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Effect of thermocycling on the surface properties of CAD-CAM denture base materials after different surface treatments

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ABSTRACT

Purpose: To evaluate the effect of thermocycling on the water contact angle (WCA), surface roughness (SR), and microhardness (MH) of different CAD-CAM PMMA denture base materials after different surface treatments (conventional laboratory polishing, polishing kit, or surface sealant).

Materials and methods: Disk-shaped specimens (10 × 2 mm) of 3 different CAD-CAM PMMAs, AvaDent (AV); Merz M-PM (M-PM); Polident (Poli), and a conventional heat-polymerized PMMA (Vynacron) (CV) (n=21) were divided into 3 different surface treatment groups (n=7): conventional laboratory polishing (CLP), polishing with acrylic resin polisher kit (PK), and a surface sealant (Palaseal) (SSC). Stereomicroscopic images were taken both before and after thermocycling. WCA, SR, and MH of all specimens were measured before and after thermocycling and compared by using a 2-way ANOVA (α=0.05).

Results: After thermocycling, WCA significantly increased for CLP- or PK -applied ($P<.001$) specimens of all materials and SSC-applied M-PM ($P=.002$), SR significantly increased for CLP-applied M-PM ($P=.027$) and PK-applied Poli ($P=.041$), and MH significantly decreased for CLP- or PK-applied AV ($P = .001$, $P < .001$, respectively), CV ($P=.033$, $P=.023$, respectively), and M-PM ($P=.003$, $P=.001$, respectively), SSC-applied M-PM ($P<.001$), and CLP-applied Poli ($P<.001$). Stereomicroscopic images revealed rougher surfaces for PK-applied specimens.

Conclusions: After thermocycling, surface treatment had a significant effect on water contact angle and surface roughness. CLP or PK application resulted in hydrophobic surfaces compared with before thermocycling. CLP or SSC application on CAD-CAM PMMAs resulted in smoother surfaces. Thermocycling lowered the microhardness of all PMMAs, and the decrease was significant in CLP- or PK-applied PMMAs, except for PK-applied Poli.

1. Introduction

Polymethyl methacrylate (PMMA) is a synthetic polymer prepared by the free radical addition and polymerization of methyl methacrylate to poly methyl methacrylate. The polymerization reaction may be activated by the generation of free radicals by chemical inhibitor in the monomer or energy means, such as heat, light or microwaves. Heat-polymerized PMMA materials are available in powder and liquid forms. PMMA powder contains PMMA, benzoyl peroxide initiator, a plasticizer (dibutyl phthalate), opacifiers (titanium and zinc oxides), fibers, and pigments or dyes. The liquid component contains methyl methacrylate (MMA) monomer, ethylene glycol dimethacrylate, and hydroquinone. Heat-polymerized PMMA are commonly used in dentistry for the fabrication of dental and maxillofacial prostheses, as

Abbreviations: ANOVA, Analysis of variance; AV, AvaDent Pink CAD-CAM PMMA; CAD-CAM, Computer-aided design and computer-aided manufacturing; CLP, Conventional laboratory polishing; CV, Vynacron heat polymerized PMMA; MH, Microhardness; M-PM, Pink M-PM CAD-CAM PMMA; PMMA, Polymethyl methacrylate; Poli, Polident CAD-CAM PMMA; SR, Surface roughness; TC, Thermocycling; WCA, Water contact angle.

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they show good physical properties, has an adequate strength, are easy to repair, and have acceptable esthetics with a low cost (Vest, 1947; Zafar, 2020). However, its mechanical properties have shown some deficiencies such as dimensional changes, water sorption, monomer release, and increased risk of denture-associated infections. (Saponaro et al., 2016; Berger et al., 2006; Satpathy and Gujjari, 2013).

Advances in computer-aided design and computer-aided manufacturing (CAD-CAM) technologies promoted the digital workflows with new CAD-CAM materials for complete denture fabrication (Awada and Nathanson, 2015; Srinivasan et al., 2018, 2019). Although the chemistry of CAD-CAM PMMA is similar to that of conventional heat-polymerized, CAD-CAM PMMA are industrially polymerized under standardized conditions at a high temperature and pressure, in order to improve its properties, such as hardness, flexural strength, flexural modulus, impact strength. Therefore, they undergo no polymerization shrinkage, due to the milling process, and show less residual monomer (Prpic et al., 2020; Srinivasan et al., 2017, 2018; Huettig et al., 2017; Awada and Nathanson, 2015).

The surface properties of acrylic resins such as roughness, hardness, surface energy, and wettability may affect plaque accumulation (Kuhar and Funduk, 2005; Bollen et al., 1997; Hahnel et al., 2010). Acrylic resin denture bases have been traditionally polished with water and pumice or by using conventional resin polishing kits. In recent years, surface sealants have also been in use on resin-based materials to eliminate surface defects and to increase wear and stain resistance (Al-Rifaiy, 2010; Berger et al., 2006; Gungor et al., 2014; Kuhar and Funduk, 2005).

