

# The effect of thermal cycling on the surface roughness of nano hybrid and high-viscosity bulk-fill resin-based composites

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## ABSTRACT

**Introduction:** The aim of this study was to investigate the effect of thermal cycling on the surface geometry of high-viscosity bulk-fill resin-based composites (RBCs) compared to conventional nano hybrid composites.

**Materials and methods:** Four conventional nano hybrid composites (Tetric EvoCeram – TEC, GrandioSO – GD, Filtek Z550 – FZ, and Ceram-X Mono – CX) and 4 high-viscosity bulk-fill composites (Tetric EvoCeram Bulk Fill – TBF, X-tra fil – XF, Filtek Bulk Fill Posterior – FBF, and QuixFil – QF) were tested. After the 2-step polishing procedure, the samples were divided into 2 groups: control group (K) and thermal cycling group (TC). Samples from the TC were subjected to thermal cycling according to ISO 11405 (THE-1100, SD Mechatronik GmbH). Surface geometry was evaluated by profilometry (Turbowave v. 7.36, Hommel-Etamic) and scanning electron microscope – SEM (VEGA 3, Tescan Analytics).

**Results:** The applied thermal cycles increased the surface roughness of conventional RBCs: TEC ( $p = 0.000007$ ), GD ( $p = 0.04$ ), and CX ( $p = 0.0005$ ). A reduction in the surface roughness of bulk-fill composites was observed in the case of materials: XF ( $p = 0.000003$ ) and QF ( $p = 0.0002$ ). Thermal cycling was shown to alter the surface roughness of the TEC, CX, XF, and QF materials.

**Conclusion:** The application of thermal cycling in a water environment in accordance with the ISO 11405 standard causes the degradation of the organic matrix and the exposure of filler molecules on the surface of both conventional and high-viscosity bulk-fill composites. Some of the tested RBCs, especially those containing modern hydrophobic monomers, are less susceptible to these processes.

**Keywords:** thermal cycling; surface roughness; resin-based composite; bulk-fill composite.

## INTRODUCTION

Resin-based composites (RBCs), the most popular materials in restorative dentistry for direct restorations, still have several disadvantages, such as lack of direct adhesion to the dental tissues, lack of bioactivity, time-consuming and costly treatment, sensitivity to procedural technique and gradual degradation of their surface, regardless of the degree of final polishing [1, 2]. The damage to the RBC surface is influenced by the harsh and varied conditions in the oral cavity, of which repeated temperature changes contribute to the aging of the surface of composite fillings [2]. The resting temperature in the oral cavity is 35.7–37.7°C for men, and 33.2–38.1°C for women. According to Michalesco et al. research, these values can reach as low as 12°C when ice cream is consumed and as high as +85°C when hot drinks are consumed [3]. However, Moore et al. recorded a temperature of more than 50°C, and the lowest result obtained was a temperature of about 5°C during a 24-h observation [4]. Cyclic changes in temperature lead to shrinkage and expansion of the structure of RBCs, resulting in easier diffusion of solvents into the material and increased water sorption and hydrolytic effects [5]. The aqueous environment of the oral cavity, with its variable characteristics, different pH, and titrated acidity, also favors the degradation of the surface of RBC fillings.

According to Ferracane's research, composites are susceptible to water sorption, especially in the first days after application, and become saturated after 7–60 days of incubation, resulting in a weakening of the bonds in the polymer network through affinity to water molecules, thus reducing the stability of the association of filler molecules with the organic matrix [6]. Sorption creates pathways for solvent penetration into the matrix and initiates unfavorable chemical and physical processes (volume change, swelling, plasticization, softening, oxidation, solubility, and hydrolytic degradation) [7]. Although swelling can compensate for the polymerization shrinkage of composites, it mainly affects their mechanical properties. The processes in the aquatic environment contribute to the loss of microhardness of the composite material, which over time reduces the compressive strength and wear resistance, clinically evidenced by increased surface roughness and discoloration [6, 8, 9].

