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Surface roughness of different types of resin composites after artificial aging procedures: an in vitro study

Zeynep Biçer[1*], Batu Can Yaman[1], Özge Çeliksöz[1] and Hatice Tepe[1]

Abstract

Background The temperature changes, chemical agents, and brushing activity that resin composite restorations are exposed to in the oral environment can cause changes in surface roughness. In this study, the aim was to investigate in vitro the clinical one-year surface roughness changes of different types of composites (flowable or conventional) from the same companies by subjecting them to immersion in solutions, brushing, and thermal cycling procedures to simulate intraoral conditions.

Methods Four different resin composite brands were included in the study using both their conventional (Charisma Smart, 3M Filtek Ultimate Universal, Omnichroma, Beautifil II) and flowable resin composites (Charisma Flow, 3M Filtek Ultimate Flowable, Omnichroma Flow, Beautifil Flow Plus F00), giving 4 groups with 2 types of resin composite in each. 40 samples were prepared for each group/resin type, for a total of 320 samples. After initial surface roughness measurements by a mechanical profilometer, the samples were divided into 4 subgroups (n = 10) and immersed in solutions (distilled water, tea, coffee, or wine) for 12 days. The samples were then subjected to 10,000 cycles of brushing simulation and 10,000 cycles of thermal aging. Surface roughness measurements were repeated after the procedures. For statistical analysis, the 3-way analysis of variance and the Tukey test were used ($p < 0.05$).

Results It was concluded that composite groups and types had an effect on surface roughness at time t_0 ($p < 0.001$). At time t_1, the highest surface roughness value was obtained in the Beautifil-conventional interaction. When the surface roughness values between time t_0 and t_1 were compared, an increase was observed in the Beautifil II and Beautifil Flow Plus F00, while a decrease was observed in the other composite groups.

Conclusion Composite groups, types, and solutions had an effect on the surface roughness of resin composites. After aging procedures, it was concluded that the Beautifil group could not maintain the surface structure as it exceeded the threshold value of 0.2 µm for bacterial adhesion.

Keywords Brushing, Immersion in solutions, Resin composite, Surface roughness, Thermal aging

Introduction

With the developments in dental materials, the use of resin composites in anterior and posterior restorations has become quite common. Ensuring surface smoothness with good color harmony is the key to aesthetic success in resin composite restorations [1]. The finishing and polishing procedure provides restorations that are smooth and shiny, similar to natural teeth. It also ensures the harmony of the restoration with oral tissue,

*Correspondence:
Zeynep Biçer
zeynokacan@gmail.com
[1] Department of Restorative Dentistry, Faculty of Dentistry, Osmangazi University, Eskişehir, Turkey

and prevents mismatch and discoloration at the tooth-restoration transition [2]. In addition, a smooth restoration surface increases patient comfort, facilitates oral hygiene, prevents bacterial adhesion and plaque formation, and reduces tissue inflammation and the risk of secondary caries and fractures [3, 4]. A roughness of 0.3 µm on the restoration surface can be easily recognized by the patient through the tongue, and 0.2 µm is the critical value for bacterial adhesion. Therefore, a well-finished restoration is very important for surface characteristic [3, 4].

The structure and mechanical properties of the restorative material affect surface roughness [5], as a result, reducing the particle size in the filler and increasing the filler ratio in resin composites are important factors in material development [6, 7]. It is generally accepted that resin composites with smaller filler particle sizes show better surface properties [8]. Supra-nano filler and nanohybrid composites have smaller filler particles compared to microhybrid composites, whereas giomers, which are characterized by fluoride release, contain surface pre-reactive glass (S-PRG) particles with larger sizes [9]. Improving the mechanical properties by improving the filler content has become a priority for the next generation of flowable composites, as with other materials [10]. Resin composite restorations are subjected to many traumas in the mouth, such as chewing forces, temperature changes, chemical agents from eating and drinking, smoking, and brushing activity. However, there are few studies in the literature on the effect of these conditions on the surface roughness of restorations [8, 11]. The acid in consumed beverages causes matrix hydrolysis and deterioration, and alcohol causes matrix deterioration due to particle loss, both effects causing wear and increased surface roughness in restorations [1, 8]. The toothbrush used, the abrasive content of the toothpaste, increased brushing time, and force can increase the surface roughness of composite resins due to abrasion, which causes aesthetic problems such as reducing their shine [12]. In order to observe the effects of intraoral conditions on the restoration surface, aging methods, such as immersion in solutions, brushing simulation, and thermal cycling can be used in in vitro studies [13–15].

Surface roughness measurements can be performed by a mechanical profilometer, optical profilometer, scanning electron microscope (SEM), and atomic force microscope (AFM) [16]. Mechanical profilometers perform two-dimensional measurements by scanning the surface of the sample with a fixed stylus at a certain distance from the surface, while SEM performs two-dimensional measurements by scanning the sample surface with an electron beam [10, 16]. In AFM, three-dimensional surface images of the examined samples with high atomic resolution are obtained [17]. In the literature, some researchers have used the average surface roughness (Ra) value as the criterion for measuring surface roughness, while others have used the maximum surface roughness (Rmax) value [18].

There are limited studies evaluating the surface roughness of resin composites using a combination of various aging procedures. The more clinical conditions can be simulated in an in vitro study, the more realistic results can be obtained. Therefore, this study aimed to investigate the clinical one-year surface roughness change in vitro by subjecting different types of resin composites (flowable or conventional) from the same companies to immersion in solutions, brushing, and thermal cycling procedures to simulate intraoral conditions. For this purpose, the null hypotheses of the study were that (1) the resin composite type, (2) the solution, and (3) the resin composite brands (resin composite groups) would not affect the surface roughness.

Methods

The sample size in the study was determined using the G*Power analysis with a reference study [19]. With 95% confidence (1-α), 95% test power (1-β), and $f=0.25$ effect size, the number of samples required to be included in the study was 280. Since the study was completed with 320 samples, 97.3% test power was determined by the post hoc power analysis.

The study included 4 different resin composite brands, both conventional and flowable, and formed 4 groups, each with 2 types of resin composites. The 4 different conventional resin composites used were Charisma Smart (Heraeus Kulzer, Hanau, Germany), 3M Filtek Ultimate Universal (3M ESPE, St. Paul, MN, USA), Omnichroma (Tokuyama, Tokyo, Japan), Beautifil II (Shofu Inc., Kyoto, Japan) and 4 different flowable resin composites used were Charisma Flow (Heraeus Kulzer, Hanau, Germany), 3M Filtek Ultimate Flowable (3M ESPE, St. Paul, MN, USA), Omnichroma Flow (Tokuyama, Tokyo, Japan), Beautifil Flow Plus F00 (Shofu Inc., Kyoto, Japan) (Table 1). Resin composite groups are shown in Fig. 1. 40 samples were prepared for each group/resin type, for a total of 320 samples. The flow chart of the study is shown in Fig. 2. All procedures were performed by the same operator to avoid operator variability.

In the present study, a 2 mm deep, 10 mm diameter metal mold was used to create samples of the same dimensions. Mylar strips and glass coverslips were placed on the upper surfaces of the composites placed in the metal molds so that no air bubbles remained. The samples were polymerized with the SmartLite Focus (Dentsply Sirona, USA) LED light device first for 20 s on the glass coverslip and then for 10 s by removing the

Table 1 Conventional and flowable resin composite materials used in the study

Material	Code	Manufacturer Lot No	Material Type	Organic matrix and inorganic filler	Filler % (wt/vol)	Particle size
Charisma Smart	CHR Conventional	Heraeus Kulzer, Hanau, Germany Lot: K010524	Submicrohybrid resin composite	Bis-GMA, TEGDMA; Ba-Al-F silicate glass, SiO_2	78 wt/ 59 vol	0.005–10 µm
Charisma Flow	CHR Flowable	Heraeus Kulzer, Hanau, Germany Lot: K010305	Hybrid flowable resin composite	EBADMA, TEGDMA; Ba-Al-F silicate glass, SiO_2	62 wt/ 38 vol	0.005–5 µm
3M Filtek Ultimate Universal	FLTU Conventional	3M ESPE, St. Paul, MN, USA Lot: NE49412	Nanohybrid resin composite	Bis-GMA, UDMA, TEGDMA, Bis-EMA, PEGDMA; silica and zirconia particles	78.5 wt/ 63.3 vol	0.5–1 µm
3M Filtek Ultimate Flowable	FLTU Flowable	3M ESPE, St. Paul, MN, USA Lot: N985364	Nanohybrid flowable resin composite	Bis-GMA, UDMA, TEGDMA, Bis-EMA, procrylate resins; ytterbium trifluoride, silica, and zirconia particles	65 wt/ 46 vol	0.1–5 µm
Omnichroma	OMN Conventional	Tokuyama, Tokyo, Japan Lot: 1211	Supra-nano spherical filled resin composite	UDMA, TEGDMA, supra-nano spherical silica-zirconia filler	79 wt/ 68 vol	0.2–0.4 µm
Omnichroma Flow	OMN Flowable	Tokuyama, Tokyo, Japan Lot: 0197	Supra-nano spherical filled flowable resin composite	UDMA, TEGDMA, supra-nano spherical silica-zirconia filler	71 wt/ 57 vol	0.2–0.4 µm
Beautifil II	BT Conventional	Shofu Inc., Kyoto, Japan Lot: 121,956	Giomer (Fluoride Releasing Restorative Material)	Bis-GMA, TEGDMA; S-PRG filler based on flouroboroalumi-nosilicate glass	83.3 wt/ 68.6 vol	0.01–4 µm
Beautifil Flow Plus F00	BT Flowable	Shofu Inc., Kyoto, Japan Lot: 032147	Giomer (Fluoride Releasing Flowable Restorative Material)	Bis-GMA, TEGDMA; S-PRG filler based on flouroboroalumi-nosilicate glass	67.3 wt/ 47 vol	0.01–4 µm