To the authors' knowledge, it is not well-known which surface treatment improves the surface characteristics of these new materials and what the effect of thermocycling (TC) on these materials is. The aim of the present in vitro study was to evaluate the effect of TC on the water contact angle (WCA), surface roughness (SR), and microhardness (MH) of different CAD-CAM PMMA denture base materials after the application of varying surface treatments (conventional laboratory polishing, polishing kit, or surface sealant). The first null hypothesis was that the surface treatment type would not affect the WCA, SR, and MH of CAD-CAM PMMAs after TC. The second null hypothesis was that the WCA, SR, and MH of tested CAD-CAM PMMAs would not be different.

2. Materials and methods

2.1. Specimen preparation

Disk-shaped specimens (10 × 2 mm) were prepared from 3 different prepolymerized CAD-CAM PMMAs (AvaDent, Global Dental Science, AZ, USA (AV); Merz M-PM, Merz Dental GmbH, Lütjenburg, Germany (M-PM); Polident d.o.o, Volčja Draga, Slovenia (Poli)) and a heat-polymerized PMMA (Vynacron Dental Resins Inc., Manalapan, USA) (CV) (n = 21) (Table 1).

For the preparation of prepolymerized CAD-CAM PMMA specimens, cylinders 10 mm in diameter were CAD, converted to standard tessellation language (STL) files, and wet-milled (M1 Milling Unit, Zirkonzahn, South Tyrol, Italy). Disk-shaped specimens with a final thickness of 2 ± 0.02 mm was obtained by slicing cylinder specimens using a cutting machine (Vari/cut VC-50, Leco Corporation, St Josephs, MI, USA) under water cooling.

For the preparation of conventional heat-polymerized PMMA specimens, wax patterns in identical size as CAD-CAM specimens were flasked, boiled out and eliminated. PMMA was mixed and packed into flasks under pressure after having a doughy stage. The specimens were heat polymerized (74 °C, 8 h) (Jadhav et al., 2013). After cooling, specimens without voids and porosity were gently trimmed.

One surface of all specimens was smoothed with 1200# SiC papers (Leco, Leco Corporation, St Josephs, MI, USA) under water and divided into 3 surface treatment groups (n = 7): conventional laboratory polishing (CLP), polishing with an acrylic polisher kit (PK), and surface sealant coupling (Palaseal denture lacquer, Kulzer GmbH, South Bend, IN) (SSC). All surface treatments were performed only on previously smoothed surfaces of specimens and by one operator (G.Ç.).

2.2. Surface treatment

In CLP groups, pumice slurry (Pumice fine, Benco Dental, Pittston, PA) was applied for 90 s (1500 rpm) (Red-Wing, Handler Mfg, Westfield, NJ, USA) (Sahin et al., 2016) and the specimens were fine polished using a polishing paste (Fabulustre, Grobet USA, Carlstadt, USA) for 90 s.

In PK groups, specimens were manually polished using a 3-stage polishing kit (Acrylic Polisher HP blue, Edenta AG, Switzerland) for 60 s (20 s for each instrument) at 10 000 rpm. The specimens were trimmed with dark blue instruments and then polished with light blue and brown instruments, respectively.

In SSC groups, specimens were coupled with one uniform thin layer of surface sealant by using a soft microbrush in one direction (Cakmak et al., 2020). After 20 s, specimens were inserted in a light-polymerizing device (Triad, 2000; Dentsply Sirona, York, PA) and surface sealant was polymerized for 90 s (Cakmak et al., 2020). All specimens were then ultrasonically cleaned for 10 min in distilled water (Eltrosonic Ultracleaner 07–08, Eltrosonic GmbH, Wiesbaden, Germany) before WCA, SR and MH measurements.

2.3. Water contact angle assessment

For the evaluation of WCA, contact angles of deionized water droplets were measured by using the sessile drop method (Olympus TGHM, Rame-hart Inc, N.J, USA). The right and the left static contact angles of single water droplets (2.0 μL) were measured immediately after the droplet contact with the center of the specimen, and averaged. For each specimen, 3 consecutive measurements were done and averaged (Hahnel et al., 2010; Rosentritt et al., 2009; Qian et al., 2016).

2.4. Surface roughness assessment

After WCA measurements, the SR of treated surface of each specimen was measured with a contact profilometer (Surftest SV-3100, Mitutoyo Corporation, Kawasaki, Japan). After calibrating the profilometer, 3 measurements were done at the center of each specimen and at least 0.5 mm away from each other at a tracing length of 5.5 mm, cut-off value of

Table 1
Materials used in this study.