The method of cyclic temperature changes in the aquatic environment is used to accelerate the degradation of the composite material *in vitro* under conditions that mimic those found in the oral cavity [10, 11, 12, 13]. In this method, samples of the material are sequentially placed in cold and warm water baths. The simulation of continuous temperature changes accelerates the degradation processes of the composite [14, 15]. Repeated temperature changes create stresses within the material structure and lead to the

formation of micro-cracks in the matrix or degradation of the bond between inorganic filler molecules and the organic matrix [16]. In addition, thermal cycling increases the hydrolytic effect of water on the composite by destroying the coupling agents on the surface of the filler molecules and swelling the organic matrix.

Most studies agree that the use of thermal cycling increases the surface roughness of the composite. This is caused by the degradation of the organic matrix and the exposure of irregular filler particles on the surface of composite materials [9, 17]. Therefore, the maintenance of appropriate surface roughness parameters of the material after the application of thermal loads could be considered as one of the signs of its quality. It could also be one of the factors for predicting its stability and resistance to the hard stresses that occur in the oral cavity, especially in the posterior region. The surface degradation of the composite material after thermal cycling is influenced, apart from the variable parameters of the procedure, by the properties of the composite material such as the percentage-weight ratio of the filler content to the organic matrix, the size and shape of the filler particles, the effect of water on the filler particles, and the presence of hydrophobic monomers. The value of the surface roughness coefficient of the composite should not exceed  $R_a = 1 \mu\text{m}$  [18]. Higher values of  $R_a$  mean that the filling does not meet the aesthetic requirements and is conducive to bacterial plaque retention. Lower roughness values are desirable. According to Willems et al., the roughness

coefficient should be less than  $R_a = 0.64 \mu\text{m}$ , which corresponds to the enamel roughness [19]. Some studies suggest that the surface roughness after polishing should be below  $0.20 \mu\text{m}$  to ensure resistance to bacterial adhesion and reduce susceptibility to discoloration [20, 21, 22].

The effect of thermal cycling on the surface roughness of a RBC has been discussed in several studies [11, 23, 24]. However, there is very little research on the effect of thermal aging on the surface preservation of the high-viscosity bulk-fill RBCs used for the direct reconstruction of posterior tooth tissues [25, 26]. The aim of this study was to investigate the effect of thermal cycling on the surface geometry of these RBCs in comparison with conventional nanohybrid RBCs. The study is a continuation of previous research [27]. The null hypothesis was that the use of thermal cycling in accordance with ISO 11405 standards will not affect the value of the quantitative and qualitative assessment of the surface of conventional nanohybrid and high-viscosity bulk-fill composites.

## MATERIALS AND METHODS

Four high-viscosity bulk-fill composites: Tetric EvoCeram Bulk Fill – TBF (Ivoclar Vivadent, Schaan, Liechtenstein), X-tra fil – XF (VOCO, Cuxhaven, Germany), Filtek Bulk Fill Posterior – FBF (3M-ESPE, St. Paul, USA), and QuixFil – QF (Dentsply, Konstanz, Germany) and 4 conventional nanohybrid RBCs: Tetric

TABLE 1. Basic data on the composition of the inorganic and organic components of the tested resin-based composites

Composite type	Material	Manufacturer	Organic matrix	Inorganic filler	Filler content by weight (%)	Filler particle size ( $\mu\text{m}$ )
Conventional	Ceram-X™ Mono	Dentsply (Konstanz, Germany)	Methacrylate-modified polysiloxane, Bis-GMA	barium aluminum boron silicon glass, silicon oxide as a nanofiller particle	76	0.01–1.5
	GrandioSO®	VOCO (Cuxhaven, Germany)	Bis-GMA, TEGDMA, Bis-EMA	glass-ceramic filler, silicon dioxide, pigments (iron oxide, titanium dioxide)	89.5	0.02–1
	Filtek™ Z550	3M-Espe (St. Paul, USA)	Bis-GMA, UDMA, Bis-EMA, PEGDMA, TEGDMA	surface-modified zirconium dioxide/silica, surface-modified silica particles	82	0.02–3
	Tetric EvoCeram®	Ivoclar Vivadent (Schaan, Liechtenstein)	Bis-GMA, UDMA, Bis-EMA	barium glass filler, Ytterbium trifluoride, mixed oxide	79.5	0.04–3
Regular viscosity bulk-fill	Filtek™ Bulk Fill Posterior	3M-Espe (St. Paul, USA)	AUDMA, AFM, DDDMA, UDMA	nonagglomerated/nonaggregated silica filler, nonagglomerated/nonaggregated zirconia filler, aggregated zirconia/silica cluster filler, ytterbium trifluoride	76.5	0.004–0.1
	Tetric EvoCeram® Bulk Fill	Ivoclar Vivadent (Schaan, Liechtenstein)	Bis-GMA, UDMA, Bis-EMA	barium aluminum silicate glass, Isofiller, ytterbium fluoride, spherical mixed oxide	62.5 + 17 Isofiller	0.04–3
	QuixFil™	Dentsply (Konstanz, Germany)	UDMA, TEGDMA, di- and trimethacrylate resins, carboxylic acid-modified methacrylate resins	silanized strontium-aluminum glass with the addition of sodium fluoride	86	0.15–30
	Xtra-fil	VOCO (Cuxhaven, Germany)	Bis-GMA, UDMA, TEGDMA	inorganic fillers (no accurate data)	86	>20

