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RESEARCH ARTICLE

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Effect of coffee thermocycling on the surface roughness and stainability of denture base materials with different chemical compositions manufactured with additive and subtractive technologies

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Abstract

Objective: To evaluate the effect of coffee thermocycling (CTC) on the surface roughness (R_a) and stainability of denture base materials with different chemical compositions fabricated by using additive and subtractive manufacturing.

Materials and Methods: Disk-shaped specimens were additively (FREEPRINT denture, AM) or subtractively (G-CAM, GSM and M-PM, SM) fabricated from three pink denture base materials in different chemical compositions ($n = 10$). R_a was measured before and after polishing, while color coordinates were measured after polishing. Specimens were subjected to CTC (5000 cycles) and measurements were repeated. Color differences (ΔE_{00}) after CTC were calculated. R_a among different time intervals within materials was evaluated by using repeated measures analysis of variance (ANOVA), while 1-way ANOVA was used to evaluate the R_a of different materials within each time interval and the ΔE_{00} values. Color coordinates within each material were compared by using paired samples t-tests ($\alpha = 0.05$).

Results: R_a before polishing was the highest for all materials ($p < 0.001$), while SM had its lowest R_a after CTC and AM had its lowest R_a after polishing ($p \leq 0.008$). Before polishing, AM had the highest R_a among the materials ($p < 0.001$). After polishing, SM had higher R_a than AM ($p < 0.001$). After CTC, GSM had the lowest R_a ($p \leq 0.048$). SM had the lowest ($p \leq 0.031$) and AM had the highest ($p < 0.001$) ΔE_{00}. CTC decreased the a^* and b^* values of SM and AM ($p \leq 0.017$), and increased the L^* values of AM ($p < 0.001$).

Conclusions: Polishing significantly reduced the surface roughness of all materials. CTC did not increase the surface roughness of materials above the clinically acceptable threshold. Only AM had perceptible color change when previously reported threshold values for denture base materials were considered.

Clinical Significance: Tested denture base materials may have similar surface stability after coffee thermocycling. However, subtractively manufactured denture base materials may have improved color stability when subjected to long-term coffee consumption.

KEYWORDS
CAD-CAM, coffee thermocycling, denture base, roughness, stainability

1 | INTRODUCTION

Polymethyl methacrylate (PMMA) is the most commonly used material for denture bases[1,2] considering its advantages such as ease of processing, repairing, and polishing, low density, being cost-effective, and adequate physiochemical properties.[3] However, conventional PMMA's polymerization shrinkage, susceptibility to bacterial plaque accumulation, and possible allergic potential due to release of unreacted monomers,[4] along with the advancements in computer-aided design and computer-aided manufacturing (CAD-CAM) technologies have increased the popularity of additively and subtractively manufacturing denture bases.[5–8]

Reinforcement of PMMA with fibers and nanoparticles to improve different physical and mechanical properties has been investigated broadly.[1] Even though subtractively manufactured prepolymerized disks has improved mechanical properties,[9] reinforcement of prepolymerized PMMA disks with nanographene has also been introduced.[10] Graphene, which is referred as the thinnest material in the universe,[11] is a 2-dimensional honeycomb-shaped crystalline form of carbon[12,13] that is used as a reinforcement phase in PMMA.[14] Nanographene-reinforced PMMA could be considered as promising as a recent study has reported higher flexural strength of nanographene-reinforced PMMA compared with prepolymerized PMMA.[10]