Material	Type	Composition	Code	Manufacturer	Lot No
Pink AvaDent	Prepolymerized poly (methyl methacrylate)	PMMA 99.5% Pigments < 1.0%	AV	Global Dental Science, AZ, USA	14988
Pink M-PM Disc	Prepolymerized poly (methyl methacrylate)	PMMA and cross-linked polymers, dyes, residual peroxide, and MMA	M-PM	Merz Dental GmbH, Lütjenburg, Germany	Z5355486
Pink Polident CAD-CAM discs	Prepolymerized poly (methyl methacrylate)	PMMA and Pigments	Poli	Polident d.o.o, Volčja Draga, Slovenia	31215
Vynacron	Heat-polymerized poly (methyl methacrylate)	Powder: PMMA, benzoyl peroxide, dibutyl phthalate, opacifier, and pigments. Liquid: MMA monomer, ethylene glycol dimethacrylate, and hydroquinone	CV	Vynacron Dental Resins Inc, Manalapan, USA	32538

0.8 mm and stylus speed of 1 mm/s. The mean R_a values were calculated in micrometers (μm).

2.5. Microhardness assessment

After SR measurements, the Knoop MH of treated surface of each specimen was measured using a microhardener (M-400 Hardness Tester, Leco Corporation, St. Joseph, MI), under 25-g load during 10s (Goiato et al., 2014). For each specimen, 5 consecutive indentations 500 μm distant from each other were made for Knoop MH and averaged (Goiato et al., 2014).

After baseline WCA, SR and MH measurements, the specimens were subjected to 5000 TC (Buchi 461 Water Bath, Buchi Corporation, New Castle, DE, USA) in distilled water (5 °C/55 °C; dwell time-30 s, transfer time-10 s) (Cakmak et al., 2020) and the measurements were repeated by using the methods used for the baseline measurements.

2.6. Stereomicroscopic images

Stereomicroscopic images were taken before and after TC using an optical microscope (SMZ-U Stereoscopic Zoom Microscope, Nikon, Melville, New York, USA) at a × 40 magnification (Cakmak et al., 2021).

2.7. Statistical analysis

Descriptive statistical analyses were performed (SPSS Inc, Chicago, IL). Means and 95% confidence limits for WCA, SR, and MH values were calculated for each combination of material type and surface treatment both before and after TC. Data were analyzed with a repeated-measures 2-way analysis of variance (ANOVA) in which the main effects were material type and surface treatment, and their 2-way interaction. After the initial analysis, it was observed that the sample size allowed the detection of highly statistically significant differences; therefore, no additional specimen preparation was deemed necessary. Any significant interactions were resolved by using a Tukey test ($\alpha = 0.05$).

3. Results

After TC (Table 2), for WCA and SR, the surface treatment had a significant effect ($P < .001$) and a significant interaction was found between the surface treatment and the material ($P = .007$, $P = .049$, respectively). For MH, effects of material ($P < .001$), surface treatment ($P < .001$), and their interaction were significant ($P < .001$) both before and after TC.

After TC, SSC resulted in the lowest WCA for AV ($P < .001$), CV (P values for SSC-CLP <.001, SSC-PK = .002) and M-PM (P values for SSC-CLP = .009, SSC-PK = .007), and SSC resulted in significantly lower WCA than PK application ($P = .004$) for Poli. When after TC was compared with before TC, WCA significantly increased within CLP- ($P < .001$) or PK-applied ($P < .001$) materials and SSC-applied M-PM ($P = .002$), whereas decreased within SSC-applied CV ($P = .019$) (Table 3).

Table 2
Summary of ANOVA of water contact angle, surface roughness and microhardness after thermocycling. df, numerator degrees of freedom.

Test	Effect	After Thermocycling		
		df	F	*P*
Water contact angle	Material	3	0.498	.685
	Surface treatment	2	48.130	<.001
	Material × Surface treatment	6	3.266	.007
Surface roughness	Material	3	2.584	.060
	Surface treatment	2	36.508	<.001
	Material × Surface treatment	6	2.237	.049
Microhardness	Material	3	89.929	<.001
	Surface treatment	2	25.975	<.001
	Material × Surface treatment	6	13.787	<.001

PK resulted in the highest (P values for PK-CLP <.001, PK-SSC = 0.005) and CLP resulted in the lowest (P values for CLP-PK <.001, CLP-SSC = .017) SR for M-PM material after TC. After TC, PK resulted in the highest SR for AV (P values for PK-CLP = .009, PK-SSC = 0.005) and Poli (P values for PK-CLP<.001, PK-SSC = 0.001). For CV, no significant difference was found in SR among surface treatments ($P \geq .231$). When after TC was compared with before TC, SR of CLP-applied M-PM ($P = .027$) and PK-applied Poli ($P = .041$) significantly increased (Table 4).