Bis-GMA – bisphenol A-glycidyl methacrylate; UDMA – urethane dimethacrylate; Bis-EMA – ethoxylated bisphenol-A dimethacrylate; TEGDMA – triethylene glycol dimethacrylate; PEGDMA – poly (ethylene glycol) dimethacrylate; AUDMA – aromatic urethane dimethacrylate; AFM – addition-fragmentation monomers; DDDMA – 1, 12-dodecanediol dimethacrylate

EvoCeram – TEC (Ivoclar Vivadent, Schaan, Liechtenstein), GrandioSO – GD (VOCO, Cuxhaven, Germany), Filtek Z550 – FZ (3M-ESPE, St. Paul, USA), and Ceram-X Mono – CX (Dentsply, Konstanz, Germany) were evaluated. Basic data on the composition of the inorganic and organic components of the tested RBCs are presented in Table 1.

### Sample preparation

Experimental RBC samples were prepared according to our own research protocol [27]. Twelve samples were collected for each RBC and divided into 2 different groups – the control group – K (n = 6), and the thermal cycling group – TC (n = 6). In the K, 5 representatives of each RBC material were examined by profilometry, while 1 was examined by SEM. Similarly, in the TC, 5 samples of each RBC material were tested by profilometry and 1 sample was tested by SEM after thermal cycling.

### Thermal cycling

Thermal cycling was performed according to ISO 11405 recommendations: temperature: 5–55°C, dwell time: 30 s, number of cycles: 500 (THE-1100, SD Mechatronik GmbH, Feldkirchen-Westerham, Germany).

### Profilometric examination

Profilometric tests were carried out at the Department of Mechanical Engineering of the West Pomeranian University of Technology in Szczecin. A Hommel-Etamic Turbowave v. 7.36 profilometer was used. Each composite sample surface was scanned 5 times with a TK100 measuring tip at a speed of 0.50 mm/s. The roughness coefficient value – Ra – was evaluated for the purpose of the study.

### Scanning electron microscope examination

The imaging of the composite surface microstructure was carried out at the Center for Bioimmobilisation and Innovative Packaging Materials of the West Pomeranian University of Technology in Szczecin. A VEGA 3 SEM (Tescan Analytics, Fuveau, France) was used. The surface quality of each specimen was evaluated descriptively using photographic prints at 1000x and 3000x magnification, according to the method described in the previous study [27]. The samples were coded according to their qualitative assessment: 1 for undamaged surfaces; 2 for surfaces with minor damage; 3 for rough surfaces; and code 4 for rough surfaces with clear damage. The microphotographs were scored by 2 people.

### Statistical analysis

The R program, version 4.0.0 was used for statistical analysis. The intraclass correlation coefficient (ICC) test was used to demonstrate the reliability of the measurements. Normality of the distribution was assessed using the Anderson–Darling test. The ANOVA test was used to check for interactions. For non-parametric data, the following tests were used to check statistical significance: the Mann–Whitney U-test and the Kruskal–Wallis test. The statistical significance threshold was  $p \leq 0.05$  for the overall analysis, while the Bonferroni correction was