Surface roughness (R_a) is a critical determinant for the long-term use of a prosthesis as increased R_a might lead to plaque accumulation and biofilm formation along with esthetic complications due to discoloration.[3] Previous studies have reported 0.2 μm as the acceptability threshold for R_a.[3,9] Along with increased R_a, staining beverages may also result in discoloration.[15] Even though nanographene-reinforced PMMA has been investigated in previous studies,[10,12–14,16–19] only one study has evaluated the effect of coffee thermocycling (CTC) on nanographene-reinforced PMMA's R_a and stainability, which tested tooth-colored materials.[20] In addition, the number of studies on the R_a and stainability of additively manufactured denture base materials after CTC is limited to one and that study did not involve a comparison with nanographene-reinforced PMMA.[5] Therefore, the present study aimed to evaluate the effect of CTC on the R_a and stainability of denture base materials with different chemical compositions, one of which was a nanographene-reinforced PMMA, fabricated by using either additive or subtractive manufacturing. The hypotheses were that (i) time interval (before polishing, after polishing, and after CTC) would affect the R_a within each material, (ii) material type would affect the R_a within each time interval, (iii) material type would affect the stainability of tested materials after CTC, and (iv) CTC would affect the color coordinates of tested materials.

2 | MATERIALS AND METHODS

Table 1 lists detailed information regarding the materials tested in the present study. Disk-shaped specimens (Ø10 × 2 mm) were fabricated by using one subtractively manufactured prepolymerized PMMA (Merz M-PM; Merz Dental GmbH [SM]), one subtractively manufactured nanographe-reinforced PMMA (G-CAM; Graphenano DENTAL [GSM]), and one additively manufactured denture base resin (FREEPRINT denture; DETAX GmbH & Co KG [AM]) ($n = 10$). All materials were in pink shade. The number of specimens per group was decided based on previous studies on the roughness and stainability of additively and subtractively manufactured denture base materials[5,6,9] or subtractively manufactured nanographene-reinforced PMMA.[20] In addition, a priori power analysis based on the results of a pilot study that investigated the effect of polishing and CTC on the R_a and stainability of polymers had been conducted in one of those studies and yielded 10 specimens per material sufficient to detect an effect size of 0.1 with $1 - \beta = 80\%$ and $\alpha = 0.05$.[20] Subtractively manufactured specimens (SM and GSM) were fabricated by using a 10 mm-wide cylinder-shaped standard tessellation language (STL) file. This STL file was imported into a software (PrograMill CAM V4; Ivoclar AG) and cylinder-shaped specimens were milled from CAD-CAM disks with a 5-axis milling unit (PrograMill PM7; Ivoclar AG). These cylinder-shaped specimens were then wet-sliced with a precision cutter (Vari/cut VC-50; Leco Corp) to obtain 2 mm-thick specimens. Additively manufactured specimens (AM) were fabricated by using an STL file designed in the final dimensions. This STL file was imported a nesting software (Composer; ASIGA), positioned vertically on the build platform, and this configuration duplicated to arrange a total of 10 specimens on the build platform. A 3-dimensional printer with digital light processing technology (MAX UV; ASIGA) was used to fabricate AM specimens. After fabrication, AM specimens were initially placed in a beaker containing isopropyl alcohol and cleaned by using an ultrasonic cleaner (Eltrosonic Ultracleaner 07–08; Eltrosonic GmbH) for 3 min. Specimens were then transferred into another beaker containing fresh isopropyl alcohol and cleaned for an additional 3 min by using the same ultrasonic cleaner. After cleaning, specimens were light-polymerized by using a xenon polymerization unit

TABLE 1 Materials used in this study.

Material	Type	Composition	Manufacturer
M-PM (SM)	Subtractively manufactured prepolymerized PMMA	PMMA: >98% Methyl 2-methylprop-2-enoate; Methyl 2-methylpropenoate; Methyl methacrylate: <1% Dibenzoyl peroxide; Benzoyl peroxide: <1%	Merz Dental GmbH, Lütjenburg, Germany
G-CAM (GSM)	Subtractively manufactured prepolymerized nanographene-reinforced PMMA	Not available	Graphenano Dental, Valencia, Spain
FREEPRINT denture (AM)	Additively manufactured denture base resin	Isopropylidenediphenol peg-2 dimethacrylate 35%–<60% 7,7,9-trimethyl-4,13-dioxo-3,14-dioxa-5,12-diazahexadecane-1,16-diyl bismethacrylate 30%–<35% 1,6-hexanediol dimethacrylate 1%–<5% 2-hydroxyethyl methacrylate 1%–<5% Diphenyl(2,4,6-trimethylbenzoyl)phosphine oxide 1%–<5% Hydroxy propyl methacrylate 1%–<5% Phenyl bis(2,4,6-trimethylbenzoyl)-phosphine oxide <1%	DETAX GmbH & Co KG, Ettlingen, Germany

