Before	0.19±0.08	0.1	0.44	0.003
	After	0.23±0.11	0.12	0.55	
*SL-G	Before	0.13±0.02	0.09	0.18	0.031
	After	0.15±0.04	0.1	0.26	
*SL-S	Before	0.18±0.04	0.09	0.26	0.492
	After	0.19±0.04	0.1	0.3	

*FP: Finishing-polishing, TMC: Thermocycle, OG: Optiglaze, DG: Diamond glaze TD, VE: Vita Enamic polishing set, VE-G: VE + Gradia Diapolisher paste, VE-S: VE + Super-Snap SuperBuff, SL: Sof-Lex Disk kit, SL-G: SL + Gradia Diapolisher paste, SL-S: SL + Super-Snap SuperBuff

Ra values ranged between 1.39 ± 0.67 (Groups DG-after TMC) and 0.13 ± 0.02 (Group SL-G-before TMC). Differences between mean Ra values of all groups before and after TMC were significant ($p < 0.05$) except groups DG and SL-S ($p > 0.05$).

The differences between mean surface roughness values for the groups are presented in Figure 1. Groups OG and DG showed comparable surface roughness values ($p > 0.05$) which were significantly higher than the other groups ($p < 0.05$). Also, differences between VE, VE-G, VE-S, SL, SL-G, and SL-S were not statistically significant ($p > 0.05$).

Considering SEM analysis, prominent surface irregularities were detected for glaze groups, especially for DG. Lines across the surface transversely and longitudinally were observed for groups VE, VE-G, SL, and SL-G. However, these lines were not seen in groups VE-S and SL-S. A slight degradation on the surface was seen for all groups after TMC; however, for group SL-S, this finding was not that apparent.

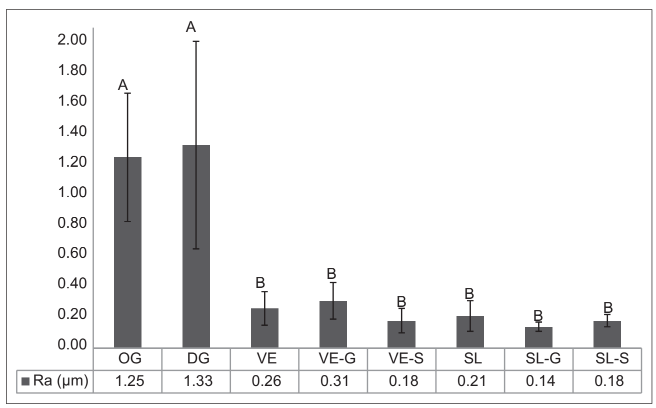

Figure 1: Differences between FP procedures regarding surface roughness. Same letters indicate no statistical difference between groups. * FP: Finishing-polishing, OG: Optiglaze, DG: Diamond glaze TD, VE: Vita Enamic polishing set, VE-G: VE + Gradia Diapolisher paste, VE-S: VE + Super-Snap SuperBuff, SL: Sof-Lex Disk kit, SL-G: SL + Gradia Diapolisher paste, SL-S: SL + Super-Snap SuperBuff

Discussion

This study investigated the effect of different FP procedures and thermocycle aging on the surface roughness of a nano-ceramic hybrid CAD/CAM material. Both null hypotheses were rejected because significant differences were found before–after TMC aging for a particular FP procedure and between different FP procedures.

The surface roughness of nano-ceramic CAD/CAM blocks may vary depending on the composition, particle type, and grain size of the materials used in

FP processes.[17] Two light-cured resin containing glaze materials and six different mechanical polishing methods were applied as the FP procedures in the present study. The surface roughness of glaze applied groups was found to be higher than the mechanical polishing methods. Unlike other mechanical polishing systems, light-cured glaze application requires a sandblasting process which results in increased surface roughness. In fact, increased surface roughness is specially recommended by the manufacturers before this type of glaze application to enhance adhesion and increase surface energy for the sake of durability of the glaze material.[10,11] However, this also appears to increase the final surface roughness of the restoration. Similar results regarding higher surface roughness of glazed specimens were reported in the literature for ceramic materials.[18-20] However, these findings contradict with the results of the study by Kara *et al*.[11] in which similar Ra values were reported for the groups treated with Optiglaze and mechanical FP methods. Different findings can be attributed to methodological variations considering that the latter study used surface topography techniques to evaluate surface roughness. To decrease the surface roughness after glaze application, air drying may help the material to evenly spread through the surface and into the pits that generated by sandblasting. In our study, air drying was not utilized because the instructions did not include air drying after glaze application and the recommended protocol was followed.[10,11] Further studies are needed to evaluate the effect of air drying application on the surface roughness of glazed surfaces.

