Clinical and Experimental Health Sciences

Effect of Thermomechanical Aging on the Surface Roughness and Color Stability of Novel CAD-CAM Materials: An In-Vitro Study

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ABSTRACT

Objective: This in-vitro study aimed to evaluate the surface roughness (R_a) and color stability of novel monolithic CAD-CAM materials after thermomechanical aging.

Methods: Forty specimens were obtained from 4 different materials (a resilient ceramic (RC), ceramic-reinforced PEEK (PE), lithium disilicate glass ceramic (EX), and a resin-based composites (CO) (n=10). Initial R_a and color coordinates were recorded. All specimens were than subjected to thermomechanical aging. R_a and color coordinate measurements were repeated. CIEDE2000 formula was used to calculate the color changes (ΔE_{00}). Kruskal-Wallis and Dunn tests were used to analyze data, while the effect of aging on R_a was analyzed with Wilcoxon test (α=.05).

Results: Regardless of aging, PE showed higher R_a than EX (p< .001), whereas the differences between PE and the other materials were nonsignificant (p≥ .202). Thermomechanical aging led to higher R_a for all materials tested (p= .005). The greatest ΔE_{00} was observed in PE (p≤ .002), while the difference among the other materials was nonsignificant (p> .05). R_a of ceramic-reinforced PEEK was above clinical threshold, regardless of thermomechanical aging.

Conclusion: Thermomechanical aging caused a color change that was perceptible for all the materials tested, while it was also unacceptable for ceramic-reinforced PEEK.

Keywords: CAD-CAM, dental materials, roughness, color

1. INTRODUCTION

Computer aided design-computer aided manufacturing (CAD-CAM) technologies have facilitated more natural and standardized restorations with a reduced cost and fabrication time (1-3). Parallel to this technology, various monolithic restorative materials have become viable (4). The increasing trend towards monolithic restorations with reduced chipping and easier fabrication has led to material diversity (5). Resilient ceramics, which contain both ceramic and resin have been introduced as an alternative to ceramics. These materials stand out with their favorable Young's moduli, relatively low fracture tendencies, and ease of processing (6). CAD-CAM composite resins offer better mechanical and esthetic properties than those fabricated by conventional methods due to increased polymerization (7,8).

Among the available materials that can be processed by CAD-CAM technologies, polyetheretherketone (PEEK) is a popular high-performance polymer due to its dentin-like elastic modulus, light weight, ease of machinability, and favorable chemical and mechanical properties (9-13). A more recent iteration of this materials is the ceramic-reinforced PEEK, which has improved esthetic and thereby used for the fabrication of frameworks, crowns, abutments, and inlays (3-14).

Mechanical and optical properties of a restorative material are fundamental for its clinical success (1,15-19). Considering the dynamic intraoral environment involving various chemical and mechanical factors that may cause substance loss (20), a restorative material should resist wear as increased surface roughness (R_a) is directly proportional to plaque accumulation, bacterial adhesion, and periodontal problems (21-24). Moreover, previous researches have shown the adverse effects of R_a on the esthetic and color stability of CAD-CAM materials (16,25). The clinical threshold of R_a has been reported as 0.2 µm (26).

Optical characterizations are an important parameter that affects the longevity, esthetic appearance, and ultimately patient acceptability of a prosthetic material (16). Color stability can be affected by many factors such as water absorption, aging, patient's cleaning habits, R_a, and material content (25). Color alteration may be associated with aging owing to the degradation of the polymer matrix or unreacted monomers, depending on the material type (27).

Previous studies have focused on the R_a and color stability of restorative materials after different aging procedures (1,15,17,27-37), given the constant exposure to the dynamic loads and temperature changes in the oral environment (1,31). However, there is limited information on the effect of thermomechanical aging on the R_a (38) and color stability (34) of PEEK after thermomechanical aging. Thus, the present study aimed to compare the R_a of ceramic-reinforced PEEK with 3 different CAD-CAM materials before and after thermomechanical aging as well as evaluate materials' color change. The null hypotheses were that i) the R_a values would not be affected by the material type and thermomechanical aging, and ii) the color change would not be affected by the material type.