(Otoflash G171; NK Optik) under a nitrogen oxide gas atmosphere with 4000 lighting exposures (2×2000).[21]

After the fabrication of all specimens, their initial R_a was measured by using a non-contact optical profilometer equipped with an H0 sensor (FRT MicroProf 5; Fries Researsch & Technology GmbH). The parameters were set to 5.5 mm of tracing length, 0.8 mm of cut-off Lc value, 1 μm of resolution, and 1000/mm of pixel density.[6] Integrated proprietary software of the profilometer (Mark III; Fries Research & Technology GmbH) was used to measure the R_a of three vertical and three horizontal traces that were 1 mm apart from each other. These values were then averaged.

After initial R_a measurements, all specimens were conventionally polished. Each specimen was initially ground by consecutively using silicon carbide abrasive papers (Struers Labo-Pol 21 #280, #360, and #1000; Struers), polished by using a pumice slurry (Pumice fine; Benco Dental) for 90 s (1500 rpm) followed by fine polishing with a polishing paste (Fabulustre; Grobet USA) for an additional 90 s.[9] Specimens were then cleaned in a distilled water containing ultrasonic bath, air-dried, and the R_a values were remeasured.

A digital spectrophotometer (CM-26d; Konica Minolta), which has a Commission International de l'Eclairage (CIE) D65 illuminant and uses CIE Standard (2-degree) human observer characteristics, was used to measure the color coordinates of each specimen on a gray background.[5,9,20] All measurements were performed in the same temperature- and humidity-controlled room with daylight by a single operator and the spectrophotometer was calibrated before each scan. The optical contact between the specimen and the background was ensured by using saturated sucrose solution, which provides more clinically relevant measurements as the refractive index of air is avoided. Three measurements were recorded for each specimen and these values were averaged.

Specimens were finally subjected to 5000 thermocycles (SD Mechatronik Thermocycler; SD Mechatronik GmbH) at 5–55°C with a dwell time of 30 s and a transfer time of 10 s.[9] A freshly brewed coffee solution that had a ratio of 177 mL of water for every tablespoon of coffee (Intenso Roasted and Grounded; Kaffeehof GmbH) was used as the thermocycling medium. Coffee solution was changed every 12 h.[3] After CTC, coffee extracts were removed by brushing each specimen 10 times with a toothpaste (Colgate Total Pro Breath Health; Colgate-Palmolive) under running water and ultrasonically cleaning in distilled water for 10 min (Figure 1). After specimens were air-dried, R_a and color coordinate measurement of each specimen were repeated.

Shapiro Wilk test was used to analyze the distribution of both R_a and ΔE_{00} data. Both R_a and ΔE_{00} data did not refute normal distribution; thus, parametric tests were preferred. Repeated measures analysis of variance (ANOVA) and Bonferroni corrected paired samples t-tests were used to evaluate the differences in R_a among different time intervals within each material. Comparison of different materials within a time interval was performed by using 1-way ANOVAs followed either by Tukey's HSD (after thermocycling) or Tamhane's T2 (before and after polishing) tests. ΔE_{00} values of materials were compared by using 1-way ANOVA and Tamhane's T2 tests. Color coordinates of each material between after polishing and after thermocycling were compared by using paired samples *t*-tests. A statistical analysis software (SPSS Statistics v24; IBM Corp) was used for all analyses ($\alpha = 0.05$). Perceptibility (1.72 units) and acceptability (4.08 units) of ΔE_{00} values were also evaluated by the thresholds set by a previous study.[22]