Bis-EMA bisphenol A diglycidyl methacrylate ethoxylated, *Bis-GMA* bisphenol A diglycidyl methacrylate, *PEGDMA* Polyethylene glycol dimethacrylate, *SiO_2* Silicon dioxide, *S-PRG* surface pre-reacted glass ionomer, *TEGDMA* triethylene glycol dimethacrylate, *UDMA* urethane dimethacrylate

glass coverslip. The samples were then removed from the metal mold and polymerized for 10 s on their back surfaces. The same light curing unit with an intensity of at least 1000 mW/cm2 was used for all polymerization steps. The light output was checked every 5 samples using a radiometer (Woodpecker LED-F, Woodpecker Medical Instrument Co., China). The samples were placed in distilled water and stored in a 37 °C incubator for 24 h. The samples were then subjected to finishing and polishing processes. Polishing disks (Optidisc, Kerr Corporation, Bioggio, Switzerland) were applied at 10,000 rpm for 20 s, extra-coarse, coarse-medium, fine, and extra fine, respectively, with each disk pass being washed with air–water spray for 5 s. A new set of disks was used for each sample. The surface polishing of the resin composite samples was then completed by applying polishing paste (SDI Limited, Australia) with a goat hairbrush (Jiffy, Ultradent Product, South Jordan, Utah, USA). The samples were washed with an air–water spray to remove debris from the surface.

The samples in the resin composite group/resin type where the first surface roughness measurement was performed were divided into 4 subgroups to be immersed in 4 different solutions (wine, coffee, tea, or distilled water). The samples were randomly divided into subgroups. There were 40 samples in each group/resin type and 10 samples in each subgroup for immersion in the solutions.

Wine (Öküzgözü, Doluca, Istanbul, Turkey), coffee (Nescafe Gold Nestle, Switzerland), tea bags (Yellow Label, Lipton, Istanbul, Turkey), and distilled water were the solutions used in the study. For the coffee solution, 200 ml of boiling water at 100 °C and 2 g of coffee were used. For tea, 200 ml of water was used in the same way, with a tea bag, which was shaken gently in boiling water according to the manufacturer's instructions and left for 2 min before being removed from the water. No additional treatment was applied for the wine or distilled water. After reaching 37 °C, the solutions were placed in 1.5 mm tubes, and one sample was placed in each. The samples, which were kept separate from each other using Eppendorf tubes, were kept

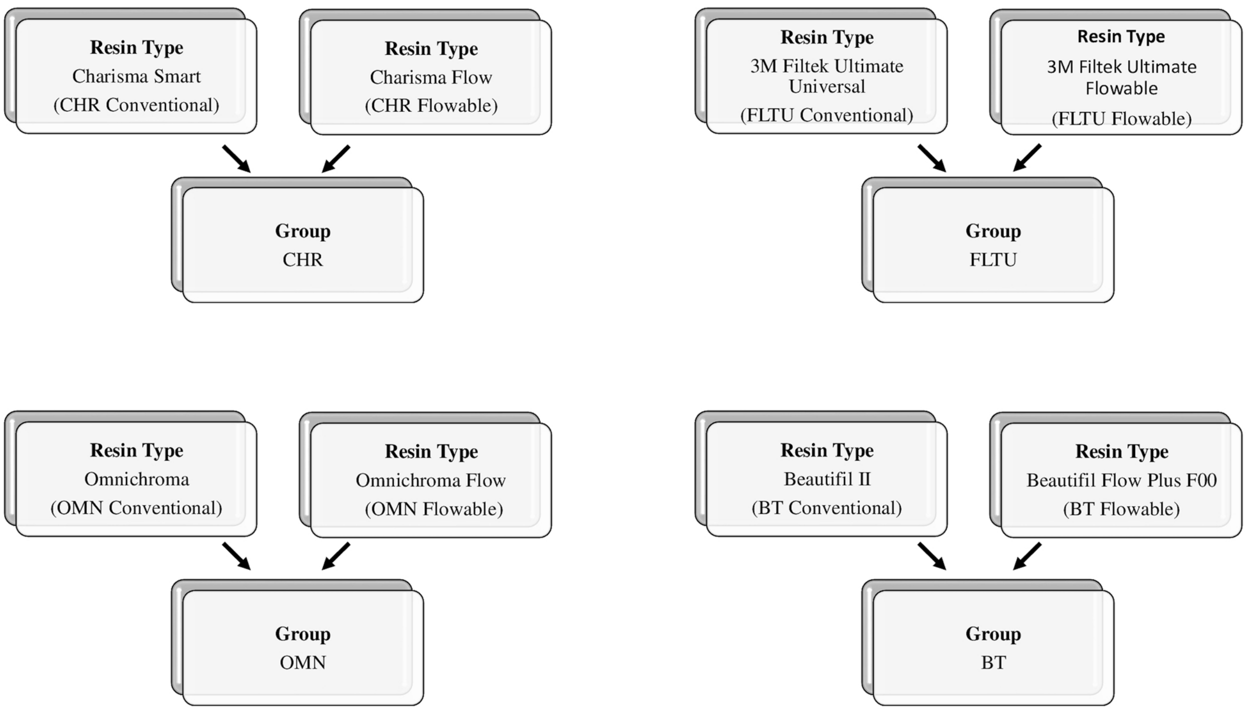

Fig. 1 The resin composite groups in this study

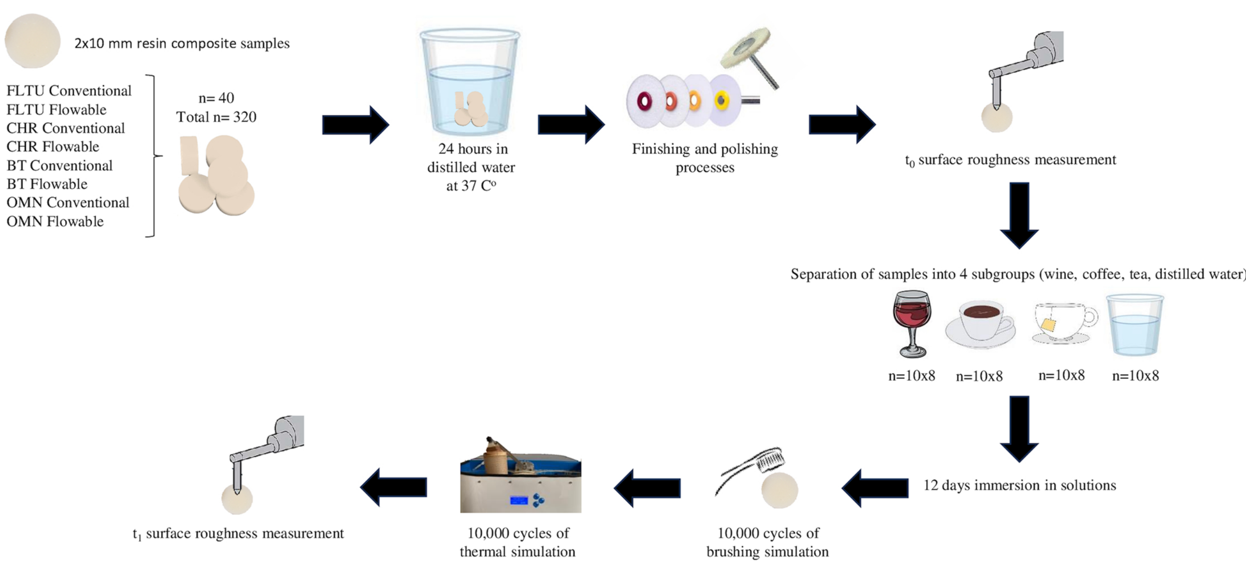

Fig. 2 Flow chart of the study

at 37 °C for 12 days to be suitable for intraoral conditions. The solutions in the tubes were renewed daily to prevent any bacterial or fungal contamination [13, 20]. Once the samples were removed from the solution they were washed for 5 s and made ready for brushing simulation.