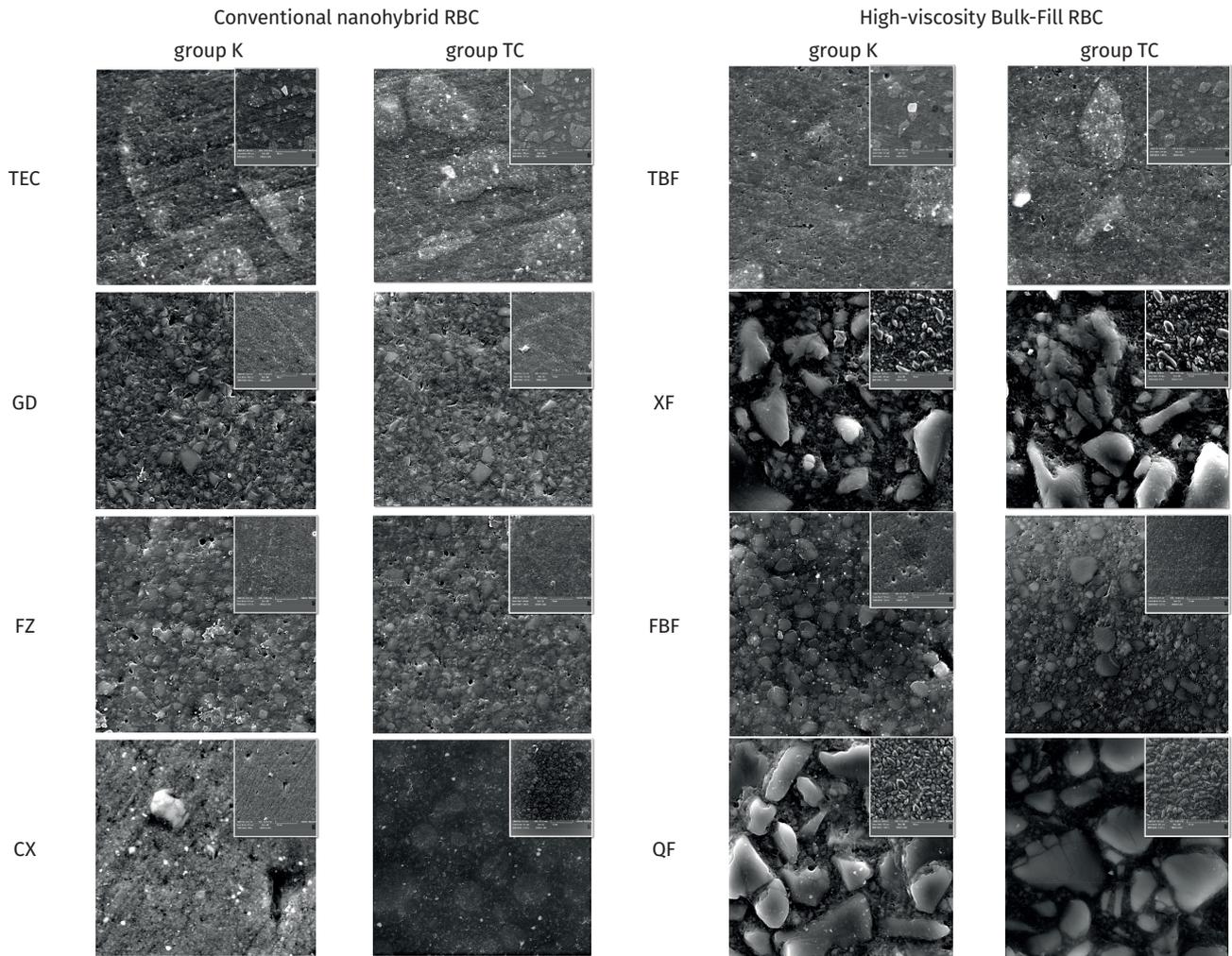
used for multiple analyses, where the significance threshold was  $p \leq 0.006$ , depending on the number of hypotheses tested.