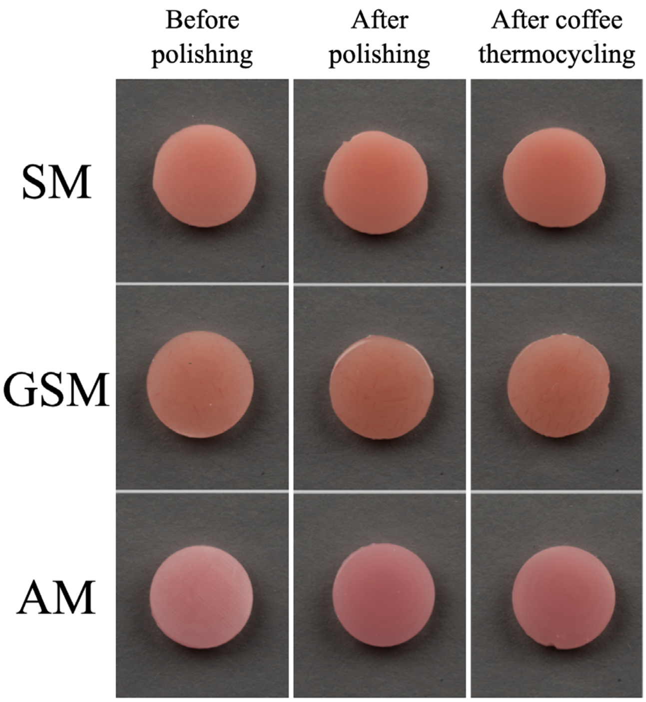

FIGURE 1 Representative image of one specimen after each time interval.

TABLE 2 Mean ± standard deviation surface roughness values of each material-time interval pair.

	Before polishing	After polishing	After CTC
SM	0.46 ± 0.06^{Aa}	0.16 ± 0.03^{Bb}	0.12 ± 0.02^{Bc}
GSM	0.41 ± 0.11^{Aa}	0.12 ± 0.04^{ABb}	0.1 ± 0.01^{Ab}
AM	5.47 ± 0.69^{Ba}	0.09 ± 0.01^{Ac}	0.13 ± 0.02^{Bb}

Note: Different superscript letters indicate significant differences (uppercase letters for columns and lowercase letters for rows) ($p < 0.05$).

3 | RESULTS

Table 2 summarizes descriptive statistics of R_a values of each material-time interval pair. Repeated measures ANOVA tests revealed significant differences among time intervals for each material ($p < 0.001$). Tested materials had the highest R_a before polishing ($p < 0.001$). SM had its lowest R_a after CTC ($p \leq 0.008$), while AM had its lowest R_a after polishing ($p < 0.001$). The difference between after polishing and after CTC R_a of GSM was nonsignificant ($p = 0.392$). Significant differences were observed among tested materials within each time interval ($p \leq 0.001$). Before polishing, SM and GSM ($p = 0.539$) had similar values that were lower than that of AM ($p < 0.001$). After polishing, SM had higher R_a than AM ($p < 0.001$), while GSM had similar values to the other materials ($p \geq 0.098$). After CTC, SM and AM had similar values ($p = 0.253$) that were higher than that of GSM ($p \leq 0.048$).

Table 3 summarizes descriptive statistics of ΔE_{00} values and color coordinates. SM had the lowest ($p \leq 0.031$) and AM had the highest

TABLE 3 Mean ± standard deviation color coordinate and ΔE_{00} values of each material.

