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RESEARCH ARTICLE

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An evaluation of surface roughness after staining of different composite resins using atomic force microscopy and a profilometer

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Abstract

Aim: The aim of this study was to compare the surface roughness of different composite resins using atomic force microscope (AFM) and a profilometer after storage in different solutions.

Materials and methods: Eight different composite resins were used in this study. Twenty specimens of each composite resin material were prepared using a 2-mm thick and 8-mm diameter stainless steel mold. After the composites had been placed in the mold, they were polymerized with a LED curing unit. The surfaces of all specimens were polished using aluminum oxide discs, and the specimens were then divided into four groups. The specimens in the experimental groups were stored in cola, coffee, or red wine, while the control group was stored in distilled water. Specimen surface roughness was examined after 30 days using an AFM and a profilometer, and the data obtained were subjected to analysis.

Results: Evaluation of the surface roughness of composite resins using a profilometer revealed no statistically significant difference between the groups, but significant differences were found using the AFM. The mean surface roughness of nanohybrid composites was lower than that of microhybrid composites.

Conclusions: The surface roughness of the composite resins varies with storage in different solutions, depending on the organic matrix structure and inorganic fillers of the resin.

KEYWORDS

AFM, composite resin, profilometer and surface roughness

1 | INTRODUCTION

Recent advances in the physical, cosmetic, and mechanical properties of dental composites have significantly increased their clinical use. Composite resins have become usable, not only in the anterior, but also in the posterior region due to changes in resin matrix content, and improvements in composite particle structure and size, and surface properties.(Abdelaziz & Saleh, 2018; Korkmaz, Ozel, Attar, & Aksoy, 2008) One of the most important properties of composite resins, which achieve significant cosmetic success, is surface roughness. The surface smoothness of composite resins is associated with plaque accumulation, the health of the periodontal tissues, discoloration and optical properties, water absorption, and surface damage in the restoration (Janus, Fauxpoint, Arntz, Pelletier, & Etienne, 2010). Secondary caries and pulpal irritation may also occur due to plaque, bacterial involvement, and microleakage. Surface roughness is also associated with patient comfort, because the tongue can detect roughness exceeding 0.3 μm. This may cause the patient to be uncomfortable with the restoration (Jones, Billington, & Pearson, 2004).

The surface roughness of composite resins depends on the filler size, shape, and content, the monomer type, and the degree of polymerization. Advances in composite resin technology, and in the surface

smoothness and polishing properties of composite resins, are occurring on a frequent basis (Ereifej, Oweis, & Eliades, 2013). Composites with different filler contents and sizes are used in dental restorations. The most commonly employed composites are microhybrid composites, which provide optimum physical, mechanical, and optical properties in dental restorations (Janus et al., 2010). In recent years, nanocomposites have been developed using nanoparticle technology in composite resin production. These smaller particle-containing composites exhibit good mechanic and surface properties. In addition, composites with ormocer have also been produced in order to increase the mechanical and physical properties of the resin (Cavalcante, Schneider, Silikas, & Watts, 2011; Egilmez, Ergun, Cekic-Nagas, Vallittu, & Lassila, 2012).

Composite resin restorations are subjected to numerous traumatic effects, such as masticatory forces in the mouth, stress-induced forces, plaque, and bacterial effects, and the effects of food and beverages. Chemical beverages can cause wear and degradation on the surface of composite resin restorations. In general, acidic beverages cause erosive effects, while alcoholic beverages may cause deterioration in the polymer matrix structure. In such cases, the deterioration that may occur on the restoration surface leads to failure of the restoration by damaging its mechanical properties, cosmetic appearance, and surface integrity (Bansal, Acharya, & Saraswathi, 2012).

Various methods can be employed to measure the surface roughness of composite resins, such as optical and scanning electron microscopy, contact profilometry, laser noncontact profilometry, and the atomic force microscope (AFM; Ereifej et al., 2013). Profilometers employ several optical principles, such as interferometry, focus detection and light scattering. These devices have a wide amplitude measurement range, and are therefore frequently used to measure the surface hardness of materials (Yilmaz & Ozkan, 2010). The AFM is a highly effective instrument for measuring and documenting the structural character of a material, and permits the surface topography of composite resins to be visualized at a high spatial resolution. With the advantages of their imaging properties and the fact that they do not cause deformation on the sample surface, these devices have become increasingly used in the evaluation of the surface properties of dental materials (Varanda, Do Prado, Simao, & Dias, 2013). Even minimal roughness on the surface of composite resins leads to plaque accumulation and damage to the structural properties of resin (Han et al., 2014). Surface roughness measurement should therefore be as precise as it is sensitive.

The aim of this study was to evaluate the effects of different beverages on the surface roughness of different dental composites using a profilometer and an AFM. The null hypothesis was that there would be no difference in surface roughness following storage of composite resins in chemical beverages.