Brushing simulation was performed using an MF-100 Toothbrushing Device (Mod Dental, Esetron Smart Robotechnologies, Ankara, Turkey), Colgate Extra Clean 1 + 1 (Colgate Palmolive, USA) toothbrush, and Sensodyne Promine Repair^{+} (GlaxoSmithKline, EU) toothpaste. The simulation was performed under a load of 250 g, with a circular motion of 15 mm diameter at a speed of 40 mm/sec. A total of 10,000 cycles of brushing simulation were performed [7, 12]. During the simulation, the toothpaste was diluted 1/3 by volume. For each sample, the toothbrush was changed, and new paste was used. Samples were removed from the brushing simulator, washed to remove paste residue, and prepared for placement in the thermal cycler.

A MTE-101 Thermal Cycle Device (Mod Dental, Esetron Smart Robotechnologies, Ankara, Turkey) was used for the thermal aging process. The device temperature values were set to 5–55 °C (± 2 °C), the waiting time in the hot and cold water tanks was 15 s each, the total waiting time in the tanks was 30 s, and the transfer time between the tanks was 10 s. 10,000 cycles of the thermal aging process were applied [13, 21].

The study was implemented to represent a 1 year of clinical conditions, assuming that 12 days of immersion in solutions is equivalent to 1 year of clinical beverage consumption [8, 22], 10,000 cycles of brushing simulate 1 year of an individual with a habit of brushing twice daily [7], and 10,000 thermal cycles simulates 1 year of sudden intraoral temperature fluctuations [13].

At the beginning (t_0) and the end of the aging procedures (t_1), surface roughness measurements were made from 3 different points of the samples with a Surftest SJ-400 (Mitutoyo, Japan) profilometer, and the average surface roughness values were obtained. During the measurements, the device was set in contact mode, the cut-off length was 0.25 mm, the evaluation length was 1.00 mm, and the probe speed was 0.5 mm/sec.

One sample from each group/resin type was examined with SEM (Hitachi Regulus 8230 FE-SEM, Japan) at time periods t_0 and t_1. The samples were treated with a 4 nm gold/palladium surface coating for surface conductivity before the examination. Images were taken from the samples at 3 kV at 1000 × magnification. Similarly, one sample from each group/resin type was examined with an atomic force microscope (Park Systems XE 100 Atomic Force Microscope, Korea) using a non-contact probe at time periods t_0 and t_1. 3D images were obtained from a 5000 µm, 5 × 5 field at a speed of 1 Hz. The samples for AFM and SEM images were randomly selected.

The data obtained in the study were analyzed with IBM SPSS V23. Comparisons were made according to resin composite groups (FLTU, CHR, BT, OMN), solution (wine, coffee, tea, distilled water), and types (flowable, conventional). The 3-way analysis of variance and Tukey test were used. The significance level was taken as $p < 0.05$.

Results

In the present study, the surface roughness values at time periods t_0 and t_1 were compared within themselves. The results obtained at time period t_0 are as follows:

Statistically significant differences were found between the mean surface roughness values according to groups ($p < 0.001$), types ($p < 0.001$) and group-type ($p < 0.001$) (Table 2.). The surface roughness values related to t_0 are shown in Table 3 and Fig. 3. According to the groups, the highest mean value was obtained in the BT group (0.18). There was no statistically significant difference between FLTU-CHR and CHR-OMN ($p > 0.05$). According to the types, the conventional type had a mean Ra value of 0.15, while the flowable type had a mean Ra value of 0.12. In group-type interactions, the highest mean value was obtained in the BT-conventional interaction (0.21) and the lowest mean value was obtained in the OMN-flowable interaction (0.09).

The results obtained in time period t_1 are as follows:

Statistically significant differences were found between the mean surface roughness values according to groups ($p < 0.001$), types ($p < 0.001$), solutions ($p = 0.003$), group-type interactions ($p < 0.001$), group-solution interactions ($p < 0.001$) (Table 4.). The surface roughness values related to t_1 are shown in Table 5. While the mean value obtained in the BT group (0.25) was higher than all other groups, no statistically significant difference was found between FLTU (0.12), OMN (0.11) and CHR (0.11) ($p > 0.05$). The mean value was 0.16 for the conventional type and 0.14 for the flowable type. According to the solutions, the highest mean value was obtained for tea and coffee. Statistically significant differences were found between distilled water-tea and distilled water-coffee ($p < 0.05$). Wine was statistically similar to all other solutions (Table 6.). According to the group-type interactions, the highest mean value was obtained in the

Table 2 Comparison of t_0 roughness values

	Total of squares	sd	Average of squares	F	*p*	KEK
Group	0.229	3	0.076	60.350	**< 0.001**	0.386
Type	0.054	1	0.054	42.480	**< 0.001**	0.129
Group*Type	0.061	3	0.02	16.110	**< 0.001**	0.144

KEK Partial Eta Squared, *sd* degree of freedom, *F* Analysis of variance ($p < 0.05$)

Table 3 Descriptive statistics of t_0 roughness values

Solution	Group	Type		Total
		Conventional	Flowable	
Wine	FLTU	0.11 ± 0.02	0.14 ± 0.04	0.12 ± 0.04
	CHR	0.11 ± 0.03	0.12 ± 0.03	0.11 ± 0.03
	BT	0.22 ± 0.05	0.15 ± 0.04	0.18 ± 0.05
	OMN	0.12 ± 0.04	0.08 ± 0.03	0.10 ± 0.04
	Total	0.14 ± 0.06	0.12 ± 0.04	0.13 ± 0.05
Coffee	FLTU	0.14 ± 0.04	0.14 ± 0.04	0.14 ± 0.04
	CHR	0.11 ± 0.03	0.12 ± 0.04	0.11 ± 0.04
	BT	0.22 ± 0.03	0.14 ± 0.04	0.18 ± 0.05
	OMN	0.12 ± 0.04	0.10 ± 0.01	0.11 ± 0.03
	Total	0.14 ± 0.06	0.13 ± 0.04	0.13 ± 0.05
Tea	FLTU	0.14 ± 0.05	0.11 ± 0.02	0.12 ± 0.04
	CHR	0.11 ± 0.03	0.13 ± 0.03	0.12 ± 0.03
	BT	0.21 ± 0.03	0.14 ± 0.03	0.18 ± 0.04
	OMN	0.15 ± 0.04	0.10 ± 0.03	0.12 ± 0.04
	Total	0.15 ± 0.05	0.12 ± 0.03	0.14 ± 0.05
Distilled Water	FLTU	0.13 ± 0.04	0.11 ± 0.04	0.12 ± 0.04
	CHR	0.15 ± 0.04	0.11 ± 0.03	0.13 ± 0.04
	BT	0.20 ± 0.04	0.14 ± 0.03	0.17 ± 0.05
	OMN	0.12 ± 0.05	0.09 ± 0.02	0.10 ± 0.04
	Total	0.15 ± 0.05	0.11 ± 0.03	0.13 ± 0.05
Total	FLTU	0.13 ± 0.04	0.12 ± 0.04	0.13 ± 0.04
	CHR	0.12 ± 0.03	0.12 ± 0.03	0.12 ± 0.03
	BT	0.21 ± 0.04	0.14 ± 0.03	0.18 ± 0.05
	OMN	0.13 ± 0.04	0.09 ± 0.02	0.11 ± 0.04
	Total	0.15 ± 0.05	0.12 ± 0.04	0.13 ± 0.05

BT-conventional interaction. While the values obtained in BT-conventional and BT-flowable interactions were statistically significantly different from all other interactions ($p < 0.05$), the mean values in all other interactions were not statistically significantly different ($p > 0.05$).

In the present study, surface roughness change values between t_0 and t_1 time periods were compared. Statistically significant differences were found in the mean surface roughness change values according to group ($p = 0$), solution ($p = 0.014$), solution-type interaction ($p = 0.019$) and group-type interactions ($p < 0.001$) (Table 7.). The surface roughness change values between t_0 and t_1 time periods are shown in Table 8 and Fig. 4. The mean value obtained in the CHR, FLTU and OMN groups differed from the BT group. While an increase was observed in the BT group, a decrease was observed in the others. While there was a statistically significant difference between distilled water and tea ($p < 0.05$), there was no statistically significant difference between the other solutions ($p > 0.05$).

SEM images taken from the group/resin type are shown in Figs. 5, 6, 7, 8 and 9. SEM images show residual material particles due to finishing and polishing processes and aging procedures. In particular, BT images taken at time t_1 show gaps and crevices due to aging procedures. These gaps and crevices resulted in a more irregular structure. AFM images taken from the group/resin type are shown in Figs. 10, 11, 12, 13 and 14.