2. METHODS

Table 1 gives detailed information about the materials tested. Forty square-shaped specimens were wet-sliced (Microcut 201; Metkon Instruments Inc.) by using 4 monolithic CAD-CAM materials (BioHPP; Bredent, IPS e.max CAD; Ivoclar Vivadent, Cerasmart; GC Corporation, Brilliant Crios; Coltène) (n=10). All specimens were then wet-ground (#400 and 600) and polished (#1000) with silicone carbide abrasive papers for the final dimensions of 12x12mm and a thickness of 1.2 mm. A digital caliper (Absolute Digimatic; Mitutoyo) was used to control the dimensions of all specimens. EX specimens were then crystallized in a porcelain furnace (Programat P310; Ivoclar Vivadent AG). No additional glazing or polishing was performed (3,39,40).

Table 1. *List of tested materials*

Material	Abbreviation	Chemical Composition	Manufacturer
BioHPP	PE	Polyetheretherketone, ceramic filler (20%)	Bredent
IPS e.max CAD	EX	57%-80% SiO_2, 11%-19% Li_2O,0%-13% K_2O, and other oxides	Ivoclar Vivadent
Cerasmart	RC	(71%) Silica, barium glass, Bis-MEPP, UDMA, DMA	GC Corporation
Brilliant Crios	CO	(70%) Resin matrix cross-linked with methacrylates, amorphous silica, barium glass	Coltène AG

PE: Polyetheretherketone, EX: Glass ceramic, RC: Resilient ceramic, CO: Resin-based composite

A single researcher (A.T.) measured the initial color coordinates (L*, a*, and b* values) of the prepared specimens on a gray background 3 times by using a digital spectrophotometer (VITA EasyShade V; Vita Zahnfabrik) and the mean values were recorded. The spectrophotometer was calibrated before each reading. Standardization was achieved by positioning the probe tip perpendicular to the surfaces during the measurements, and the measurements were not affected by the ambient light (41). After color measurements, a contact profilometer (Marsurf PS1; Mahr) was utilized for the initial R_a values of the specimens. Three measurements were performed on each specimen (stylus tip radius: 5 µm, stylus driving speed: 0.5 mm/s, traversing length: 1.75 mm, and five cut-off length: 0.25 mm) (42) and the mean values were calculated.

Sound maxillary premolar teeth, which were stored in 0.5% chloramine solution at room temperature for a week and afterwards kept in distilled water at 5°C for 14 days (8) were used as enamel antagonists (Biruni University Ethics Committee, Decision No: 2020/37-04). For thermomechanical aging, the specimens were placed in silicone molds in the chamber of the biaxial chewing simulator (MOD CS; MOD Dental) and fixed by position screws. Prior to thermomechanical aging, buccal cusps of the antagonist teeth were adjusted to a spherical shape by using a diamond instrument (801-314-018-C; Coltene/Whaledent AG). Simultaneous mechanical (240.000 cycles, 49 N, 1.2 Hz, and 0.7 mm lateral movement) and thermal aging (5-55°C, 60 s dwell time) were applied in distilled water (3). The specimens were then cleaned in an ultrasonic tester, and measurements were repeated. Color differences (ΔE_{00}) before and after thermomechanical aging were calculated by using the CIEDE2000 color formula and the parametric factors (KL, KC, and KH) were considered as 1 (1,43):

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

Based on the results of a previous study (3), the number of samples was determined (effect size f= 0.71, 1-β= 0.95, and α= 0.5) with a priori power analysis. Normality of the data were analyzed by using Shapiro-Wilk tests. Due to the non-normal distribution of the R_a and ΔE_{00} data, Kruskal-Wallis and Dunn's tests were performed. The differences within each material's R_a values before and after thermomechanical aging were further resolved with Wilcoxon tests (SPSS v23; IBM) (α=.05).

3. RESULTS

Descriptive R_a statistics before and after thermomechanical aging are summarized in Table 2. Significant differences were observed among the tested materials before and after thermomechanical aging (p<.001). Regardless of the thermomechanical aging, PE had higher R_a than EX (p<.001), whereas no significant differences were seen among PE and other materials (p≥.202). EX had lower R_a than RC before (p=.01) and after thermomechanical aging (p=.018). In addition, CO showed similar R_a to the other materials tested before (p≥.056) and after thermomechanical aging (p≥.101). Thermomechanical aging increased the Ra of all materials significantly (p=.005). Material type significantly affected ΔE_{00} values (p<.001), as PE presented the greatest ΔE_{00} (p≤.002). However, the differences among the other materials were nonsignificant (p>.05) (Table 3).