2 | MATERIALS AND METHODS

2.1 | Specimen preparation

Eight different composite materials, shown in Table 1, were used in this study. Twenty specimens of each material were prepared using a 2-mm thick and 8-mm diameter stainless steel mold. A2 color tone was selected for each composite. Teflon molds were positioned onto celluloid matrix strips, and the composite resins were placed into the molds using an incremental technique. After the composite resins had been placed in the molds, celluloid matrix strips, and the microscope glass were applied over the top surface of the composite resins using finger pressure, and the excess composites were removed. Each sample was light-cured for 40 sn using a LED curing unit (Elipar Freelight II, 3 M ESPE, St. Paul, MN) at a light intensity of 1,000 mw/cm^2 from the top and bottom surfaces. The light intensity of the LED curing unit was checked with a radiometer (Hilux Ultra Plus Curing Units, Benlioğlu Dental) every three specimens. The specimens were stored in distilled water for 24 hr and then polished with medium, fine and superfine aluminum oxide discs (Sof-Lex, 3M ESPE, St. Paul, MN). After polishing, the specimens were divided into four groups, and the specimens from each group were stored in different solutions. The first group was stored in distilled water (control group), the second group in coffee solution (Nescafe Classic, Bursa, Turkey—2 g of coffee dissolved in 150 mL of boiling water), the third group in cola (The Coca-Cola Company, Turkey), and the fourth group in red wine (DLC Öküzgözü 2009, Doluca, Istanbul, Turkey). The specimens in the experimental groups were placed into staining solution for 3 hr each day and were kept in distilled water at all other times. The surface roughness of the specimens was examined after 30 days of storage.

2.2 | AFM roughness evaluation

Mean surface roughness was assessed with tapping mode atomic force microscopy (Nanomagnetics Instruments, Turkey) in air. The specimen surface was scanned using an Al-coated highly doped silicon tip with 10-nm mean nominal radi (Nanosensors, PPP-NCHR), at a frequency of 330 Hz, measurements being performed in nanometers (nm), and a nominal spring constant of 42 N/m. Three areas were randomly selected from each sample for measurement, and two-dimensional (2D) and three-dimensional (3D) AFM images were then taken at 10 μm × 10 μm planes, at 512 × 512 resolutions, and at a scan rate of 1.97 Hz in tapping mode. The mean roughness (Ra) was calculated for each sample using the formula

$$\mathrm{Ra} = \frac{1}{LxLy}\int_0^{Ly}\int_0^{Lx} |\, f(x,y)|dxdy,$$

where f (x, y) is the surface relative to the center plane, and Lx and Ly are the dimensions of the surface (Silikas, Watts, England, & Jandt, 1999). Data were recorded after measurement.

2.3 | Profilometer roughness evaluation

The surface roughness of the specimens was evaluated using contact mode profilometer after the AFM evaluation. Surface roughness values were obtained using the 5-μm radius diamond tip of the

TABLE 1 Details of the investigated restorative materials

			Content			
Product	**Manufacturer**	**Type**	**Organic matrix**	**Fillers**	**Particle size**	**Filler load (wt-v)%**
Clearfil majestry Esthetic	Kuraray Medical, Okayama, Japan	Nanohybrid	Bis-GMA, aromatic dimethacrylate	Silanated barium glass, filler, prepolymerized nano-organic filler	0.7 μm, 20 nm	78–66
Gradia Direct Anterior	GC Dental Products Corp, Japan	Microhybrid	Bis-GMA, UDMA	Prepolymerized organic fillers and silica	2.5 μm	78–66
Grandio	VOCO GmbH Cuxhaven Germany	Nanohybrid	Bis-GMA, TEGDMA, UDMA	Glass–ceramic (microfiller) Si02 (nanofiller)	1 μm 20–60 nm	87–71.4
Valux Plus	3M ESPE	Hybrid	Bis-GMA, TEGDMA	Silanetreated ceramic	0.01–3.5 μm	80–71
Grandio Flow	VOCO GmbH Cuxhaven Germany	Flowable Nanohybrid	Bis-GMA, TEGDMA,	Silicium dioxide, glass ceramic particles	SiO_2-nano particles (40 nm) glass fillers (1 μm)	80.2–65.7
Admira	VOCO GmbH Cuxhaven Germany	Ormocer	Ormocer, Bis-GMA, UDMA	Ba-Al-B-silicate glass, SiO_2	0.7 μm	78–56
Filtek Z250	3M ESPE, St. Paul, MN	Microhybrid	Bis-GMA Bis-EMA UDMA TEGDMA	Zirconium/silica	0.6 μm	82–78
Admira Flow	VOCO GmbH Cuxhaven Germany	Microhybrid	Bis-GMA, TEGDMA, UDMA	Bariumaluminiumsilicate glass, lithium aluminum silicate glass ceramic	0.7 μm	77–56

Abbreviations: Bis-GMA, bisphenol A glycidyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

profilometer (Surtronic 25, Taylor Hobson, Leicester, UK),with a cut-off value of 0.25 mm, a transverse length of 1.25 mm, a range of 100 μm, and at a speed of 1 mm/s. This procedure was performed on three different sites, and mean Ra values were obtained for each sample.