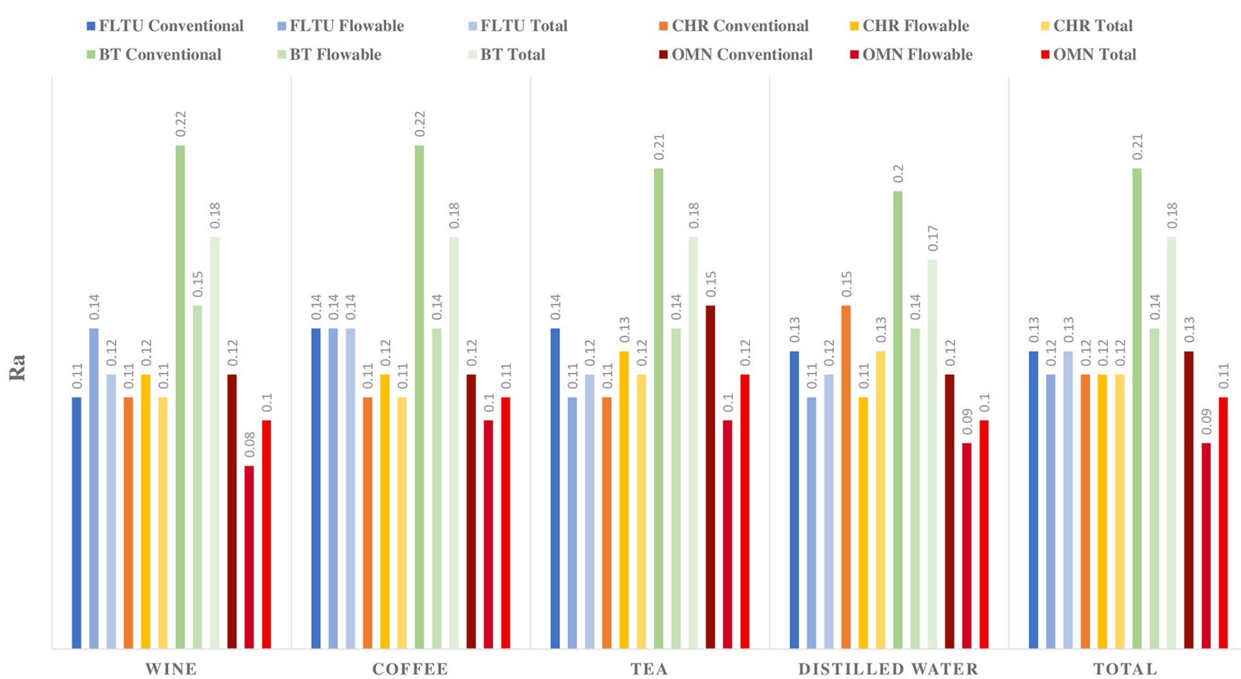

Fig. 3 Surface roughness values at time t_0

Table 4 Comparison of t_1 roughness values

	Total of squares	sd	Average of squares	F	*p*	KEK
Group	1.077	3	0.359	196.08	**< 0.001**	0.671
Type	0.032	1	0.032	17.69	**< 0.001**	0.058
Solution	0.026	3	0.009	4.68	**0.003**	0.046
Group*Type	0.141	3	0.047	25.74	**< 0.001**	0.211
Group*Solution	0.057	9	0.006	3.44	**< 0.001**	0.097
Type*Solution	0.012	3	0.004	2.10	0.10	0.021
Group*Type*Solution	0.022	9	0.002	1.33	0.22	0.04

KEK Partial Eta Squared, *sd* degree of freedom, *F* Analysis of variance ($p < 0.05$)

Table 5 Descriptive statistics of t_1 roughness values

Solution	Group	Type		
		Conventional	Flowable	Total
Wine	FLTU	0.10 ± 0.02	0.12 ± 0.04	0.11 ± 0.04
	CHR	0.10 ± 0.02	0.11 ± 0.03	0.10 ± 0.02
	BT	0.33 ± 0.06	0.22 ± 0.04	0.27 ± 0.08
	OMN	0.10 ± 0.05	0.09 ± 0.03	0.10 ± 0.04
	Total	0.16 ± 0.11	0.13 ± 0.06	0.15 ± 0.09
Coffee	FLTU	0.12 ± 0.03	0.14 ± 0.05	0.13 ± 0.04
	CHR	0.09 ± 0.02	0.11 ± 0.03	0.10 ± 0.03
	BT	0.31 ± 0.07	0.22 ± 0.04	0.26 ± 0.08
	OMN	0.11 ± 0.03	0.12 ± 0.04	0.11 ± 0.03
	Total	0.15 ± 0.1	0.15 ± 0.06	0.15 ± 0.08
Tea	FLTU	0.14 ± 0.06	0.11 ± 0.05	0.12 ± 0.06
	CHR	0.11 ± 0.03	0.13 ± 0.04	0.12 ± 0.04
	BT	0.30 ± 0.04	0.18 ± 0.05	0.24 ± 0.08
	OMN	0.15 ± 0.05	0.12 ± 0.04	0.14 ± 0.05
	Total	0.17 ± 0.09	0.14 ± 0.05	0.16 ± 0.07
Distilled Water	FLTU	0.12 ± 0.05	0.13 ± 0.05	0.12 ± 0.05
	CHR	0.11 ± 0.04	0.10 ± 0.03	0.11 ± 0.03
	BT	0.24 ± 0.07	0.19 ± 0.03	0.21 ± 0.05
	OMN	0.08 ± 0.02	0.09 ± 0.04	0.08 ± 0.03
	Total	0.14 ± 0.07	0.13 ± 0.05	0.13 ± 0.06
Total	FLTU	0.12 ± 0.04	0.13 ± 0.05	0.12 ± 0.05
	CHR	0.10 ± 0.03	0.11 ± 0.04	0.11 ± 0.03
	BT	0.29 ± 0.07	0.20 ± 0.04	0.25 ± 0.07
	OMN	0.11 ± 0.05	0.11 ± 0.04	0.11 ± 0.04
	Total	0.16 ± 0.09	0.14 ± 0.06	0.15 ± 0.08

Table 6 Comparison of solutions at time t_1

Solution	Mean
Tea	0.155000[a]
Coffee	0.149625[a]
Wine	0.145750[ab]
Distilled Water	0.130875[b]

Means that do not share share a letter are significantly different. ($p < 0.05$)

Discussion

In this study, it was concluded that the type of resin composite affects the surface roughness at time periods t_0 and t_1, but does not affect the surface roughness change values between time periods t_0 and t_1. Thus, the first null hypothesis was partially rejected. The second null hypothesis was rejected by concluding that the solution used affects the surface roughness. In the results obtained from all time periods, it was concluded that different resin composite brands, i.e. resin composite groups, affect the surface roughness. Thus, the third null hypothesis is rejected.

In the present study, Ra values lower than 0.2 µm, the threshold value for bacterial adhesion, were obtained in all composite groups at time t_0. In a study evaluating the surface roughness of composite resins with a microhybrid (G-aenial Anterior, GC Corporation, Leuven, Belgium), a nanohybrid (Harmonize, Kerr Corporation, Orange, CA, USA), and a supra-nano filler (Estelite Asteria, Tokuyama Dental, Tokyo, Japan) brushed with whitening toothpaste, G-aenial Anterior gave the highest surface roughness values before brushing [7]. This was associated with large filler particles. Similar to the present study where the OMN group with it's supra-nano filler obtained low RA values, Asteria, with the same filler type, gave low RA values [7]. The reason for the highest Ra value being found in the BT group in the present study may be that the large voids in the resin matrix structure form a rough structure and contain large filler particles.

In the present study, when the effects of all factors on Ra values at time t_1 were evaluated, it was concluded that resin composite groups had the highest effect, while group-type interaction was in second place. The effect of the solutions used on the surface roughness of the resin composite materials was found to be very small. Although there are studies in the literature showing that coffee and tea consumption change the surface roughness by causing ion precipitation and disruption in the resin matrix structure [23, 24], there are also studies that have not found a statistically significant

Table 7 Comparison of surface roughness change values between t_0 and t_1 time periods

	Total of squares	sd	Average of squares	F	*p*	KEK
Group	0.324	3	0.108	62.05	**0**	0.393
Type	0.003	1	0.003	1.55	0.214	0.005
Solution	0.019	3	0.006	3.59	**0.014**	0.036
Group*Type	0.034	3	0.011	6.43	**< 0.001**	0.063
Solution*Group	0.025	9	0.003	1.62	0.108	0.048
Type*Solution	0.018	3	0.006	3.37	**0.019**	0.034
Group*Type*Solution	0.006	9	0.001	0.36	0.954	0.011

KEK Partial Eta Squared, *sd* degree of freedom, *F* Analysis of variance ($p < 0.05$)

Table 8 Descriptive statistics of surface roughness change values between time periods t_0 and t_1