## RESULTS

Figure 1 presents representative examples of SEM images of RBCs used in a study before and after thermal cycling. Table 2 presents the results of the qualitative surface assessment of composites before and after thermal cycling (groups K and TC) carried out by 2 independent researchers based on SEM photographs. The qualitative assessment of the surface of the composites of the K showed that the surface of all tested conventional RBCs was characterized by a lower number of defects than the surfaces of bulk-fill RBCs. The average score for the conventional RBCs was 18, classifying the surface as smooth with minor damage. On the other hand, the bulk-fill type RBCs obtained an average score of 28.4, which proves a rough surface. Tetric EvoCeram Bulk Fill, as the only bulk-fill RBC, obtained a more favorable qualitative score (12.5) than the 3 conventional RBCs (GD, FZ, CX), comparable to the score obtained by TEC (10.5). The qualitative assessment of the surface of the conventional composites of the TC also showed a lower number of surface defects (average score – 9.3), compared to the bulk-fill materials, whose average qualitative assessment score was 24 (Tab. 2). Compared to the K, all RBCs in the TC had better surface quality scores. The conventional RBCs in the TC achieved an average score of 9.3, which was 2 times lower than the average score of the K (9.3 vs. 18). Conventional RBC surfaces were classified as smooth and undamaged after thermal cycling. The bulk-fill RBCs after thermal cycling also scored lower in the qualitative assessment of surface degradation (24) than the K (28.4). However, their surfaces were still classified as rough, with the exception of the TBF material.

The results of the reliability analysis of the SEM images, performed by 2 independent examiners were analyzed by the ICC test, which showed the excellent reliability of the measurements. The score of a single examiner was 0.975, and the mean score of 2 investigators was 0.987. The value of the significant factor P was statistically significant ( $p = 1.94e-27$ ). The confidence interval for the single examiner's score was 0.954–0.987 (mean 0.976–0.993).

Table 3 shows a comparison of the mean surface roughness coefficient of conventional and bulk-fill composites in the K and TC groups. Statistical analysis showed that the applied thermal cycling increased the surface roughness of conventional RBCs: TEC ( $p = 0.000007$ ), GD ( $p = 0.04$ ), and CX ( $p = 0.0005$ ). A reduction in the surface roughness of bulk-fill composites was observed in the case of materials: XF ( $p = 0.000003$ ) and QF ( $p = 0.0002$ ). After applying the Bonferroni correction, it was shown that thermal cycling changes the surface roughness of TEC, CX, XF, and QF materials. The results are shown graphically in Figure 2. Again, the heterogeneity of the group of bulk-fill materials is highlighted in terms of the value of the roughness coefficient in both groups – K and TC.



RBC – resin-based composites; K – control group; TC – thermal cycling group; TEC – Tetric EvoCeram; TBF – Filtek™ Bulk Fill Posterior; GD – GrandioSO®; XF – Xtra-fil; FZ – Filtek™ Z550; FBF – Filtek™ Bulk Fill Posterior; CX – CeramX™ Mono; QF – QuixFil™; SEM – scanning electron microscope

**FIGURE 1.** Representative examples of SEM images of RBCs used in a study, before and after thermal cycling. Magnification 3000x, upper right corner 1000x

**TABLE 2.** Results of qualitative surface evaluation of conventional and high-viscosity bulk-fill composites in the control group (K) and thermal cycling group (TC) by 2 independent investigators