For MH (Table 5), CLP resulted in the highest MH for AV after TC ($P < .001$). After TC, SSC resulted in the highest MH for CV (P values for SSC-CLP = .016, SSC-PK = .022). CLP resulted in significantly higher MH than SSC application ($P = .006$) for M-PM. CLP resulted in the highest MH ($P < .001$) and SSC resulted in the lowest MH (P values for SSC-CLP <.001, SSC-PK = .006) for Poli. When after TC was compared with before TC, MH significantly decreased in CLP- or PK-applied AV ($P = .001$, $P < .001$, respectively), CV ($P = .033$, $P = .023$, respectively), M-PM ($P = .003$, $P = .001$, respectively), SSC-applied M-PM ($P < .001$), and CLP-applied Poli ($P < .001$).

According to the stereomicroscopic images (Figs. 1 and 2), PK-applied PMMAs were mechanically rougher including surface irregularities in the form of scratches compared with other surface treatments. CLP- or SSC-applied surfaces were relatively smoother. Surface sealant maintained its integrity on tested PMMAs after TC. When after TC was compared with before TC, no significant difference was visually observed on tested PMMAs' surface morphology with different surface treatments.

4. Discussion

The first null hypothesis was rejected as the surface treatment significantly affected the WCA, SR, and MH of CAD-CAM PMMAs after TC. The second null hypothesis was also rejected since the PMMA material significantly affected the MH after TC.

The present study found a significant effect of surface treatment on WCA and also a significant interaction between material and surface treatment. WCA significantly increased after TC for CLP and PK applied materials, resulting in more hydrophobic surfaces. WCA decreased in sealant applied CV whereas increased for M-PM after TC. Although water sorption was not evaluated, it was previously reported to occur with sealants and the surface of the sealant may have changed after TC in different levels on tested materials. Whether the sealant surface becomes hydrophobic or hydrophilic after TC needs to be evaluated in detail in future studies. The WCA findings of the present study are in line with the results found by Zissis et al. (2001) and Al-Dwairi et al. (2019). Two of the materials (a heat polymerized PMMA and the CAD-CAM PMMA AvaDent) evaluated in the study by Al-Dwairi et al. (2019) were also compared in the present study and the values reported for the WCA were similar. Higher WCA values are related to higher hydrophobicity or lower surface free energy (Absolom et al., 1983). An explanation for this may be the presence of less residual monomer as the CAD-CAM PMMAs are manufactured under high pressure and temperature, which alter the polarity of the molecules changing the wettability properties (Arslan et al., 2018; Liebermann et al., 2016; Munchow et al., 2014).

A significant interaction was found between material and surface treatment, and CLP and SSC, in general, resulted in lower SR values, which were all below commonly used plaque accumulation threshold of 0.2 μm. Some PK-applied groups had values slightly higher than 0.2 μm. Stereomicroscope images also supported this finding. PK resulted in more surface irregularities with more defects and scratches, whereas CLP- or SSC-applied materials had smoother surface morphology. Sealant preserved its integrity on all tested materials after TC. In addition, when after TC was compared with before TC, significant differences in SR were found for only 2 material-surface treatment combinations. The present study findings are partially in agreement with those of Arslan et al. (2018), Ayaz et al. (2015) and Wieckiewicz

Table 3
Water contact angle (°). Significant differences among surface treatment groups of the same material are indicated with different uppercase superscript letters in the same column for before and after thermocycling, independently. Significant differences between before and thermocycling groups of same surface treatment group of the same material are indicated with different symbols in the same row. ST, surface treatment; SD, standard deviation; TC, thermocycling; CLP, Conventional laboratory polishing; PK, Acrylic Polisher kit; SSC, Surface sealant coupling.

Material	ST	Mean ± SD Before TC	Mean ± SD After TC	Pairs	*P* Before TC	*P* After TC	*P* Before- After TC
Avadent (AV)	CLP	61.18 ± 0.61**A,** *	69.88 ± 2.49**L,** †	CLP-PK	.259	.802	<.001
				CLP-SSC	.003	<.001	
	PK	59.64 ± 2.46**A,** *	69.13 ± 1.89**L,** †	PK-CLP	.259	.802	<.001
				PK-SSC	<.001	<.001	
	SSC	64.92 ± 1.71**B,** *	63.24 ± 2.25**M,** *	SSC-CLP	.003	<.001	.087
				SSC-PK	<.001	<.001	
Conventional (CV)	CLP	60.21 ± 1.11**C,** *	71.94 ± 1.94**N,** †	CLP-PK	.002	.145	<.001
				CLP-SSC	<.001	<.001	
	PK	62.40 ± 0.61**D,** *	68.57 ± 2.19**N,** †	PK-CLP	.002	.145	<.001
				PK-SSC	<.001	.002	
	SSC	66.51 ± 1.22**E,** *	61.71 ± 4.67**O,** †	SSC-CLP	<.001	<.001	.019
				SSC-PK	<.001	.002	
Merz M-PM Disc (M-PM)	CLP	51.93 ± 1.49**E,** *	67.79 ± 2.39**P,** †	CLP-PK	<.001	.994	<.001
				CLP-SSC	<.001	.009	
	PK	55.89 ± 1.16**F,** *	67.90 ± 1.42**P,** †	PK-CLP	<.001	.994	<.001
				PK-SSC	.001	.007	
	SSC	58.63 ± 0.90**G,** *	64.16 ± 2.08**R,** †	SSC-CLP	<.001	.009	.002
				SSC-PK	.001	.007	
Polident (Poli)	CLP	53.67 ± 1.06**H,** *	67.56 ± 1.75**RS,** †	CLP-PK	.001	.437	<.001
				CLP-SSC	<.001	.058	
	PK	57.99 ± 2.52**J,** *	69.10 ± 0.79**R,** †	PK-CLP	.001	.437	<.001
				PK-SSC	.001	.004	
	SSC	61.94 ± 1.20**K,** *	64.51 ± 3.49**S,** *	SSC-CLP	<.001	.058	.089
				SSC-PK	.001	.004	

