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RESEARCH AND EDUCATION

Effect of coffee thermocycling on the surface roughness and stainability of nanographene-reinforced polymethyl methacrylate used for fixed definitive prostheses

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ABSTRACT

Statement of problem. A nanographene-reinforced polymethyl methacrylate (PMMA) has been introduced for definitive prostheses. However, knowledge on the surface roughness and stainability of this material is lacking.

Purpose. The purpose of this in vitro study was to compare the surface roughness and stainability of nanographene-reinforced PMMA with those of a prepolymerized PMMA and a reinforced composite resin after coffee thermocycling.

Material and methods. Disk-shaped specimens (Ø10×1.5-mm) were prepared from 3 different A1-shade millable resins (prepolymerized PMMA [M-PM; PMMA]; nanographene-reinforced PMMA [G-CAM; G-PMMA]; reinforced composite resin [Brilliant Crios; RCR]). Surface roughness (R_a) values were measured before and after conventional polishing by using a noncontact profilometer. Initial color coordinates were measured over a gray background with a spectrophotometer after polishing. Specimens were then thermocycled in coffee for 5000 cycles. Measurements were repeated after coffee thermocycling, and color differences (ΔE_{00}) were calculated. R_a values among different time intervals were analyzed by using either the Friedman and Dunn tests (RCR) or repeated measures analysis of variance (ANOVA) and Bonferroni corrected paired samples *t* tests (PMMA and G-PMMA), while R_a values within a time interval were analyzed by using either the Kruskal-Wallis and Dunn tests (before polishing) or 1-way ANOVA and Tukey HSD (after polishing) or Tamhane T2 tests (after coffee thermocycling). ΔE_{00} values were analyzed by using 1-way ANOVA and Tukey HSD tests, while color coordinates of the specimens after polishing and after coffee thermocycling were compared by using paired samples *t* tests (α=.05).

Results. All materials had their highest R_a values before polishing ($P \leq .011$), while differences after polishing and after coffee thermocycling values were nonsignificant ($P \geq .140$). PMMA had higher R_a than RCR before polishing (P=.002), and RCR had higher values than G-PMMA after polishing and after coffee thermocycling ($P \leq .023$). RCR had the highest ΔE_{00} (P<.001). Polishing increased the b* values of PMMA, and coffee thermocycling increased the a* values of G-PMMA and all values of RCR ($P \leq .012$).

Conclusions. The tested materials had similar and acceptable surface roughness after polishing. The surface roughness of materials was not affected by coffee thermocycling. Considering the reported color thresholds, all materials had acceptable color change, but the computer-aided design and computer-aided manufacturing composite resin had perceptible color change after coffee thermocycling. (J Prosthet Dent 2023;■:■-■)

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Clinical Implications

G-PMMA may be a suitable alternative to millable composite resin for patients with high coffee consumption because of its favorable surface roughness and stainability after polishing.

Polymethyl methacrylate (PMMA) is a commonly used dental material for the fabrication of removable or interim fixed prostheses because of its biocompatibility, ease of processing, and low cost.[1,2] However, conventionally processed PMMA has disadvantages,[3] and, along with the advancements in computer-aided design and computer-aided manufacturing (CAD-CAM), prepolymerized PMMA disks have become available.[4,5] Even though prepolymerized PMMA has a similar chemical composition to that of conventional PMMA,[3] the high temperature and pressure processing leads to improved mechanical properties.[6] CAD-CAM technology also enables the subtractive fabrication of composite resins that can be used for definitive prostheses.