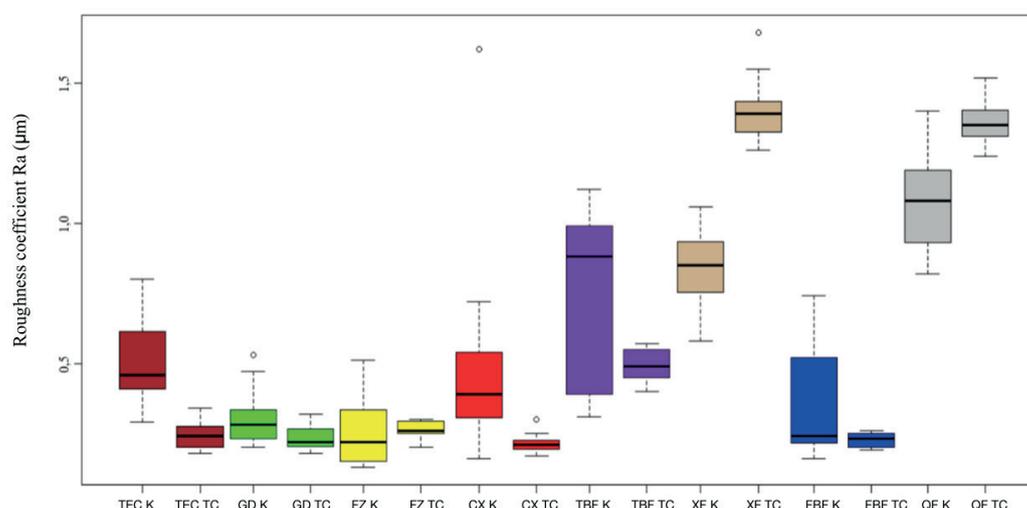
RBC	Material	Researcher 1		Researcher 2		Mean		Code	
		K	TC	K	TC	K	TC	K	TC
<b>Conventional nanohybrid</b>	TEC	9	8	12	6	10.5	7	0	0
	GD	24	7	21	6	22.5	6.5	1	0
	FZ	19	12	20	11	19.5	11.5	1	0
	CX	24	12	15	12	19.5	12	1	0
<b>Mean</b>		19	9.75	17	8.75	18	9.25	0.75	0
<b>High-viscosity bulk-fill</b>	TBF	15	10	10	9	12.5	9.5	1	0
	XF	35	29	36	28	35.5	28.5	2	2
	FBF	36	26	26	24	31	25	2	2
	QF	33	33	36	33	34.5	33	2	2
<b>Mean</b>		29.75	24.5	27	23.5	28.375	24	1.75	1.5

RBC – resin-based composites; TEC – Tetric EvoCeram; GD – GrandioSO®; FZ – Filtek™ Z550; CX – CeramX™ Mono; TBF – Filtek™ Bulk Fill Posterior; XF – Xtra-fil; FZ – Filtek™ Z550; FBF – Filtek™ Bulk Fill Posterior; QF – QuixFil™

**TABLE 3.** Comparison of the mean values of the surface roughness coefficient (Ra) of conventional and bulk-fill composites in the control group (K) and the thermal cycling group (TC)

RBC	Acronym	Group K						Group TC						p
		Ra ( $\mu\text{m}$ ) n = 5	SD	median	min.	max.	SE	Ra ( $\mu\text{m}$ ) n = 5	SD	median	min.	max.	SE	
Conventional nanohybrid	TEC	0.24	0.05	0.24	0.18	0.34	0.01	0.51	0.16	0.46	0.29	0.8	0.04	0.000007
	GD	0.24	0.04	0.22	0.18	0.32	0.01	0.3	0.1	0.28	0.2	0.53	0.03	0.04168
	FZ	0.26	0.03	0.26	0.2	0.3	0.01	0.25	0.12	0.22	0.13	0.51	0.03	0.454
	CX	0.21	0.03	0.21	0.17	0.3	0.01	0.49	0.35	0.39	0.16	1.62	0.09	0.000562
High viscosity bulk-fill	TBF	0.49	0.06	0.49	0.4	0.57	0.02	0.75	0.31	0.88	0.31	1.12	0.08	0.09668
	XF	1.4	0.12	1.39	1.26	1.68	0.03	0.84	0.13	0.85	0.58	1.06	0.03	3.3E-06
	FBF	0.23	0.03	0.23	0.19	0.26	0.01	0.35	0.19	0.24	0.16	0.74	0.05	0.204
	QF	1.36	0.07	1.35	1.24	1.52	0.02	1.08	0.19	1.08	0.82	1.4	0.05	0.000259

RBC – resin-based composites; TEC – Tetric EvoCeram; GD – GrandioSO®; FZ – Filtek™ Z550; CX – CeramX™ Mono; TBF – Filtek™ Bulk Fill Posterior; XF – Xtra-fil; FBF – Filtek™ Bulk Fill Posterior; QF – QuixFil™; SD – standard deviation; SE – standard error



K – control group; TC – thermal cycling group; TEC – Tetric EvoCeram; GD – GrandioSO®; FZ – Filtek™ Z550; CX – CeramX™ Mono; TBF – Filtek™ Bulk Fill Posterior; XF – Xtra-fil; FBF – Filtek™ Bulk Fill Posterior; QF – QuixFil™

**FIGURE 2.** Comparison of the roughness Ra values of resin-based composites materials of the control group and the group subjected to thermal cycling