Table 2. *Median (min-max) and mean ±standard deviation values of the surface roughness before and after thermomechanical aging.*

	Before (µm)		After(µm)	
	Median	Mean	Median	Mean
BH	0.33^{bA} (0.12-0.47)	0.32 ±0.11	0.41^{bB} (0.23-0.56)	0.4 ±0.11
EX	0.09^{aA} (0.04-0.16)	0.09 ±0.04	0.12^{aB} (0.08-0.35)	0.14 ±0.08
RC	0.22^{bA} (0.08-0.43)	0.23 ±0.11	0.26^{bB} (0.16-0.48)	0.3 ±0.1
CO	0.18^{abA} (0.1-0.41)	0.2 ±0.1	0.24^{abB} (0.17-0.5)	0.27 ±0.1

**Different superscript letters indicate significant differences (Uppercase letters for rows and lowercase letters for columns, P <.05). PE: Polyetheretherketone, EX: Glass ceramic, RC: Resilient ceramic, CO: Resin-based composite*

Table 3. *Median (min-max) and mean ±standard deviation values of the ΔE_{00}*

	Median	Mean
BH	1.89^{a} (1.16-2.04)	1.78 ±0.27
EX	0.89^{b} (0.62-1.3)	0.91 ± 0.23
RC	0.98^{b} (0.61-1.25)	0.93 ± 0.23
CO	0.97^{b} (0.41-1.2)	0.92 ± 0.24

**Different superscript letters indicate significant differences (P <.05) PE: Polyetheretherketone, EX: Glass ceramic, RC: Resilient ceramic, CO: Resin-based composite, ΔE00 :Color differences*

4. DISCUSSION

R_a values of the tested materials showed differences before and after thermomechanical aging. In addition, thermomechanical aging significantly increased the R_a of all study groups. Therefore, the first null hypothesis was rejected. The differences among the ΔE_{00} values of the materials tested were significant, which led to the rejection of second null hypothesis.

R_a of a restorative material depends on patient and material related factors (4). Even though inherent material properties have direct effects, polishing or glazing may also influence the R_a (44). Therefore, no polishing other than wet-grounding with silicone carbide papers was performed to materials tested prior to the initial R_a measurements (11). Prior to aging procedure, EX showed significantly lower R_a than RC and PE, whereas CO showed similar values to the all-other materials. To authors' knowledge, the number of studies investigating the R_a of PE after sole wet-grounding and without a polishing procedure is limited (12-14). The initial mean R_a value of PE in the present study (0.33 µm) is in line with those previous studies, as the mean R_a of PE ranged from 0.28 to 1.11 µm when polished with silicon carbide abrasive papers (12-14). The effect of various laboratory and chairside polishing systems on the R_a of PE has also been investigated and all mean R_a values have been reported above the clinically acceptable threshold (10). Considering these results, it can be speculated that monolithic use of PE, even after polishing, could be problematic. However, given the limited number of studies, future studies are needed to corroborate this interpretation. CO and RC had similar R_a before and after aging. This can be attributed to the fact that the materials have similar compositions, although their filler shapes and filler sizes are different (3,44).

Thermomechanical aging statistically increased the R_a of all materials tested in the present study. However, EX showed lower R_a values (0.12 µm) than 0.2 µm (26) even after thermomechanical aging, while RC (0.26 µm) and CO (0.24 µm) showed R_a values slightly higher than the clinically acceptable threshold. Given the fact that no additional polishing was performed after thermomechanical aging, polishing RC and CO with chairside kits might decrease the R_a values below 0.2 µm and improve the longevity of restorations fabricated by using these materials. Even though PE showed similar R_a with RC and CO after thermomechanical aging, the roughness value of PE (0.41 µm) was greater than the clinically acceptable threshold. Considering that the number of mechanical cycles applied in our study approximately reflects 1 year of clinical use (45) and the fact that a greater number of cycles would possibly lead to a coarser surface, it may be speculated that monolithic use of PE might result in restorations with lower color stability and higher plaque accumulation than the other materials tested. However, this hypothesis needs further support with studies involving longer aging cycles as well as post-aging polishing of these materials.