The incorporation of different reinforcing agents to improve the mechanical properties of PMMA has been reported.[7] Nanotechnology has enabled the integration of graphene, a crystalline form of carbon,[8] as a reinforcement phase in PMMA.[9] Recently, a nanographene-reinforced prepolymerized PMMA (G-CAM; Graphenano DENTAL SL) has been introduced for definitive prostheses.[10] Although properties of nanographene-reinforced PMMA have been reported,[1,8,9,11-14] studies that compare this material with similar materials marketed for definitive prostheses are sparse.[9,11]

Surface texture and stainability are essential properties for esthetic definitive restorative materials.[15-17] A clinical surface roughness threshold of 0.2 μm for acceptable bacterial plaque accumulation has been reported.[3-5,18] The addition of nanoparticles may affect the physical properties of resins, including surface roughness, particularly when the susceptibility of resin-based materials to temperature changes and water sorption are considered.[16,19] How intraoral temperature changes caused by beverages effect the surface roughness and stainability of nanographene-reinforced CAD-CAM PMMA is not well known. Therefore, the present study aimed to investigate the surface roughness and stainability of nanographene-reinforced PMMA, compared with those of commonly used prepolymerized PMMA and reinforced composite resins after coffee thermocycling. The null hypotheses were that material type and time interval (polishing and coffee thermocycling) would not affect the surface roughness or stainability of CAD-CAM resins.

MATERIAL AND METHODS

A Ø10-mm cylinder was designed in standard tessellation language (STL) format with a modeling software program (Meshmixer v3.5.474; Autodesk Inc), and the STL file was transferred into a 5-axis milling unit (Milling unit M1; Zirkonzahn) to fabricate cylinder-shaped specimens from 3 different A1-shaded CAD-CAM resins (prepolymerized PMMA [M-PM; Merz Dental, PMMA]; nanographene-reinforced PMMA [G-CAM; Graphenano DENTAL SL, G-PMMA]; reinforced composite resin [Brilliant Crios; Coltène AG, RCR]). These cylinders were wet-sliced with a precision cutter (Vari/cut VC-50; Leco Corp) and a diamond-wafering blade (Buehler series 15 LC diamond; Microstructural Analyses Division) to obtain 10 specimens from each material with dimensions of Ø10×1.5-mm. The number of specimens in each group was determined by a priori power analysis (1-β=80% and α=.05) based on the results of a pilot study on the effect of polishing and coffee thermocycling on the surface roughness and stainability of polymers; 10 specimens per material were deemed sufficient to detect an effect size of 0.1 (R v3.6.1; R Core Team 2021).

Each specimen was ground (Waterproof SIC, US 280, US 360, US 1000; Struers) under running water to obtain uniform and smooth surfaces. The final thicknesses (1.5 ±0.03 mm) were controlled with digital calipers (Model number NB60; Mitutoyo American Corp), and all specimens were ultrasonically cleaned in distilled water for 10 minutes (Eltrosonic Ultracleaner 07-08; Eltrosonic GmbH). Initial surface roughness (R_a in μm) measurements were performed by using a noncontact optical profilometer (FRT MicroProf 100, equipped with a CWL 300 μm sensor; Fries Research and Technology GmbH), which had a 3-nm resolution in the z-axis.[20] The R_a of each specimen was measured along 6 lines (3 horizontal and 3 vertical) that were 1 mm apart and these values were averaged (Mark III; Fries Research & Technology GmbH).[20] Each trace had a length of 5.5 mm and a pixel density of 5501 point/line. After initial measurements, the PMMA and G-PMMA specimens were conventionally polished with pumice slurry (Pumice fine; Benco Dental) for 90 seconds at 1500 rpm, which was followed by fine polishing with a paste (Fabulustre; Grobet USA) for 90 seconds.[21] Specimens of the RCR group were polished for 60 seconds with 2 different silicone polishers (Diatech Lab finishing and polishing kit for BRILLIANT Crios; Coltène AG), which was followed by fine polishing with a textile polisher for 60 seconds. After polishing, all specimens were ultrasonically cleaned, and the R_a measurements were repeated.