		After polishing	After CTC	ΔE_{00}
SM	L^*	45.7 ± 0.17^{a}	45.6 ± 0.22^{a}	0.19 ± 0.1^{A}
	a^*	15.36 ± 0.09^{a}	15.24 ± 0.13^{b}	
	b^*	6.49 ± 0.08^{a}	6.42 ± 0.07^{b}	
GSM	L^*	39.03 ± 0.7^{a}	38.91 ± 0.27^{a}	0.51 ± 0.41^{B}
	a^*	10.5 ± 0.38^{a}	10.4 ± 0.29^{a}	
	b^*	4.99 ± 0.25^{a}	4.91 ± 0.11^{a}	
AM	L^*	39 ± 0.48^{b}	39.88 ± 0.49^{a}	1.76 ± 0.25^{C}
	a^*	12.05 ± 0.33^{a}	11.45 ± 0.15^{b}	
	b^*	6.15 ± 0.47^{a}	4.13 ± 0.51^{b}	

Note: Different lowercase letters indicate significant differences between time intervals within each material for each color coordinate, while different uppercase letters indicate significant differences in columns ($p < 0.05$).

($p < 0.001$) ΔE_{00} values. SM had lower a^* ($p = 0.005$) and b^* ($p = 0.017$) values after CTC than after polishing. While a^* ($p < 0.001$) and b^* ($p < 0.001$) values of AM were lower after CTC, its L^* values were higher after CTC ($p < 0.001$). Figure 2 illustrates the trend of color coordinates between different time intervals among materials.

4 | DISCUSSION

Significant differences in R_a were observed among tested materials within each time interval and among different time intervals within each material. Therefore, the first and the second null hypotheses were accepted. All tested materials had higher R_a than clinically acceptable threshold of 0.2 μm before polishing. However, polishing not only statistically reduced these values but also resulted in acceptable R_a, and CTC did not increase the R_a of tested materials above 0.2 μm. Both of these findings are in line with previous studies.[3,6,20,23–26] SM and GSM had significantly lower R_a than AM before polishing, which was the time interval with the widest mean difference among tested materials. The favorable R_a of SM and GSM may be attributed to their fabrication process as prepolymerized PMMA disks are polymerized under high temperature and pressure that lead to higher degree of conversion and lower residual monomers.[6] GSM and SM had similar R_a before and after polishing, while GSM had lower R_a after CTC. However, the authors think that the difference between GSM's and SM's R_a after CTC may be clinically negligible considering that the difference in mean values was 0.02 μm. Even though tested materials were from different brands, these findings may indicate the fact that nanographene inclusion did not affect the R_a. It can also be speculated that surface of tested denture base materials are resistant to excessive coffee consumption considering that a worst-case scenario was simulated in the present study; for an individual who consumes 1 cup of coffee per day, the test arrangement in the present study simulates potentially over couple decades of intraoral service. Nevertheless, future studies should investigate the