## DISCUSSION

The ISO 11405 standard, which describes the thermal cycling method for dental materials, recommends the use of temperatures between 5–55°C in water baths. This temperature range is widely used in research [28, 29, 30]. The time that the material sample spends in each bath is called the dwelling time. Amaral et al. suggest that the proposed ISO time of 30 s is too long and does not reflect *in vivo* conditions in the oral cavity [31]. Other studies report that the dwelling time does not appear to affect the results of the thermal cyler test [32, 33]. An outstanding issue in studies using thermal cycling is the determination of the appropriate number of cycles. The ISO 11405 standard suggests the use of 500 cycles, which, according to Stewardson et al., is equivalent to 2 months in the oral cavity [34]. Other studies suggest that 500 cycles are insufficient to demonstrate degradation *in vivo* and suggest using

10,000 cycles to replicate the year of material functioning in the oral cavity [35, 36]. Although thermal cycling is most widely used as an *in vitro* aging method for dental materials, studies often use different test parameters (different temperatures, immersion times, and the number of cycles), making it difficult to compare results. A review of 130 publications by Gale and Darvell shows that the most frequently used protocol in research is ISO 11405 [35]. Therefore, in this work, the protocol proposed in the ISO 11405 standard included a temperature range of 5–55°C, dwelling time of 30 s, and 500 cycles, was used. The use of 500 thermal cycles resulted in changes in the surface roughness coefficients of most conventional and bulk-fill RBCs compared to the control group (Tab. 3, Fig. 2). The results contradict the results of studies by Tuncer et al. and Hahnel et al. which showed that 10,000 thermal cycles at 5°C and 55°C did not significantly affect the surface roughness of composites [11, 23]. Minami et al. also showed that only 50,000 cycles

significantly damaged the surface structure of composites, mainly by breaking the bond between the filler molecule and the matrix, resulting in the wash out of the filler molecules [24].

In this study, the roughness coefficient of all conventional RBCs, except FZ, increased after 500 thermal cycles as shown in Table 3. The null hypothesis was therefore rejected. The FZ material obtained similar results in the study by Pala et al., where the roughness decreased after 10,000 thermal cycles and the Enhance polishing system [37]. Furthermore, in the study by Aytac et al., FZ also showed adequate resistance to 10,000 thermal cycles, with the Ra value increasing from 0.18  $\mu\text{m}$  before to 0.26  $\mu\text{m}$  after the application of thermal cycling [38]. The considerable resistance to water and temperature changes may be due to the use of the PEGDMA monomer in the FZ material (Tab. 1), which reduces the viscosity of the matrix, but is much more hydrophobic in nature than TEGDMA and therefore may be resistant to hydrolysis. In addition, although the roughness of GD material increased significantly by 0.06  $\mu\text{m}$  after thermal cycling ( $p = 0.04$ ) compared to the K, it is also a material that shows some resistance to degradation in the aqueous environment. This is also confirmed by the study by Tuncer et al., in which, the surface roughness of the GD material increased by 0.17–0.18  $\mu\text{m}$  after 10,000 thermal cycles [11]. On the other hand, CX contains no hydrophobic monomers but only Bis-GMA and polysiloxanes modified with methacrylates, showed a significant increase in surface roughness by 0.28  $\mu\text{m}$  ( $p = 0.0005$ ) compared to the K (Tab. 3, Fig. 2). Similar results were obtained by Barucci-Pfister and Göhring who observed the degradation of the CX surface after thermal cycling [39]. The roughness of the TEC surface after thermal cycling in this study increased to 0.51  $\mu\text{m}$ , which is twice the roughness value of the control group (Tab. 3, Fig. 2). A similar increase in TEC roughness increase was found by Rinastiti et al. [16]. However, their method of roughness determination was based on SEM imaging, which makes it impossible to correctly compare the results. One of the factors responsible for the increase in surface roughness of the TEC material may be the presence of the UDMA monomer in its organic matrix, which is a monomer with greater susceptibility to water sorption and degradation due to the presence of urethane bonds (Tab. 1). The SEM images of all conventional RBCs after thermal cycling were assessed as smooth surfaces, without damage (Tab. 2, Fig. 1), which, together with the profilometry results (Tab. 3, Fig. 2), proves the acceptable roughness of these materials even after thermal cycling.