Solution	Group	Type		Total
		Conventional	Flowable	
Wine	FLTU	0.006 ± 0.022	0.017 ± 0.04	0.012 ± 0.032
	CHR	0.005 ± 0.036	0.008 ± 0.05	0.007 ± 0.042
	BT	-0.109 ± 0.062	-0.064 ± 0.038	-0.087 ± 0.055
	OMN	0.015 ± 0.037	-0.008 ± 0.043	0.004 ± 0.041
	Total	-0.021 ± 0.065	-0.012 ± 0.052	-0.016 ± 0.059
Coffee	FLTU	0.020 ± 0.032	-0.006 ± 0.055	0.007 ± 0.046
	CHR	0.020 ± 0.036	0.014 ± 0.033	0.017 ± 0.033
	BT	-0.086 ± 0.063	-0.071 ± 0.059	-0.079 ± 0.06
	OMN	0.011 ± 0.038	-0.02 ± 0.041	-0.005 ± 0.041
	Total	-0.009 ± 0.062	-0.021 ± 0.056	-0.015 ± 0.059
Tea	FLTU	0.001 ± 0.027	-0.004 ± 0.041	-0.002 ± 0.034
	CHR	-0.004 ± 0.027	0.001 ± 0.034	-0.002 ± 0.03
	BT	-0.091 ± 0.032	-0.036 ± 0.036	-0.064 ± 0.044
	OMN	0 ± 0.049	-0.027 ± 0.038	-0.014 ± 0.045
	Total	-0.24 ± 0.052	-0.017 ± 0.039	-0.02 ± 0.046
Distilled Water	FLTU	0.016 ± 0.042	-0.016 ± 0.053	0 ± 0.049
	CHR	0.033 ± 0.041	0.013 ± 0.028	0.023 ± 0.036
	BT	-0.035 ± 0.064	-0.048 ± 0.023	-0.042 ± 0.047
	OMN	0.041 ± 0.036	-0.003 ± 0.031	0.019 ± 0.04
	Total	0.014 ± 0.054	-0.015 ± 0.038	0 ± 0.05
Total	FLTU	0.011 ± 0.031	-0.002 ± 0.047	0.004 ± 0.04
	CHR	0.014 ± 0.037	0.009 ± 0.036	0.011 ± 0.036
	BT	-0.08 ± 0.061	-0.055 ± 0.042	-0.068 ± 0.054
	OMN	0.017 ± 0.042	-0.015 ± 0.038	0.001 ± 0.043
	Total	-0.01 ± 0.06	-0.016 ± 0.047	-0.013 ± 0.054

change [25, 26]. As mentioned in the literature, alcohol can affect the composite matrix surface and may cause surface softening [27, 28]. In the present study, among the solutions, the highest surface roughness values were obtained in tea and coffee, while the lowest surface roughness values were obtained in distilled water. Wine was statistically similar to all other solutions. In tea and coffee, the particles in their structure may have accumulated on the surfaces of the samples and caused changes in the resin matrix structure, leading to roughness. Another reason could be that hot beverages such as coffee and tea change the thermal properties of resin composites and increase the surface roughness. However, in this study, coffee and tea were used at 37 °C. In fact, since not only solution effects were evaluated in the present study, but also other aging methods were used, we may not be able to see the direct effect of the solutions on the results we obtained.

In a different study, the effect of beverages and brushing on surface roughness was evaluated using Filtek Z350 (3M ESPE, St. Paul, MN, USA) and it was concluded that it negatively affected the surface roughness [28]. Since no intermediate measurements were made after brushing in the present study, it is not possible to make any clear statements about the negative effect of brushing on surface roughness.

In the present study, when the amount of change between the surface roughness values at time periods t_0 and t_1 was compared, the resin composite groups showed the greatest effect on the change. While a decrease was observed in the Ra value obtained in the CHR, FLTU, and OMN groups, an increase was observed in the BT group. In another study, it was concluded that low pH solutions, such as coffee and tea, remove residual monomers in the resin matrix structure, causing dissolution and possible re-precipitation on the surface, resulting in a smoother surface. This was related to the pH of the solution as well as water absorption, the solubility parameter, and resin matrix structure [8]. In the present study, a possible reason for the decrease in surface roughness in the other resin composite groups except BT may be the low pH solutions we used. A study has evaluated the effect of brushing and artificial aging on the surface roughness of restorative materials. Since the particle size removed from the surface by brushing will be different in composites with different filler contents, composite particle size and surface roughness were correlated in that

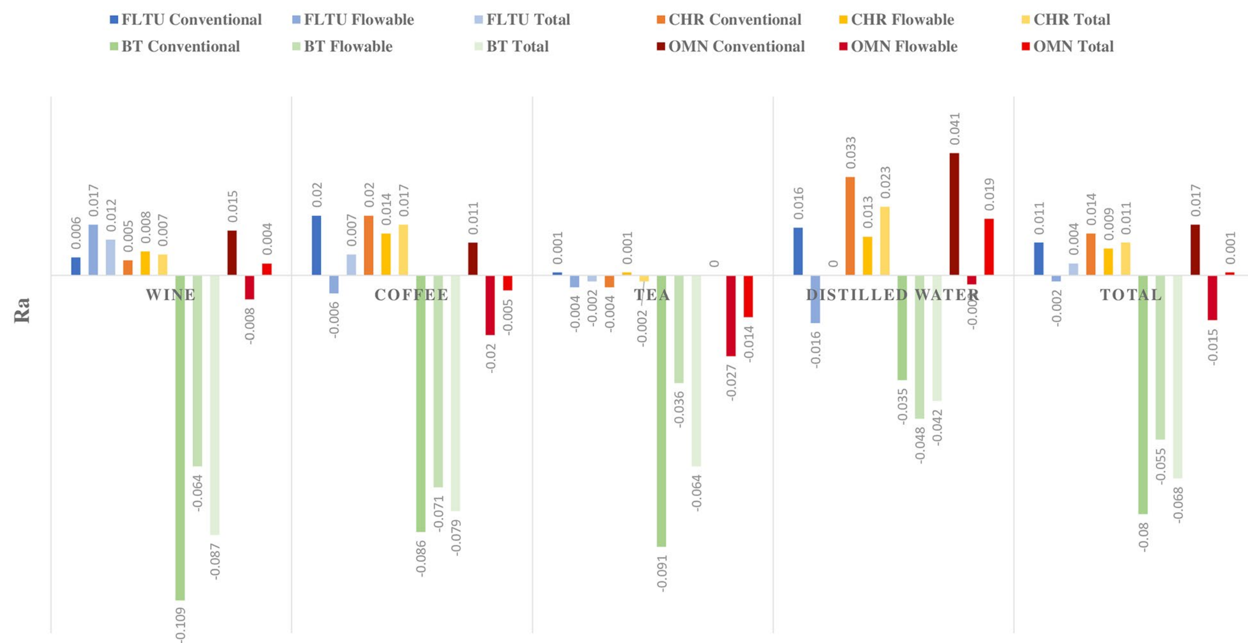

Fig. 4 Surface roughness change values between time periods t_0 and t_1 according to solutions

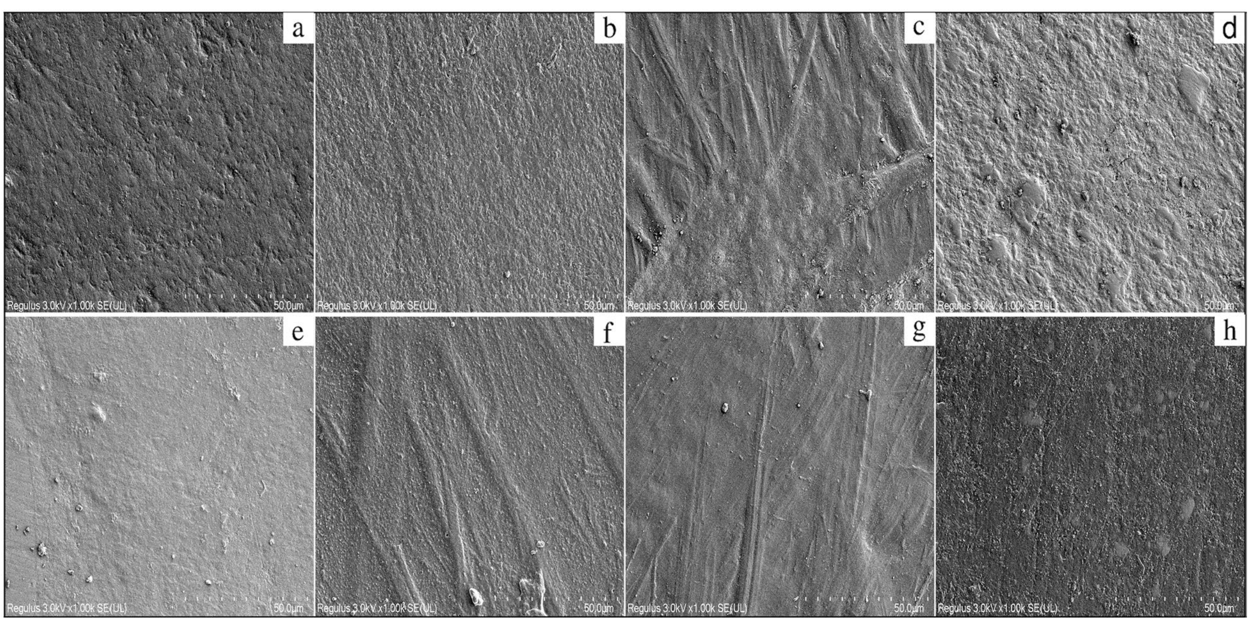

Fig. 5 2D images of surface topography by SEM at time t_0. **a** FLTU conventional; **b** CHR conventional; **c** OMN conventional; **d** BT conventional; **e** FLTU flowable; **f** CHR flowable; **g** OMN flowable; **h** BT flowable