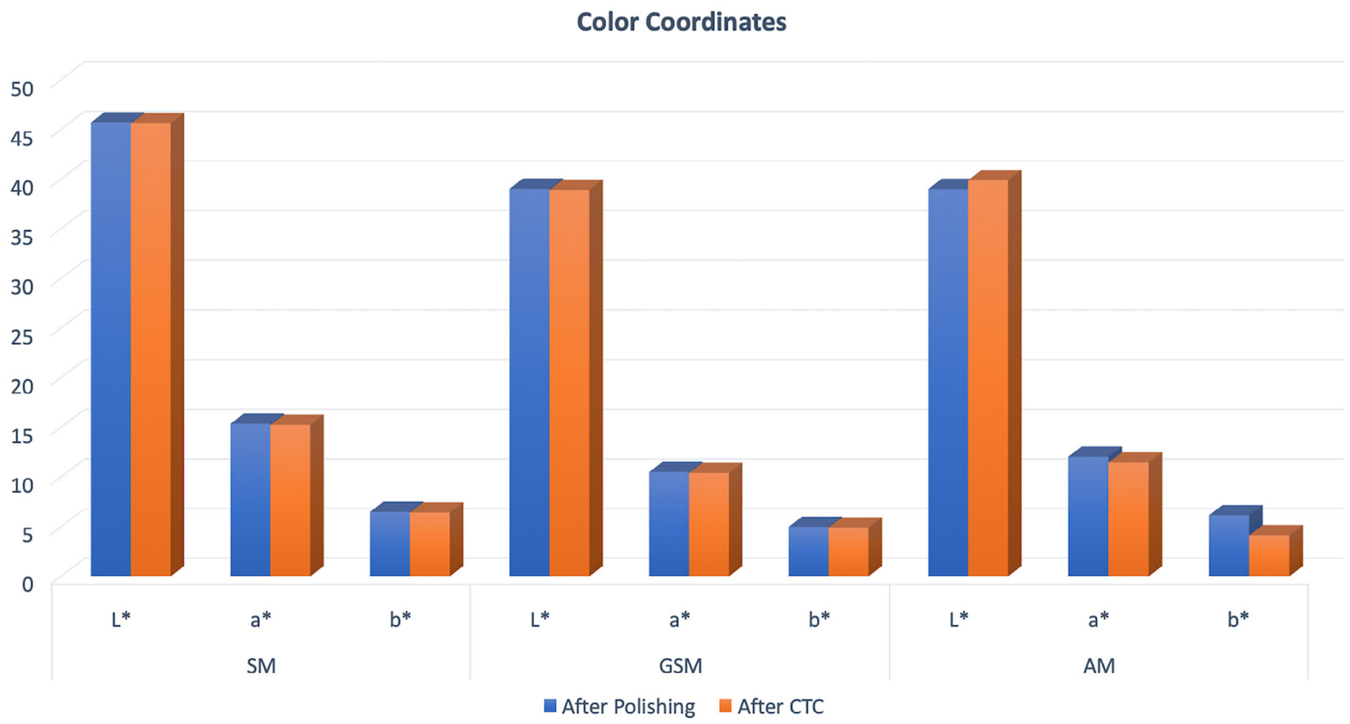

FIGURE 2 Color coordinates of each material after polishing and after coffee thermocycling.

correlation between the R_a and bacterial plaque accumulation of tested materials after CTC with longer durations to elaborate the limitations of tested materials.

Material type significantly affected the stainability of tested materials after CTC as SM had the lowest and AM had the highest ΔE_{00} values. Thus, the third null hypothesis was also accepted. Among tested materials, only AM had perceptible color change after CTC ($\Delta E_{00} = 1.76$ units) when ΔE_{00} values of tested materials were further investigated according to the threshold values reported by Ren et al.[22] However, none of the tested materials had mean ΔE_{00} values higher than 4.02, which can be interpreted as acceptable color stability for tested materials after long-term coffee consumption. Therefore, it can be hypothesized that the chemical composition of tested resins may have a small effect on the esthetic clinical outcomes of denture bases. Nevertheless, the higher susceptibility of AM to discoloration may be related with its chemical composition as it has a more heterogeneous composition when compared with SM (Table 1). However, considering that the manufacturer of GSM has not disclosed their product's composition, this speculation needs further support. Another possible explanation of AM's susceptibility to discoloration may be its R_a values after CTC as AM had a higher mean value than the other materials. Another explanation for AM's susceptibility to discoloration may be related with potentially lower degree of conversion of AM resin. A recent study has reported an inverse relationship between the degree of conversion and color stability[8] and considering that AM was the only additively manufactured material tested, higher stainability can be considered expectable due to potential low degree of conversion of AM resin, compared with the prepolymerized specimens.

The only previous study on the R_a and stainability of GSM[20] has also reported findings similar to those in the present study. Çakmak et al.[20] concluded that tooth-colored GSM had acceptable R_a after polishing that did not change after CTC, and the material had an imperceptible color difference after CTC. However, no significant difference in terms of surface roughness and color change were reported between the tooth-colored GSM and the tooth-colored prepolymerized PMMA in Çakmak et al's study.[20] But, it should be noted that the significance for color change found in the present study between GSM and SM was statistical and not clinical (0.19 vs. 0.51), considering the perceptibility threshold (1.72). The color change values in Cakmak et al's[20] study (0.34 and 0.31) and those found in the present study (0.19 and 0.51) were similar in terms of clinical perceptibility. The differences, even though small, between the values in these two studies may be attributed to the inherent color of tested materials (white vs. pink).