Initial color coordinates (L*, a*, and b*) were measured over a gray background by using a digital spectrophotometer (CM-26d; Konica Minolta) with an 8-mm

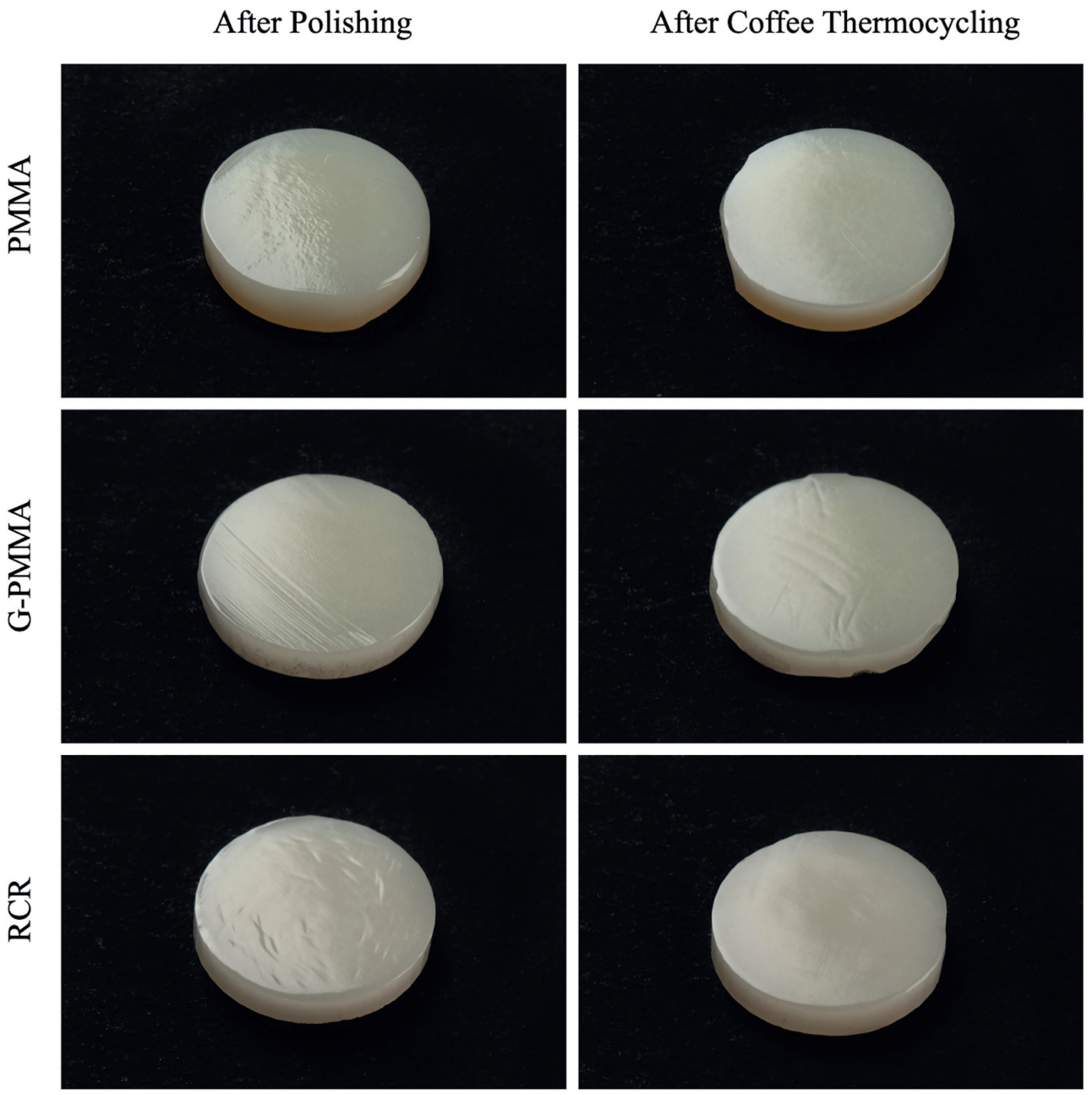

Figure 1. Representative images of specimens from each group before and after coffee thermocycling. PMMA, polymethyl methacrylate; G-PMMA, graphene-reinforced polymethyl methacrylate; RCR, reinforced composite resin.