study. No significant difference was obtained in terms of surface roughness in the samples subjected to aging after mechanical brushing [29]. In different studies using 4 different bulk-fill resin composites, the samples were kept in thermal cycling, heptane, citric acid, and ethanol, or only in chemical solutions. According to the results of the studies, no significant difference was obtained between the initial and final surface roughness values in the bulk-fill restorative materials except for the Beautifil Bulk Restorative, in which the surface

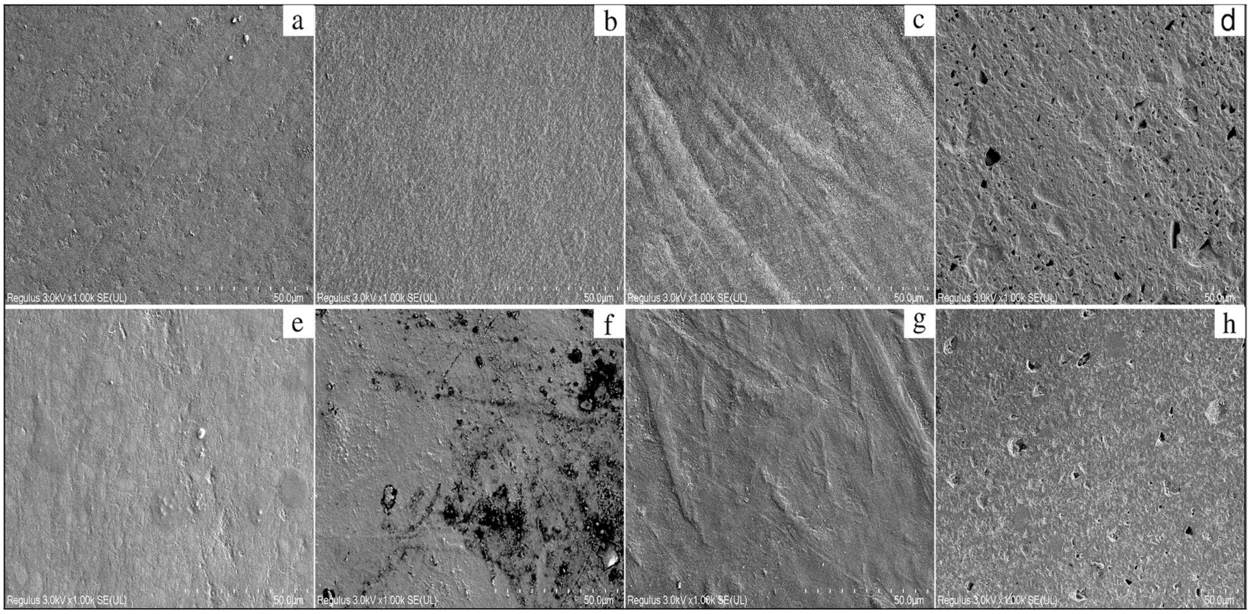

Fig. 6 2D images of the surface topography of wine immersed samples by SEM at time t_1. **a** FLTU conventional; **b** CHR conventional; **c** OMN conventional; **d** BT conventional; **e** FLTU flowable; **f** CHR flowable; **g** OMN flowable; **h** BT flowable

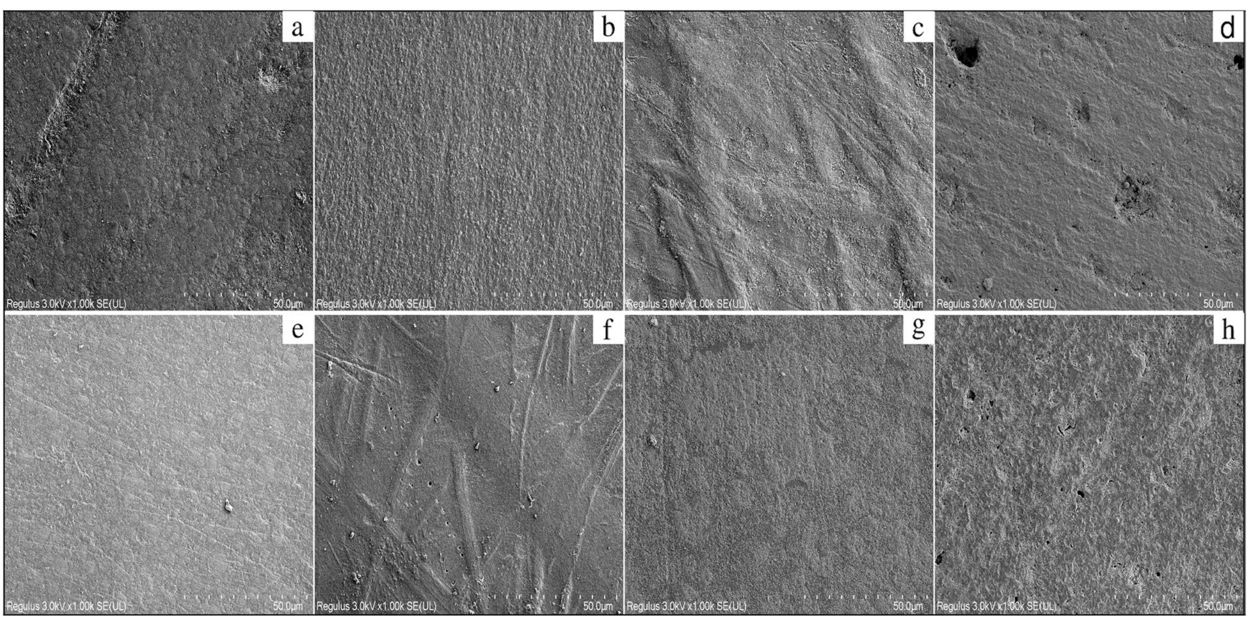

Fig. 7 2D images of the surface topography of coffee immersed samples by SEM at time t_1. **a** FLTU conventional; **b** CHR conventional; **c** OMN conventional; **d** BT conventional; **e** FLTU flowable; **f** CHR flowable; **g** OMN flowable; **h** BT flowable

roughness values increased when compared to the initial values and were attributed to the size of the S-PRG filler particle [30, 31]. In the present study, the possible reason for the increase in surface roughness in the BT groups may be the size of the SPR-G filler particles and the removal of larger particles from the surface by brushing.

In a different study, samples obtained from microhybrid (Filtek A110, 3M ESPE), hybrid (Filtek Z250, 3M ESPE), and flowable (Filtek Flow, 3M ESPE) resin composites

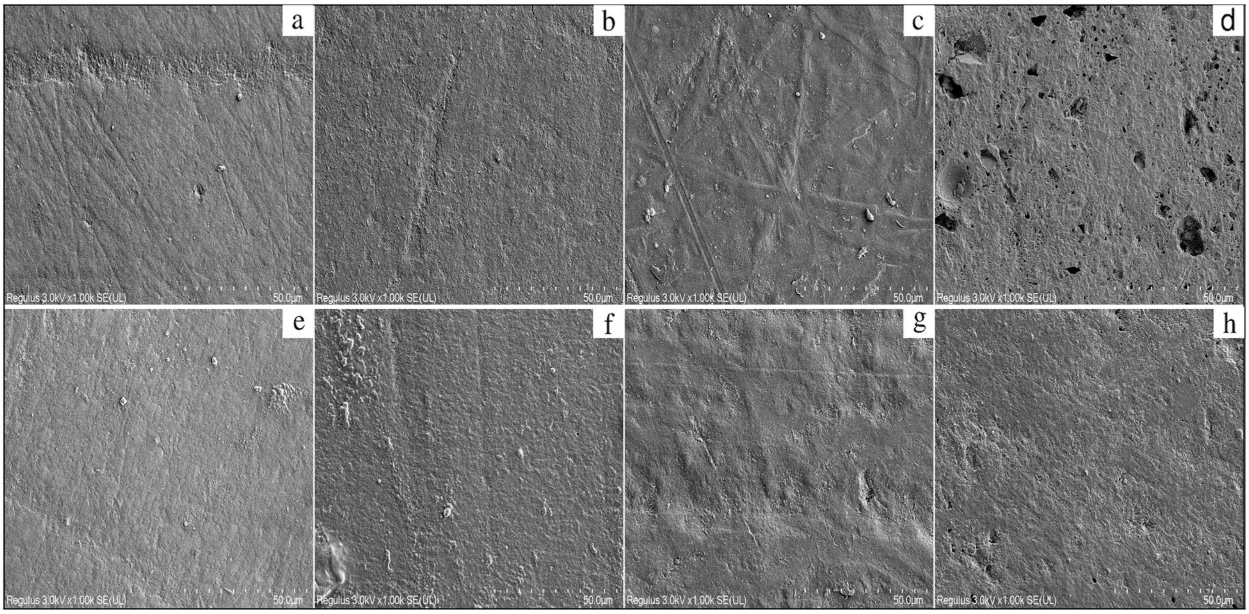

Fig. 8 2D images of the surface topography of tea immersed samples by SEM at time t_1. **a** FLTU conventional; **b** CHR conventional; **c** OMN conventional; **d** BT conventional; **e** FLTU flowable; **f** CHR flowable; **g** OMN flowable; **h** BT flowable