Table 4
Surface roughness data (Ra; μm). Significant differences among surface treatment groups of the same material are indicated with different uppercase superscript letters in the same column for before and after thermocycling, independently. Significant differences between before and thermocycling groups of same surface treatment group of the same material are indicated with different symbols in the same row. Abbreviations are as shown in Table 3.

Material	ST	Mean ± SD Before TC	Mean ± SD After TC	Pairs	*P* Before TC	*P* After TC	*P* Before- After TC
Avadent (AV)	CLP	0.14 ± 0.05**AB,** *	0.13 ± 0.04**K,** *	CLP-PK	.088	.009	.765
				CLP-SSC	.904	.966	
	PK	0.22 ± 0.08**A,** *	0.23 ± 0.07**L,** *	PK-CLP	.088	.009	.610
				PK-SSC	.038	.005	
	SSC	0.13 ± 0.04**B,** *	0.13 ± 0.03**K,** *	SSC-CLP	.904	.996	.848
				SSC-PK	.038	.005	
Conventional (CV)	CLP	0.06 ± 0.02C,*	0.12 ± 0.07**M,** *	CLP-PK	<.001	.306	.107
				CLP-SSC	.09	.981	
	PK	0.15 ± 0.05D,*	0.17 ± 0.04**M,** *	PK-CLP	<.001	.306	.336
				PK-SSC	.037	.231	
	SSC	0.10 ± 0.03C,*	0.12 ± 0.04**M,** *	SSC-CLP	.09	.981	.286
				SSC-PK	.037	.231	
Merz M-PM Disc (M-PM)	CLP	0.06 ± 0.01**E,** *	0.07 ± 0.01**N,** †	CLP-PK	<.001	<.001	.027
				CLP-SSC	.014	.017	
	PK	0.25 ± 0.04**F,** *	0.24 ± 0.07**O,** *	PK-CLP	<.001	<.001	.728
				PK-SSC	<.001	.005	
	SSC	0.11 ± 0.03**G,** *	0.15 ± 0.04**P,** *	SSC-CLP	.014	.017	.095
				SSC-PK	<.001	.005	
Polident (Poli)	CLP	0.07 ± 0.02**H,***	0.08 ± 0.02 **R,** *	CLP-PK	.002	<.001	.092
				CLP-SSC	.03	.519	
	PK	0.17 ± 0.04**J.** *	0.19 ± 0.04**S,** †	PK-CLP	.002	<.001	.041
				PK-SSC	.393	.001	
	SSC	0.14 ± 0.06**J,** *	0.10 ± 0.04**R,** *	SSC-CLP	.03	.519	.238
				SSC-PK	.393	.001	

et al. (2014), who reported no significant changes in R_a values after TC. Al-Dwairi et al. (2019) reported that CAD-CAM PMMAs had the lowest mean surface roughness value of 0.12 ± 0.02 μm. Arslan et al. (2018) reported R_a values, similar to the present study, and no significant changes in SR after TC. Murat et al. (2019) found significantly lower R_a values for CAD-CAM PMMA than in conventional PMMA.

There is no standardized thermal cycling protocol and there is a wide range of temperature extremes, transfer times between baths and dwell times (Morresi et al., 2014; Gale and Darvell, 1999). Moore et al. (1999) reported that sublingual temperature is routinely used as an indicator of oral temperature, and when measured under specific conditions, it approximates 37 °C for most individuals. According to ISO standard (ISO 11405), the majority of studies reported temperatures of 5 °C and 55 °C to test dental materials, considering these values as the closest to the physiology of the oral cavity (Morresi et al., 2014). Therefore, in the present study 5 °C/55 °C was used. Dwell time is the period of time that the specimen is immersed in a bath of a particular temperature to correspond to a latency period. The choice of dwell time shows a great variability in different studies. Amaral et al. (2007) suggested that patients would not tolerate direct contact of a vital tooth with extremely hot or cold substances for extended period of time. Several studies used 30 s dwell time, which may simulate more faithfully the abrupt changes

Table 5
Microhardness data. Significant differences among surface treatment groups of the same material are indicated with different uppercase superscript letters in the same column for before and after thermocycling, independently. Significant differences between before and thermocycling groups of same surface treatment group of the same material are indicated with different symbols in the same row. Abbreviations are as shown in Table 3.