aperture and that used the Commission International de l'Eclairage Standard (CIE) (2-degree) human observer characteristics and CIE D65 illuminant for its readings.[18,20] All measurements were performed in a temperature- and humidity-controlled room with daylight by the same operator (K.H.). The spectrophotometer was calibrated according to the manufacturer's recommendations before measuring each group. A saturated sucrose solution was used for the optical contact between the specimens and the background. Three measurements were recorded and averaged for each specimen.[18,20]

After color measurements, each specimen was subjected to 5000 cycles of coffee thermocycling (SD Mechatronik Thermocycler; SD Meachtronik GmbH) at 5 °C-55 °C with a dwell time of 30 seconds and a transfer time of 10 seconds, which corresponded to approximately 6 months of intraoral use.[16,22] The coffee solution was freshly prepared after every 12 hours at a ratio of 1 tablespoon of coffee (Intenso Roasted and Grounded; Kaffeehof GmbH) to 177 mL of water. After coffee thermocycling, specimens were brushed 10 times with toothpaste (Colgate Total Pro Breath Health; Colgate-

Table 1. Descriptive statistics of R_a (μm) values for each material-time interval pair

	Before Polishing		After Polishing		After Coffee Thermocycling	
	Mean ±SD	Median (min - max)	Mean ±SD	Median (min - max)	Mean ±SD	Median (min - max)
PMMA	0.38 ±0.05^{a}	0.38^{B} (0.32 - 0.49)	0.10 ±0.03^{bAB}	0.09 (0.06 - 0.13)	0.16 ±0.08^{bAB}	0.18 (0.06 - 0.3)
G-PMMA	0.33 ±0.05^{a}	0.31^{AB} (0.27 - 0.40)	0.08 ±0.02^{bA}	0.07 (0.06 - 0.11)	0.10 ±0.03^{bA}	0.09 (0.07 - 0.14)
RCR	0.33 ±0.16	0.28^{aA} (0.23 - 0.78)	0.11 ±0.01^{B}	0.11^{b} (0.09 - 0.13)	0.12 ±0.01^{B}	0.12^{b} (0.10 - 0.14)

G-PMMA, nanographene-reinforced PMMA; PMMA, polymethyl methacrylate; R_a, surface roughness; RCR, reinforced composite resin; SD, standard deviation. * Uppercase letters indicate differences among materials within each time interval and lowercase letters indicate differences among time intervals within each material (P<.05).

Palmolive) under running water to remove coffee stains and ultrasonically cleaned in distilled water for 10 minutes (Fig. 1). R_a and color coordinate measurements were repeated. Before each set of R_a and color coordinate measurements, the specimens were randomized by using the randomize function of a software program (Excel; Microsoft Corp). Color differences (ΔE_{00}) caused by coffee thermocycling were calculated by using the CIEDE2000 formula, where the parametric factors were set as 1.[15-17]

Distribution of data was analyzed by using Shapiro-Wilk tests. Because the data were not normally distributed, the Friedman and Dunn tests were used to analyze the R_a values of RCR among different time intervals. However, the R_a data of PMMA and G-PMMA showed normal distribution; thus, repeated measures analysis of variance (ANOVA) and Bonferroni corrected paired samples t tests were used to evaluate the differences in R_a values among different time intervals within those groups. Comparison of R_a values within a time interval was performed either by using the Kruskal-Wallis and Dunn tests (before polishing) or 1-way ANOVA and Tukey HSD (after polishing) or Tamhane T2 tests (after coffee thermocycling). One-way ANOVA and Tukey HSD tests were used to analyze the ΔE_{00} values of materials, while paired samples t tests were used to evaluate the difference in the color coordinates of each material between after polishing and after coffee thermocycling. All statistical analyses were performed by using a statistical software program (IBM SPSS Statistics, v23; IBM Corp) (α=.05). The perceptibility and acceptability of the ΔE00 values were further evaluated according to the thresholds set by a previous study (perceptibility: 0.8 units, acceptability: 1.8 units).[23]