When the color coordinates of materials before and after CTC were considered, SM and AM had significantly lower redness (a^* values) and yellowness (b^* values), which can also be interpreted as a shift towards green and blue for SM and AM specimens. In addition, AM also had higher lightness (L^* values). Therefore, the fourth null hypothesis was accepted. These changes are rather unexpected considering that coffee would be expected to darken the specimens and shift their color more towards red and yellow. However, it should be noted that there are no universally accepted threshold values for clinical perceptibility and acceptability for the changes in color coordinates. Therefore, these changes may not be clinically relevant.

Even though the number of studies on the optical properties of GSM is limited,[14,20] its mechanical properties have been broadly investigated.[10,12,13,16–19] A previous study on the R_a of GSM has reported that it had similar values to those of tested prepolymerized PMMA.[19] GSM was also reported to have microhardness that was either similar to or higher than prepolymerized PMMA.[14,16,17,19] In addition, Hernández et al.[12] reported that thermocycling did not affect GSM's microhardness. Flexural strength of GSM has also been investigated and was shown to be similar to or higher than that of conventional and prepolymerized PMMA.[13,14,19] Another study reported similar fabrication trueness for the crowns fabricated by using GSM when compared with those of fabricated by using prepolymerized PMMA, reinforced resin composite, and additively manufactured resin composite.[18] Considering the results of the present and the abovementioned studies, it can be stated that GSM can be considered as a promising material in terms of mechanical and optical properties. However, it should also be noted that the authors are unaware of an in vivo study on GSM and its limitations should be elaborated with long-term studies under different clinical situations.

Given that the present study was the first on the R_a and stainability of pink GSM, a priori power analysis based on the results of previous studies could not be performed, which could be considered as a limitation. Nevertheless, significant differences were observed among tested materials and the number of specimens in each group was based on previous studies on the R_a and discoloration of additively and subtractively manufactured PMMAs[5,6,9,20]; one of those studies reported the results of a priori power analysis based on a pilot study.[20] In addition, post hoc power analyses were performed for each parameter investigated and the sample size was deemed adequate for a minimum of 83% power with a minimum effect size of 0.62 and $\alpha = 0.05$. Another limitation was the fact that CTC led to discoloration of both sides of the specimens. However, only polished surfaces are exposed to discolorants intraorally. Therefore, ΔE_{00} values may have been amplified, and smaller color changes may be expected intraorally. Coffee was deliberately chosen given that its acidic components of tannin and chlorogenic acids accelerate discoloration.[9] However, it was the only discolorant tested and different solutions[2] or coffee solutions prepared by using different powder-to-water ratio that would affect the pH of the solution may affect the results. Even though tested CAD-CAM denture base materials in the present study can be considered as novel, the fact that only three materials tested was a limitation. Future studies should focus on other optical and mechanical properties of tested additively and subtractively manufactured denture base materials after longer CTC durations or after being subjected to other aging methods.

5 | CONCLUSIONS

Within the limitations of the present study, it can be concluded that

1. Material type and time interval affected the surface roughness.
2. Polishing reduced the surface roughness of all materials, and coffee thermocycling did not lead to an increase in surface roughness beyond clinical acceptability, considering the published threshold (0.2 μm).
3. Additively manufactured denture base resin had the highest and prepolymerized PMMA had the lowest color difference after coffee thermocycling. However, only additively manufactured denture base resin had perceptible color change when previously reported thresholds were considered. CTC decreased the redness and the yellowness of additively manufactured denture base resin and prepolymerized PMMA. The lightness of additively manufactured denture base resin increased after CTC.

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CONFLICT OF INTEREST STATEMENT

The authors declare that they do not have any financial interest in the companies whose materials are included in this article

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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