Material	ST	Mean ± SD Before TC	Mean ± SD After TC	Pairs	*P* Before TC	*P* After TC	*P* Before- After TC
Avadent (AV)	CLP	16.51 ± 2.39**A**, *	11.28 ± 0.71**G**, †	CLP-PK	<.001	<.001	.001
				CLP-SSC	<.001	<.001	
	PK	12.55 ± 0.91**B**, *	9.81 ± 0.32**H**, †	PK-CLP	<.001	<.001	<.001
				PK-SSC	.089	.997	
	SSC	10.7 ± 0.75**B**, *	9.79 ± 0.48**H**, *	SSC-CLP	<.001	<.001	.070
				SSC-PK	.089	.997	
Conventional (CV)	CLP	11.74 ± 1.59**C**,*	10.18 ± 0.66**J**, †	CLP-PK	.243	.985	.033
				CLP-SSC	.747	.016	
	PK	13.42 ± 2.55**C**, *	10.24 ± 0.35**J**, †	PK-CLP	.243	.985	.023
				PK-SSC	.624	.022	
	SSC	12.48 ± 1.29**C**, *	11.32 ± 0.93**K**, *	SSC-CLP	.747	.016	.124
				SSC-PK	.624	.022	
Merz M-PM Disc (M-PM)	CLP	17.86 ± 2.10**D**, *	13.36 ± 0.68**L**, †	CLP-PK	.878	.472	.003
				CLP-SSC	.231	.006	
	PK	17.44 ± 1.46**D**, *	12.94 ± 0.87**LM**, †	PK-CLP	.878	.472	.001
				PK-SSC	.460	.075	
	SSC	16.40 ± 1.05**D**, *	12.13 ± 0.23**M**, †	SSC-CLP	.231	.006	<.001
				SSC-PK	.460	.075	
Polident (Poli)	CLP	20.80 ± 0.93**E**, *	13.74 ± 0.47**N**, †	CLP-PK	<.001	<.001	<.001
				CLP-SSC	<.001	<.001	
	PK	14.02 ± 3.64**F**, *	11.98 ± 0.71**O**, *	PK-CLP	<.001	<.001	.162
				PK-SSC	.08	.006	
	SSC	11.30 ± 0.64**F**, *	11.03 ± 0.16**P**, *	SSC-CLP	<.001	<.001	.349
				SSC-PK	.08	.006	