As the surface roughness increases, the adhesion of microorganisms increases and the biocompatibility tends to decrease.[21] Restorations should have smooth surfaces to minimize wear on the antagonist dentition, to prevent discoloration, and not to cause a decrease in fracture-flexural strength due to superficial cracks that increases brittleness of restorative materials.[1,5,11,22] Previous studies reported that an increase in surface roughness (Ra) above 0.2 µm may substantially increase plaque accumulation which leads to caries and periodontal inflammation.[22,23] In the present study, glaze groups (OG and DG) showed considerably higher Ra values both before and after TMC than the threshold value specified in the literature. On the other hand, VE-G group also showed higher surface roughness, but not as much as the glaze groups. Also, surface roughness of hybrid nano-ceramic specimens treated with only Vita Enamic polishing drills increased to 0.3 µm after TMC. Surface roughness values of other groups were below the threshold value regardless of the TMC application. According to these considerations, a higher plaque accumulation can be anticipated when the hybrid nano-ceramic restorations are subjected to light-cured glaze materials compared to mechanical FP techniques. Also, it can be recommended that Vita Enamic polishing set is applied in combination with Super-Snap SuperBuff Set to ensure lower surface roughness.

In the present study, mechanical polishing systems were used in different combinations. Vita Enamic polishing set contains two-step silicon carbide polishing drills and Sof-Lex polishing system contains four-step aluminum oxide-coated disks with different abrasive grades. Those systems were used with or without additional polishing paste or paste impregnated disk. There was no difference between mean Ra values of these mechanical FP procedures. On the contrary, Flury *et al*.[24] found Sof-Lex polishing system more effective than Vita Enamic polishing set. However, in line with our finding, Kemaloglu *et al*.[25] reported that two-step systems can be as effective as multi-step systems in reducing the surface roughness. The "step number" factor of the polishing system should not be directly associated with surface roughness.[26] On the other hand, increased polishing time in multi-step systems was reported as a disadvantage in clinical practice.[27]

Thermomechanical stresses and chemical alterations in the oral cavity may lead to surface degradation of materials which is described by the aging of the material.[28] Thermal cycling is a widely used application to simulate artificial aging in *in vitro* studies.[3,10,11] In the present study, all specimens were subjected to 5000 cycles of thermal cycling which corresponds to six months of oral service to evaluate the maintenance of the FP procedures tested.[14,29] After artificial aging, significant increases in surface roughness were detected for all groups except the specimens subjected to Diamond glaze and Sof-Lex polishing set in combination with Super-Snap SuperBuff disk. Maintenance of the surface smoothness is as important as the immediate effectiveness of the FP procedure considering that the restoration would serve for a long time intraorally. Considering that Sof-Lex polishing set in combination with Super-Snap SuperBuff disk demonstrated lower Ra values than the threshold value of 0.2 µm that maintained after artificial aging, this technique may be recommended as the FP procedure of choice for clinical use.

Surface roughness is an important parameter used to describe the effectiveness of surface treatments. Various quantitative and qualitative methods were used to evaluate the surface roughness of restorative materials including optical microscopy and SEM, surface profile analysis, three-dimensional surface topography measurement, and profilometry.[10,11,28] Roughness parameters Ra and Rz are frequently preferred in studies since they can be evaluated quantitatively.[14,15] Quantitative values were also evaluated with a contact profilometer in this study.[18] Although profilometric analysis is a frequently used method, it has a limitation that fails to give precise measurements of surface roughness when excessively protruded or undermined regions are present.[3,5,18] In addition, another disadvantage of the linear contact surface profilometer is that the surface roughness can only be calculated in a specific area.[18] Therefore, results of linear profilometric analysis should be interpreted considering the information from the SEM analysis.

SEM image examination [Figure 2] supports our surface roughness findings as irregularities were apparent for glaze applications compared to mechanical FP procedures. The poor surface quality seen in SEM image of the group DG compared to OG can be attributed to the different chemical compositions of these materials. On the other hand, SEM analysis revealed different surface appearance for mechanical FP procedures even though the differences between Ra results were insignificant (groups VE, VE-G, VE-S, SL, SL-G, SL-S). Therefore, SEM evaluation might give an overall opinion for the groups subjected to mechanical FP procedures. In this regard, groups subjected to Super-Snap SuperBuff disks (VE-S and SL-S) were noticeably smoother than the other groups which demonstrated transverse and longitudinal lines caused by polishing disks or drills. As the manufacturer stated, Super-Snap SuperBuff disks prevent scratches while application because they do not contain a steel mandrel. However, the diamond particle containing polishing paste (Gradia Diapolisher) was applied by a chamois buffing wheel with a metal part in the middle which may have caused the scratches on the surface while application. This might also explain relatively higher surface roughness value obtained for the VE-G group, which was insignificant statistically. According to SEM images presented, polishing hybrid nano-ceramic CAD/CAM restorations with a disk set followed by finishing with aluminum oxide-impregnated woolen disks (Super-Snap SuperBuff disks) seems to result in a smooth surface.