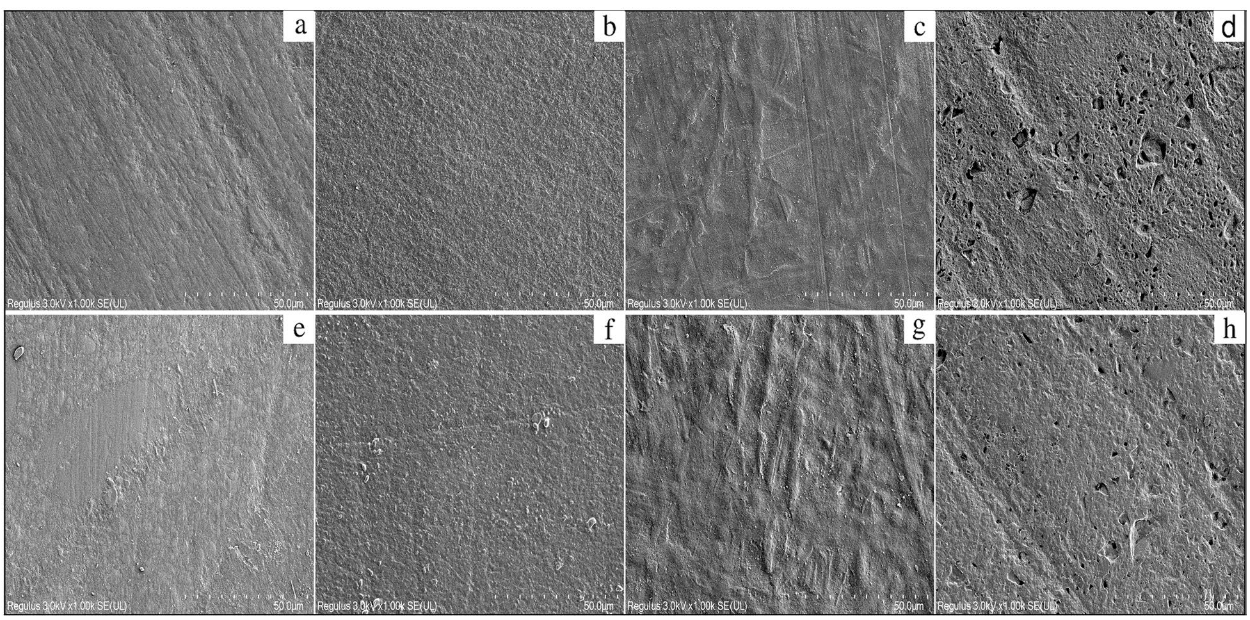

Fig. 9 2D images of the surface topography of distilled water immersed samples by SEM at time t_1. **a** FLTU conventional; **b** CHR conventional; **c** OMN conventional; **d** BT conventional; **e** FLTU flowable; **f** CHR flowable; **g** OMN flowable; **h** BT flowable

were immersed in cola, artificial saliva, and coffee, and the highest Ra values were obtained from Filtek A100, while similar results were found for the other two composites [32]. In the present study, lower Ra values were obtained from flowable resin composites compared to conventional resin composites. The resin matrix structure, filler type, and filler amount can be shown as the reason for the lower Ra values obtained in flowable resin composites. In addition, more homogeneous filler distributions may also be a possible reason. In another

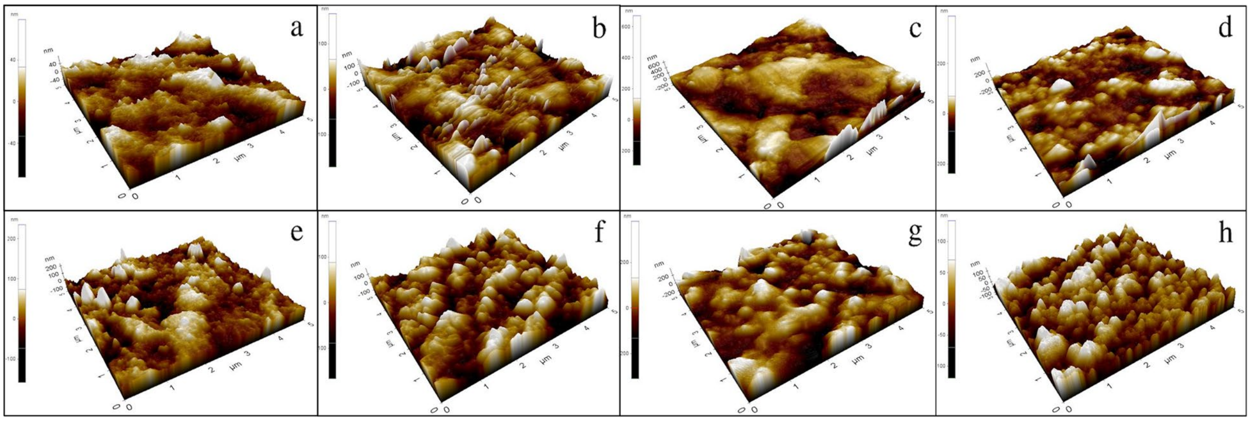

Fig. 10 3D images of the surface topography by AFM at time t_0. **a** FLTU conventional; **b** CHR conventional; **c** BT conventional; **d** OMN conventional; **e** FLTU flowable; **f** CHR flowable; **g** BT flowable; **h** OMN flowable

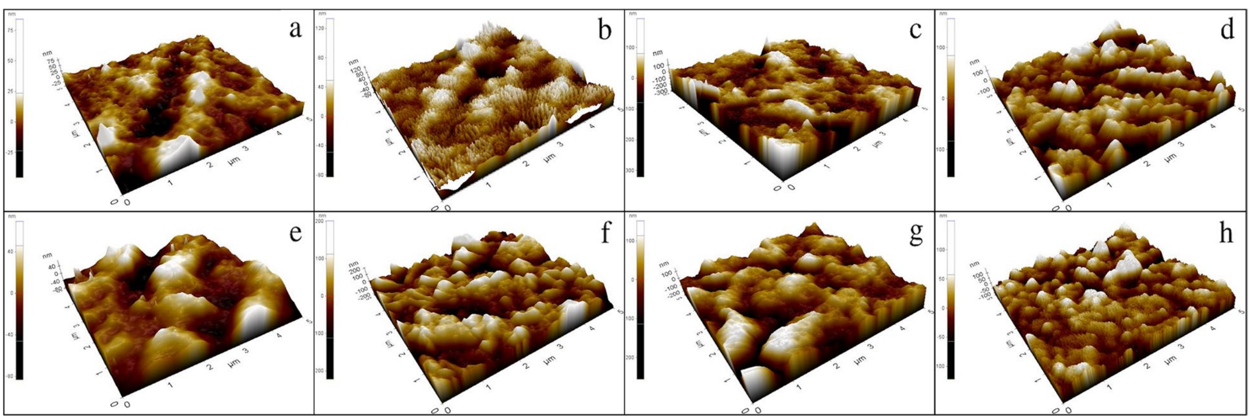

Fig. 11 3D images of the surface topography of wine immersed samples by AFM at time t_1. **a** FLTU conventional; **b** CHR conventional; **c** BT conventional; **d** OMN conventional; **e** FLTU flowable; **f** CHR flowable; **g** BT flowable; **h** OMN flowable