The varied outcomes of the surface structure of bulk-fill composites due to thermal cycling led to the rejection of the null hypothesis (Tab. 3, Fig. 2). There are few studies in the literature on the surface roughness of bulk-fill composites after thermal cycling, which prevented a wider discussion of the results obtained [25, 26]. Bulk-fill composites containing large particles of inorganic fillers (QF, XF) achieved significantly lower roughness values compared to the K ( $p = 0.0002$  for QF and  $p = 0.000003$  for XF) – Table 3. According to Milleding et al., water could act as a weak acid, and smooth the sharp edges of large glass filler particles [40]. In this study, as a result

of thermal cycling, the glass macroparticles in the QF and XF materials could be smoothed by the erosive action of water, thus reducing the roughness of the materials (Fig. 1). Despite better results in the profilometric test compared to the K, both materials still showed high values of the Ra coefficient (QF: Ra = 1.08  $\mu\text{m}$  and XF: Ra = 0.84  $\mu\text{m}$ ). This was confirmed by the qualitative test in which the surfaces of both materials were assessed as rough (Tab. 2). A different result for the XF material was presented in the study by Karadaş and Demirbuğa, in which XF achieved a significantly higher roughness than the K after 3,000 thermal cycles [26]. On the other hand, FBF and TBF, which are materials similar in structure to conventional nanohybrid RBCs, obtained higher, but not significant, surface roughness parameters compared to the K. Thus, they proved to be relatively less susceptible to the applied thermal cycling. Higher, but not significant, FBF surface roughness results were also obtained after the application of 10,000 thermal cycles in the study by Tekçe et al. [25].

One way to reduce the susceptibility of RBCs to degradation in the aquatic environment is to increase the amount of inorganic filler in the material [41]. Initially, the resistance to degradation increases with increasing inorganic filler content, but once the ability of the filler molecules to be wetted by the organic matrix is exceeded, the mechanical properties and resistance to degradation decrease and the composite becomes cohesive [42, 43, 44]. Materials with a high content of filler grains >85% in the bulk-fill RBCs group showed surface smoothing after 500 thermal cycles (QF, XF), while in the RBCs group of conventional composites, GD, which contains the highest amount of filler (89.5%), showed a significant increase in roughness (Tab. 1 and 3). On the other hand, the conventional material CX with the lowest percentage of inorganic fillers – 76%, obtained a significant increase in surface roughness after the application of thermal cycling, which is in agreement with the results obtained by Xu et al. [41].

The roughness of the composite is also influenced by the size and shape of the filler particles in addition to the inorganic/organic compound ratio. This is confirmed by numerous studies determining the surface roughness of macroparticles, microparticles, and hybrid materials over the years of RBC development [42, 45, 46]. The influence of the size and shape of the filler particles on the Ra values was also confirmed in our previous study [27]. The size and shape of the filler molecules also play an important role in the resistance of the composite to degradation in the aquatic environment [24]. A study by dos Santos et al. confirmed that RBCs made of nanoparticles are more resistant to degradation because their surface structure is homogeneous, and smooth, with filler particles not protruding above the surface [47]. This is supported by Minami et al., who found that only Filtek Supreme – a nanophilic material – had a homogeneous composite surface without damage after polishing. Adequate resistance to hydrolytic degradation was also demonstrated for this material after 20,000 and 50,000 thermal cycles, where only exposed nanofiller particles were observed, without the presence of sites from which the molecules would be washed out [24]. According to Xu et al., even the use of 10,000 thermal cycles does not

significantly affect the degradation of the properties of the organic matrix, and the resistance to degradation in the aqueous environment is due to the filler molecules and stresses at the interface between the organic and inorganic phases [41]. In the same study, it was found that the use of new nanophilic fillers made the composite material 3 times more resistant to fracture than when glass fillers were used, and much more resistant to hydrolytic degradation.

## CONCLUSIONS

The use of thermal cycling in an aqueous environment in accordance with the ISO 11405 standard results in the degradation of the organic matrix and the exposure of filler molecules on the surface of both conventional and high-viscosity bulk-fill composites. Some RBCs, particularly those containing modern hydrophobic monomers, are less susceptible to these processes.

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