The limitations of this study include the use of a single type of CAD/CAM block, aging the specimens using distilled water instead of artificial saliva and preparing flat specimen surfaces by a microcut device instead of a milling device which is used in clinical practice. Further *in vivo* studies should be conducted evaluating FP procedures' effectiveness on the surface roughness of various types of CAD/CAM blocks.

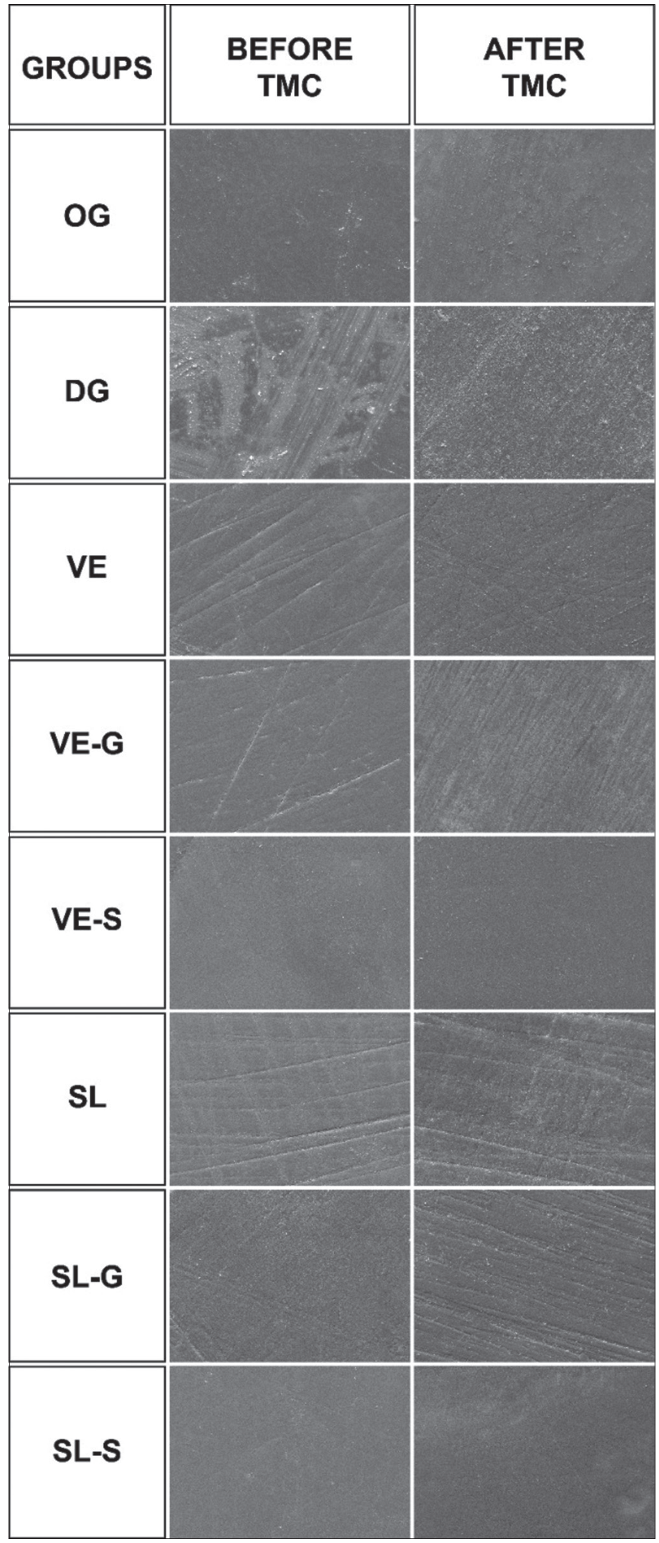

Figure 2: SEM images (×1000) of nano-ceramic hybrid material subjected to different FP procedures before and after TMC. *FP: Finishing-polishing, TMC: Thermocycle, OG: Optiglaze, DG: Diamond glaze TD, VE: Vita Enamic polishing set, VE-G: VE + Gradia Diapolisher paste, VE-S: VE + Super-Snap SuperBuff, SL: Sof-Lex Disc kit, SL-G: SL + Gradia Diapolisher paste, SL-S: SL + Super-Snap SuperBuff

Conclusions

Within the limitations of this study, it can be concluded that mechanical FP procedures were more effective in reducing surface roughness of hybrid nano-ceramic CAD/CAM material than glaze methods. Also, as the mechanical FP procedure of choice, Sof-Lex polishing set in combination with Super-Snap SuperBuff disk can be recommended considering the following results: 1) This method provided surface roughness values below the threshold value of 0.2 μm both before and after TMC. 2) Considering the maintenance of smoothness, surface roughness did not increase after TMC. 3) SEM images of this method presented the smoothest, scratch-free surfaces regardless of the TMC application. In addition, Super-Snap SuperBuff disks can be recommended as the final fine-polishing application after mechanical finishing applied on hybrid nano-ceramic material as both SEM images of the groups involved this treatment revealed smooth surfaces.

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Conflicts of interest

There are no conflicts of interest.

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