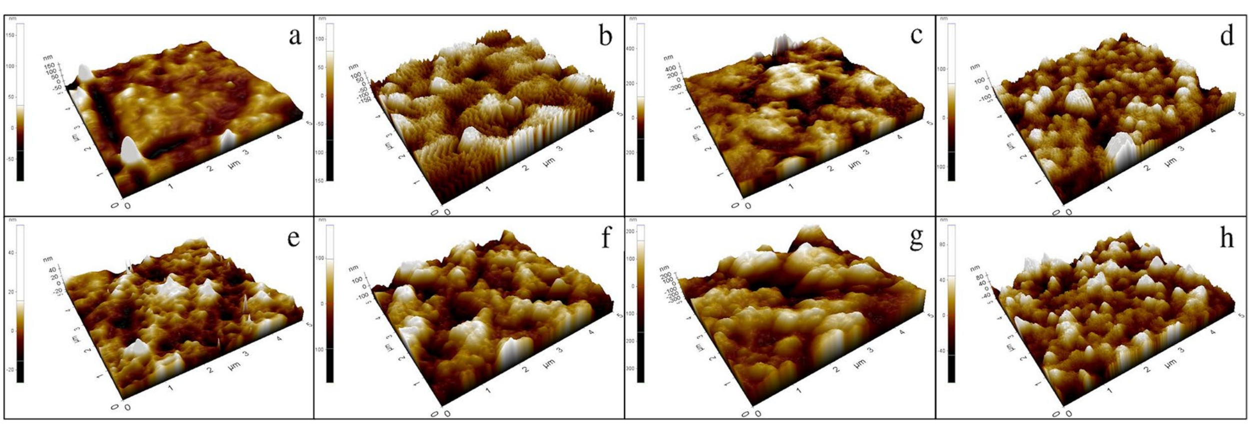

Fig. 12 3D images of the surface topography of coffee immersed samples by AFM at time t_1. **a** FLTU conventional; **b** CHR conventional; **c** BT conventional; **d** OMN conventional; **e** FLTU flowable; **f** CHR flowable; **g** BT flowable; **h** OMN flowable

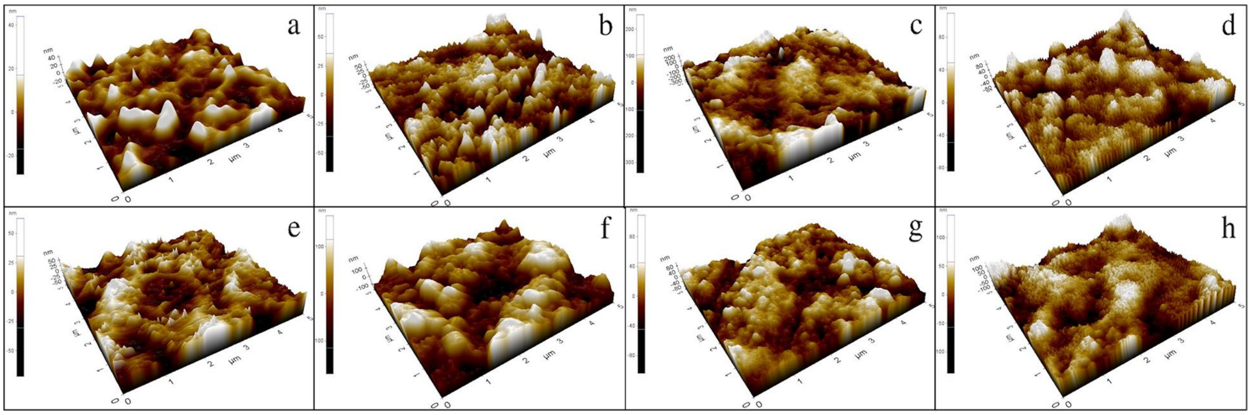

Fig. 13 3D images of the surface topography of tea immersed samples by AFM at time t_1. **a** FLTU conventional; **b** CHR conventional; **c** BT conventional; **d** OMN conventional; **e** FLTU flowable; **f** CHR flowable; **g** BT flowable; **h** OMN flowable

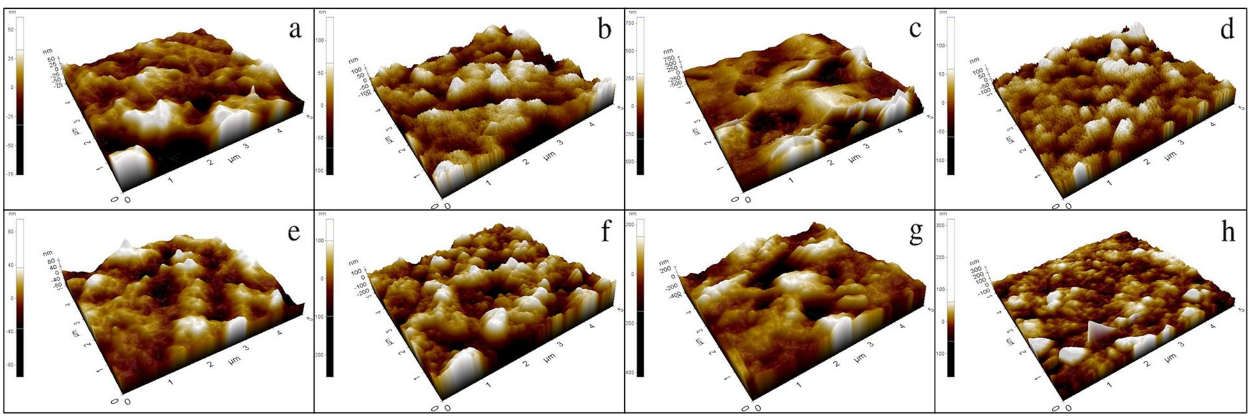

Fig. 14 3D images of the surface topography of distilled water immersed samples by AFM at time t_1. **a** FLTU conventional; **b** CHR conventional; **c** BT conventional; **d** OMN conventional; **e** FLTU flowable; **f** CHR flowable; **g** BT flowable; **h** OMN flowable

study, when the surface roughness of monochromatic composite resins was evaluated, it was found that flowable resin composites with the same supra-nano spherical filler content had lower Ra values. This was attributed to the homogeneous spherical structure [33]. The result obtained in the study is similar to the present study. In the present study, for composites with the same material type and filler content but with different filler rations, the flowable resin composites in both OMN and BT groups showed lower Ra values at time t_0 and the BT flowable resin composites showed similarly lower Ra values at time t_1. The flowable and conventional resin composites selected for the CHR group have different material types and particle size as well as different filler ratios. This may have influenced the results obtained.

When the 24-h, 7, 30, and 60-day results were evaluated, it was concluded that the surface roughness increased in the first 7 days and decreased significantly in the other measurements [32]. In the present study, similarly, the reason for the decrease in Ra values in the CHR, FLTU, and OMN groups may be the use of a 12-day retention time in the solutions. In a different study where immersion in solutions (saliva, tea, red wine) and thermal cycling procedures were applied to simulate a 1-year clinical condition, no significant difference in surface roughness was found after 10,000 thermal cycles [8]. In another study, the effect of thermal cycles on the surface roughness of nanofil, microfil, and microhybrid composites was evaluated. In general, it was concluded that the application of 3,000 thermal cycles increased the surface roughness of resin composites, while there was a tendency to decrease surface roughness in all groups after 10,000 thermal cycles. The preservation of the smooth surface of resin composites has been attributed to the organic composition of the material [34]. In the present study, another reason for the decrease in Ra values in the CHR, FLTU, and OMN groups may be the application of 10,000 cycles of thermal aging, similar to the literature.

The most important limitation of the present study is that this is an in vitro study. Under clinical conditions, colorant food consumption, brushing activity and temperature changes are in a continuous cycle over a 1-year period. However, in the present study, in order to simulate the 1-year clinical situation, first the immersion in solutions, then the brushing simulation and finally the thermal cycling procedures were applied, and these procedures were performed sequentially in blocks. Another limitation of the present study is that surface roughness measurements were made only at time periods t_0 and t_1. The fact that the measurements were not repeated at the transitions to immersion in solutions, brushing, and thermal cycling applications may not fully explain the reason for the results obtained. Considering the limitations of the present study, more long-term studies evaluating the effects of artificial aging are needed.

Conclusion

Within the limitations of the present study, it was concluded that composite groups had the most influence on the surface roughness of resin composites, while solution and the composite type had less influence. After the aging procedures, FLTU, CHR, and OMN maintained their surface structure, not exceeding the threshold value of 0.2 µm for bacterial adhesion.

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Not applicable.

Authors' contributions
All authors contributed to the study conception and design. Material preparation and data collection were performed by ZB. Data analysis and interpretation were performed by ZB, ÖÇ, and HT. Supervision was provided by BCY. The first draft of the manuscript was written by ZB and revised by ÖÇ. All authors read and approved the final version of the manuscript.

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Availability of data and materials
All data analyzed during this study are included in the article in the form of tables. Raw data supporting the findings of this study are available from the corresponding author upon reasonable request.

Declarations

Ethics approval and consent to participate
Not applicable.

Consent for publication
Not applicable.

Competing interests
The authors declare no competing interests.

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