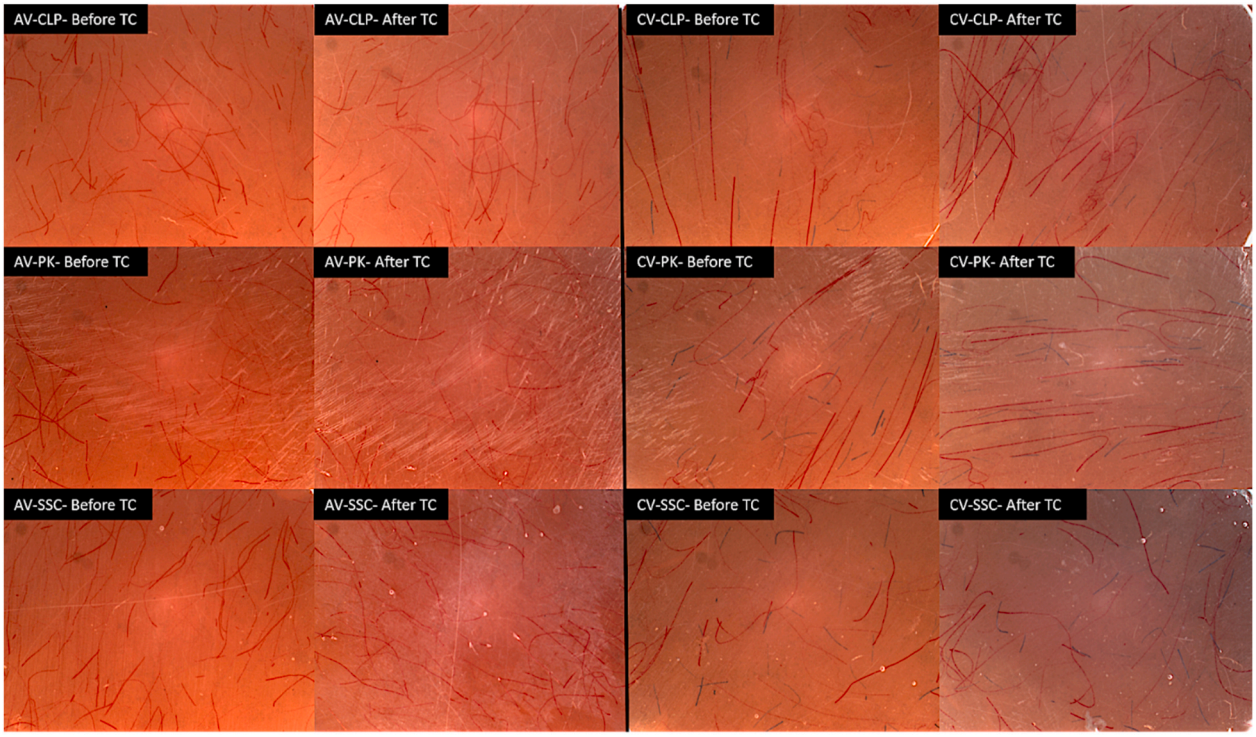

Fig. 1. Stereomicroscopic images of AV CAD-CAM PMMA and conventional heat-polymerized PMMA with different surface treatments. Abbreviations are shown in Table 3.

of temperature that occur in the oral cavity. Therefore, in the present study, 30 s dwell time was also used. The number of cycles used in experimental studies published in the last 15 years, have ranged between 100 cycles and 100.000 cycles (Morresi et al., 2014). In previous studies (Goiato et al., 2011; Goiato et al., 2013a,b), 2000 thermocycling (5 and 55 °C) in distilled water was performed to simulate 2 years of clinical wear of denture base materials. However, Li et al. (2021) applied 5000 thermal cycles (5 °C and 55 °C water) to 3D printed denture base materials to correspond 5 years of temperature changes in the oral environment. In addition, Andrade de Freitas et al. (2018) and Chaves et al. (2009) also applied 5000 thermocycling to denture base materials to correspond 5 years of clinical use. Therefore, 5000 cycles were used in the present study to simulate 5 years of physiological aging.

Previous studies assessed the effects of different polishing techniques on SR (Gungor et al., 2014; Kuhar and Funduk, 2005; Al-Rifaiy, 2010). Şahin et al. (2016) assessed the SR of conventionally polished and