RESULTS

Repeated measures ANOVA revealed significant differences among time intervals for PMMA and G-PMMA (P<.001). Both materials had the highest R_a before polishing (P<.001), while the differences between after polishing and after coffee thermocycling values were not significant (P≥.140). For RCR, significant differences were observed among time intervals (P<.001), as it had the highest R_a before polishing (P≤.011), while, after polishing and after coffee thermocycling, R_a values were similar (P>.05). The tested materials had different R_a values within each time interval (P≤.046). Before polishing, PMMA had higher values than RCR (P=.002), while G-PMMA had R_a values similar to those of the other materials (P≥.113). R_a values after polishing and after coffee thermocycling revealed that RCR had higher values than G-PMMA (P≤.023), while PMMA had values similar to other materials (P≥.120) (Table 1).

Table 2. Mean ±standard deviation color coordinates and ΔE_{00} values of each material

		After Polishing	After Coffee Thermocycling	ΔE_{00}
PMMA	L*	63.13 ±0.34^{a}	63.01 ±0.24^{a}	0.34 ±0.10^{A}
	a*	−4.48 ±0.06^{a}	−4.48 ±0.03^{a}	
	b*	0.79 ±0.16^{a}	0.50 ±0.10^{b}	
G-PMMA	L*	60.30 ±0.31^{a}	60.43 ±0.35^{a}	0.31 ±0.22^{A}
	a*	−3.43 ±0.03^{a}	−3.38 ±0.03^{b}	
	b*	2.71 ±0.21^{a}	2.63 ±0.16^{a}	
RCR	L*	60.60 ±0.29^{a}	60.84 ±0.29^{b}	0.9 ±0.21^{B}
	a*	−2.48 ±0.04^{a}	−1.99 ±0.05^{b}	
	b*	1.34 ±0.21^{a}	1.88 ±0.20^{b}	

G-PMMA, nanographene-reinforced PMMA; PMMA, polymethyl methacrylate; RCR, reinforced composite resin. *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<.05).

RCR had the highest ΔE_{00} (P<.001), while PMMA and G-PMMA had similar values (P=.381). PMMA had higher b* values after polishing (P<.001), while G-PMMA had higher a* values after coffee thermocycling (P<.001). All color coordinates of RCR were higher after coffee thermocycling (P≤.012) (Table 2).

DISCUSSION

The Ra values of the tested materials had significant differences within the different time intervals. Each material had its highest Ra before polishing. Therefore, the first null hypothesis was rejected.

Even though none of the tested materials had Ra values similar to or lower than clinically acceptable threshold before polishing, all materials had acceptable Ra after polishing. In addition, coffee thermocycling did

not increase the Ra values of the tested materials above 0.2 μm, consistent with previous studies on polymer-based materials.[3-5,21] Therefore, the tested materials appear to be resistant to long-term coffee exposure considering that 5000 cycles correspond to 6 months of intraoral service.[16,22] The inclusion of nanoparticles into the PMMA structure did not significantly affect the surface roughness of PMMA. However, coffee thermocycling may affect other mechanical properties of a denture base material such as flexural strength and hardness given that this process leads to water sorption into denture base resins that deteriorate polymeric chains.[19] In addition, longer coffee thermocycling durations may lead to different results; therefore, these results should be substantiated with studies on bacterial plaque accumulation and other mechanical properties of the tested materials after coffee thermocycling with an increased number of cycles.

Studies on the mechanical properties of G-PMMA have reported contradicting results.[1,8-14] Di Carlo et al[8] reported that G-PMMA had higher flexural strength and elastic modulus compared with conventional PMMA. Ionescu et al[14] compared different mechanical properties of G-PMMA with prepolymerized PMMA and have concluded that G-PMMA had higher tensile strength, flexural strength, and elastic modulus along with lower water sorption, solubility, and methylmethacrylate elution. Both materials were also reported to have similar hardness, consistent with other studies.[9,11,13] Similar to the results of the present study, Ionescu et al[14] reported that prepolymerized PMMA and G-PMMA had similar Ra, regardless of aging. However, ethanol aging increased the Ra and decreased the microhardness of both materials. This difference may be attributed to the aging process, as Hernández et al[1] reported that the microhardness of G-PMMA did not change after thermocycling. In another study, the biaxial flexural strengths of the G-PMMA, PMMA, resin nanoceramic, and polymer-infiltrated ceramic network were reported to be similar.[9] However, a recent study concluded that polyetheretherketone (PEEK) had higher retentive forces and less deformation than G-PMMA when designed as a removable denture clasp.[12] The authors suggested that this difference was because PEEK is more ductile than PMMA,[24,25] and possibly than G-PMMA, given its reported flexural strength values are similar to those of PMMA.[9,14] Nevertheless, these results should be interpreted carefully as the authors are unaware of studies on the clinical performance of G-PMMA, which should be further investigated.