Even though a number of studies have investigated the effect of thermomechanical aging on PE (3,10,34-37), to author's knowledge only 1 study has ever researched the effect of thermomechanical aging on the R_a of PEEK (38). PEEK specimens were subjected to thermomechanical aging with a significantly lower number of cycles (60000 cycles) in Benli et al's (38) study compared with the present study. However, a greater R_a difference was reported (0.139 µm before thermomechanical aging and 0.889 µm after thermomechanical aging) and clinically acceptable threshold of 0.2 µm was exceeded only after thermomechanical aging. A higher initial R_a of PE (0.33 µm) was reported in the present study; however, a direct comparison might be misleading considering the differences in the materials tested as Benli et al (38) utilized a conventional PEEK in their study. Considering the limited number of studies on the R_a of conventional and ceramic-reinforced PEEK after thermomechanical aging, future studies comparing these materials with longer thermomechanical aging cycles are needed.

Mechanical aging increases roughness and surface area of materials that will result in superficial discoloration (21). Previous studies have reported that aging is an important factor affecting the color change of materials (46,47). Among the materials tested, PE showed the greatest ΔE_{00} after thermomechanical aging, which may be related to the high R_a values of the material as a previous study have reported the strong inverse correlation between the R_a and optical properties (16). However, this interpretation needs further support with future studies involving correlation analyzes between the R_a and color stability of the materials tested. Nevertheless, this result corroborates the findings of a previous study (34) where PEEK crowns showed a lower color stability compared with zirconia crowns when subjected to thermomechanical aging. The aging process also can change the molecular structure of high performance polymers, which are hydrophilic in nature, by affecting the water absorption properties (48). Less color change observed in composite and ceramic content groups may be attributed to the lesser change in their molecular structures. Yet, this interpretation needs to be supported with future studies investigating the molecular structure of the tested materials before and after thermomechanical aging.

Paravina et al (43) reported clinically perceptible and acceptable levels of ΔE_{00} as 0.8 and 1.8. Thermomechanical aging caused a color change that perceptible for all the materials tested (ΔE_{00} =1.89 for BH; ΔE_{00} =0.89 for EX; ΔE_{00} =0.98 for C; ΔE_{00} =0.97 for B), whereas only PE showed a color difference that was higher than the acceptability threshold. Considering these results and the fact that PE has a dull appearance, it can be speculated that its indication for esthetic restorations is limited (49). However, this interpretation needs to be corroborated with in vivo studies, which also investigate the clinical performance of PEEK.

This study aimed to compare the inherent R_a of the materials and to evaluate the effect of thermomechanical aging. Therefore, no polishing procedure was performed before the initial R_a measurements. However, the effect of different polishing procedures on the R_a of PEEK has been shown (10). In addition, ceramic-reinforced PEEK was compared with 1 ceramic and 2 resin-based polymers due to chemical similarity. Nevertheless, future studies should compare ceramic-reinforced PEEK with different materials to corroborate the results of our study. The relatively short thermomechanical aging cycle might be considered as another limitation. In addition, thermomechanical aging was performed by using distilled water. However, proteins and enzymes in the saliva may affect the surface properties and the color stability of materials (50). Future studies involving longer aging procedures performed by using saliva might elaborate the understanding of the R_a and color stability of BH, and approximate the findings more to the clinic.

5. CONCLUSION

Based on the findings of this in vitro study, the following conclusions were drawn:

1. Ceramic-reinforced PEEK presented higher Ra than lithium disilicate glass ceramic and the clinical threshold of 0.2 µm regardless of aging.

2. Surface roughness of all materials increased after thermomechanical aging.

3. Ceramic-reinforced PEEK presented the greatest color difference after thermomechanical aging and among the materials tested only the color change of ceramic-reinforced PEEK was greater than the clinically acceptable threshold.

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Author Contributions:

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Acquisition of data for the study: AADT, DH, ÖSK, MBD

Analysis of data for the study: MBD, DH

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