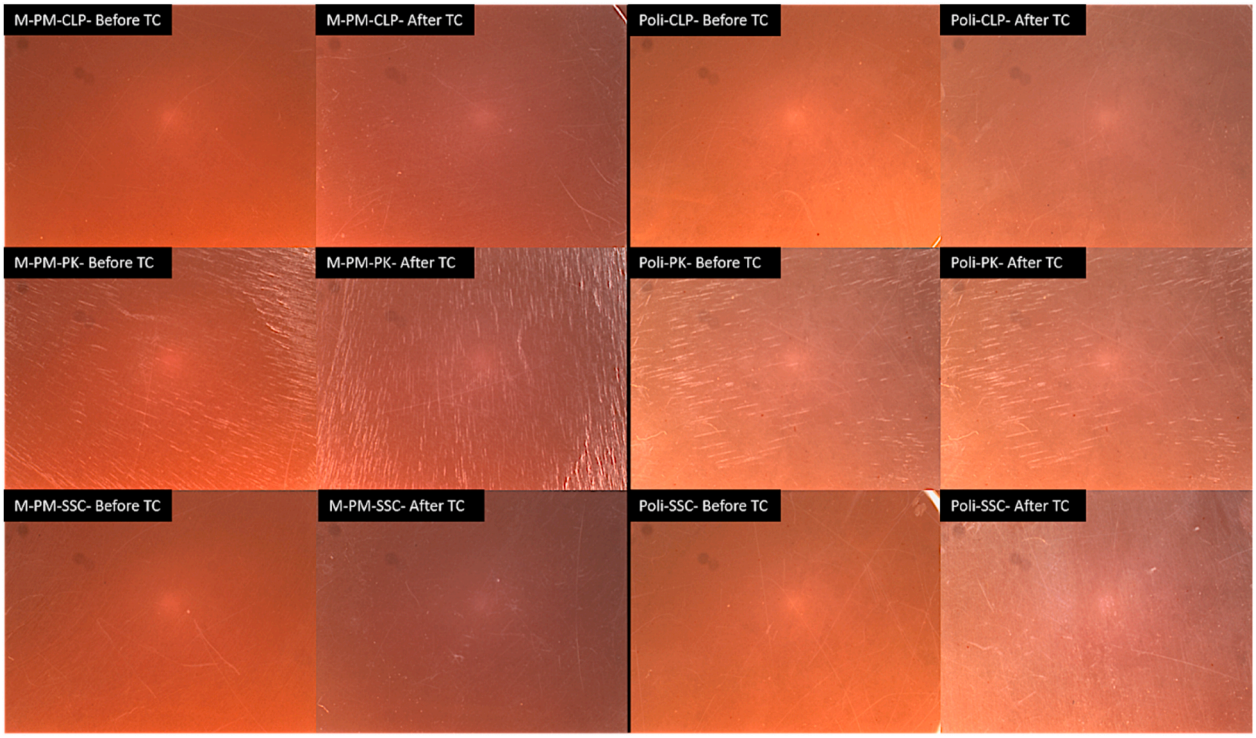

Fig. 2. Stereomicroscopic images of M-PM CAD-CAM PMMA and Poli CAD-CAM PMMA with different surface treatments. Abbreviations are as shown in Table 3.

sealant applied (Palaseal, Optiglaze, Biscover) denture base materials and did not find significant differences between them, yet the R_a values observed were over the plaque accumulation threshold (0.2 μm). Inconsistent results among studies might be due to the variations in chemical configuration and microstructure of different resins tested, which influence the material's surface properties (Liebermann et al., 2016).

In the present study, for MH, a significant interaction between the material and the surface treatment was found, and after TC, a decrease in all groups was observed some being significant. Similar results were recorded by Goiato et al. (2016), where also a reduction in hardness after TC was observed in conventional PMMA. Schoeffel et al. (2019) also evaluated the MH of PMMA materials using the same Knoop microhardness test with the same load used in the present study, and also observed a reduction in hardness after TC in the conventional PMMA group with similar values as in the present study. Al-Dwairi et al. (2019) also used a MH tester, as in the present study, but with different loads; and found higher surface hardness in the CAD-CAM PMMA group than their conventional heat-polymerized counterparts. Decrease in hardness after TC may be attributed to the penetration of water molecules into the resin, which leads to resin expansion and therefore a polymeric matrix degradation through hydroxylation that can increase the possibility of fracture and diminish the longevity of the denture base (Assuncao et al., 2010; Barbosa et al., 2008; Farina et al., 2012; Goiato et al., 2016).

PMMA is prone to water absorption because of its molecular polarity. Water absorption occurs through a diffusion mechanism among the polymer chains and may damage the bonding of the polymer network (Al-Rifaiy, 2010). TC provides repeated sorption/desorption cycles which may result in microfractures in PMMA matrix (Amin et al., 2014). Low solubility of denture base materials is desired for less monomer release (Cucci et al., 1998), however, unreacted monomers and water-soluble additives may leach out with TC. The changes in physical properties after TC in the present study may be due to these effects. Additionally, TC is known to generate a hydrolytic stress, affect the water sorption, and damage the surface integrity of the sealant materials. This mechanism may result in swelling and debonding of the sealant material (Cakmak et al., 2020). In this respect, no debonding was observed on sealant applied specimens after TC in the present study which indicates a potentially high bond strength. When the findings are considered overall, CLP or SSC can be recommended for tested PMMAs as the surfaces were smoother, CLP enabled high MH in general, and SSC-applied PMMAs maintained their MH.

The present study has limitations as it is an in vitro study, which did not completely simulate in vivo conditions, and the results are limited to the CAD-CAM materials and the surface treatment techniques evaluated. Moreover, no microbial adhesion, water sorption assessment or further mechanical measurements were performed, and the surfaces were evaluated with stereomicroscope images instead of scanning electron microscope. However, an attempt to simulate the clinical environment was made with TC, where materials were exposed to thermal stress by alternating temperature. Future studies are needed to evaluate the performance of new CAD-CAM PMMAs and determine the most appropriate surface treatment that leads to the optimal surface properties.

5. Conclusion

1. Thermocycling affected the surface roughness, microhardness, and water contact angle of tested CAD-CAM PMMAs.
2. Surface treatment's and thermocycling's combined effect on surface properties depended on PMMA material. After thermocycling, surface treatment had a significant effect on water contact angle and surface roughness; CLP and PK application resulted in hydrophobic surfaces on all tested PMMAs. CLP and SSC application provided smoother surfaces on CAD-CAM PMMAs after thermocycling. Whereas, PK resulted in rougher surfaces both before and after thermocycling.
3. Thermocycling lowered the microhardness of all PMMAs, and the decrease was significant in CLP- or PK-applied PMMAs, except for PK-applied Poli.

Author contribution statement

Sevda Atalay: Conceptualization, data curation, data collection, investigation, methodology, review and edited article.

Gülce Çakmak: Conceptualization, data curation, investigation, methodology, drafting article, critical revision of article.

Manrique Fonseca: Writing, drafting & editing article.

Martin Schimmel: Manuscript's critical revision and approval.

Burak Yilmaz: Conceptualization, data analysis, statistical analysis, data interpretation, review, edit and critical revision of the article.

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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