The second null hypothesis was also rejected, as RCR had the highest ΔE_{00} among the tested materials. In addition, RCR was the only material that had a perceptible color change (ΔE_{00}=0.9 units) when the reported thresholds[23] were considered. However, none of the materials tested had a ΔE_{00} value greater than 1.8, which can be interpreted as tested materials having acceptable stainability after long-term coffee consumption. The addition of nanoparticles in PMMA (G-PMMA) did not lead to a significant difference in stainability compared with nonreinforced PMMA. The authors are unaware of a previous study on the stainability of G-PMMA. However, its translucency has been investigated recently,[9] and it was reported to be similar to that of lithium disilicate-reinforced glass ceramic of 0.5 mm and 0.75 mm thicknesses.

Coffee thermocycling significantly reduced the yellowness (b* values) of PMMA, whereas a significant increase was observed in the redness (a* values) of G-PMMA. When the color coordinates of RCR were considered, a significant increase was observed in each coordinate after coffee thermocycling. The higher susceptibility of RCR to discoloration may be associated with its chemical composition, as it comprises barium glass, amorphous silica, and inorganic pigments in a resin matrix,[26] which might be more heterogeneous than PMMA and G-PMMA. Even though the tested PMMA comprises 98% PMMA,[27] the manufacturer of G-PMMA has not disclosed its chemical composition. Therefore, future studies on the chemical composition of tested materials are needed to support this hypothesis. The higher Ra of RCR compared with G-PMMA after coffee thermocycling may also be associated with its higher susceptibility to discoloration. Nevertheless, these results should be interpreted cautiously given the fact that the changes in color coordinates were not beyond the clinically perceptible and acceptable threshold values.

Previous studies on the Ra and the optical properties of polymers have used the noncontact optical profilometer and spectrophotometer used in the present study.[18,20,21] However, different instruments may lead to different results. Coffee thermocycling could not simulate the clinical situation completely, as saliva was not involved in the process and both surfaces of the specimens were subjected to coffee thermocycling. However, clinically, only polished surfaces are exposed to staining and lower color changes may be seen. Coffee was selected as the staining liquid in the present study as it accelerates discoloration due to its acidic components;[18] still, different staining solutions may lead to different results.[2] Another limitation of the present study was that G-PMMA was only compared with subtractively manufactured materials, and future studies should test additively manufactured materials. Future studies should also investigate the different mechanical and optical properties of G-PMMA after long-term brushing, thermomechanical aging, and discoloration with other solutions such as red wine and tea.

CONCLUSIONS

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

1. None of the tested materials had acceptable surface roughness before polishing when the reported clinical threshold was considered. However, polishing reduced the roughness of all materials to below 0.2 μm.
2. The surface roughness of tested materials was not significantly affected by coffee thermocycling.
3. Where reported color difference threshold values were concerned, only RCR had a perceptible color change after coffee thermocycling; all materials had acceptable stainability.

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Gülce Çakmak: Conceptualization, Methodology, Investigation. **Kira Vera Herren:** Methodology, Investigation. **Mustafa Borga Donmez:** Writing – original draft. **Çiğdem Kahveci:** Formal analysis. **Martin Schimmel:** Conceptualization, Supervision. **Burak Yilmaz:** Conceptualization, Methodology, Supervision, Writing – review & editing.

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