2.4 | Statistical analysis

Data obtained from AFM and profilometer measurements were recorded and subjected to statistical analysis on SPSS 18 software (SPSS Inc, Chicago, IL). The surface roughness of different composites, AFM measurements and profilometer measurements were compared using Two-Way ANOVA and Tukey's post hoc test at $\alpha = .05$.

3 | RESULTS

Eight different composite resins were kept in four different storage solutions for 30 days, and the surface roughness of the specimens was measured using an AFM and a profilometer.

The surface roughness measurements obtained using the profilometer are shown in Figures 1 and 2. The mean surface roughness values of the Filtek Z250 specimens were higher than the other groups. The lowest mean surface roughness values were seen in Grandio flow specimens (Figure 1). There were no significant differences between the surface roughness values of specimens kept in different storage solutions for each composite ($p > .05$; Table 2).

The surface roughness measurement results obtained using the AFM are shown in Figures 3 and 4. When the surface roughness of the specimens were examined by AFM, statistically significant differences were found in both composite resins and storing solutions ($p < .05$; Table 3). The highest mean surface roughness values were observed in Admira Flow and Filtek Z250 samples, while the lowest values were observed in Valux Plus samples (Figure 3). The lowest mean surface roughness values were found in specimens stored in cola and the highest mean surface roughness values were found in specimens stored in red wine (Figure 4). AFM imaging specimens for each group are shown in Figure 5.

4 | DISCUSSION

In this study, composite resin specimens were stored in different storage solutions for 30 days, and surface roughness was measured using two different methods. Restorative materials used in dentistry mimic natural tooth structures. The physical, mechanical, and esthetic properties of restorative materials are expected to exhibit similar

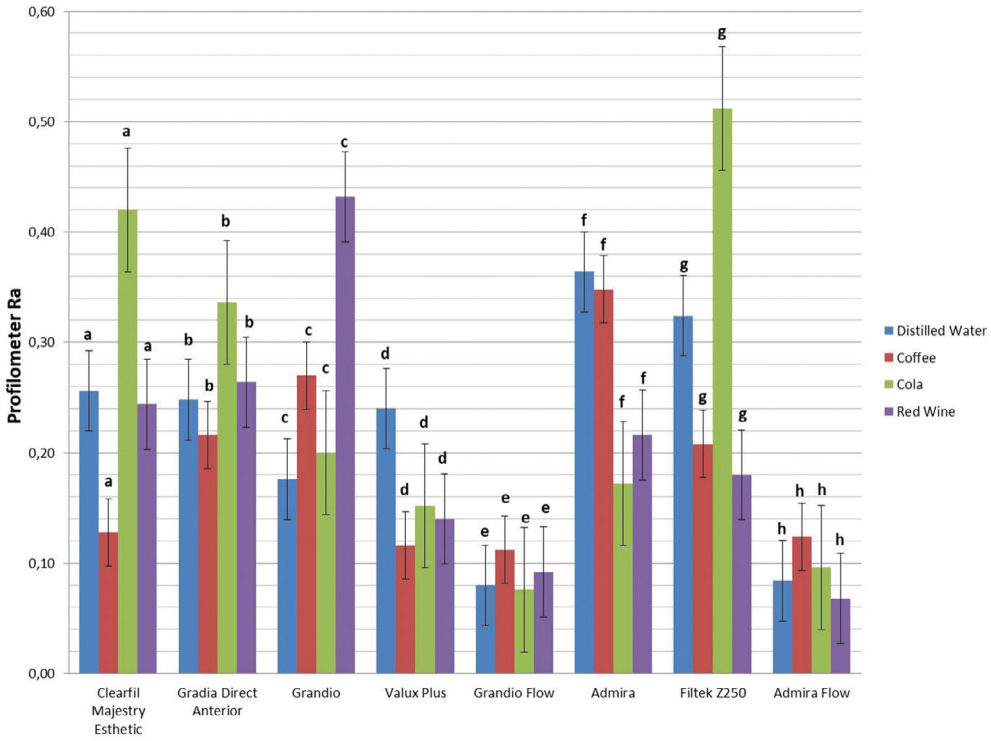

FIGURE 1 Results of surface roughness analysis by composite type by using a profilometer. Different letters indicate a statistically significant difference between the groups [Color figure can be viewed at wileyonlinelibrary.com]

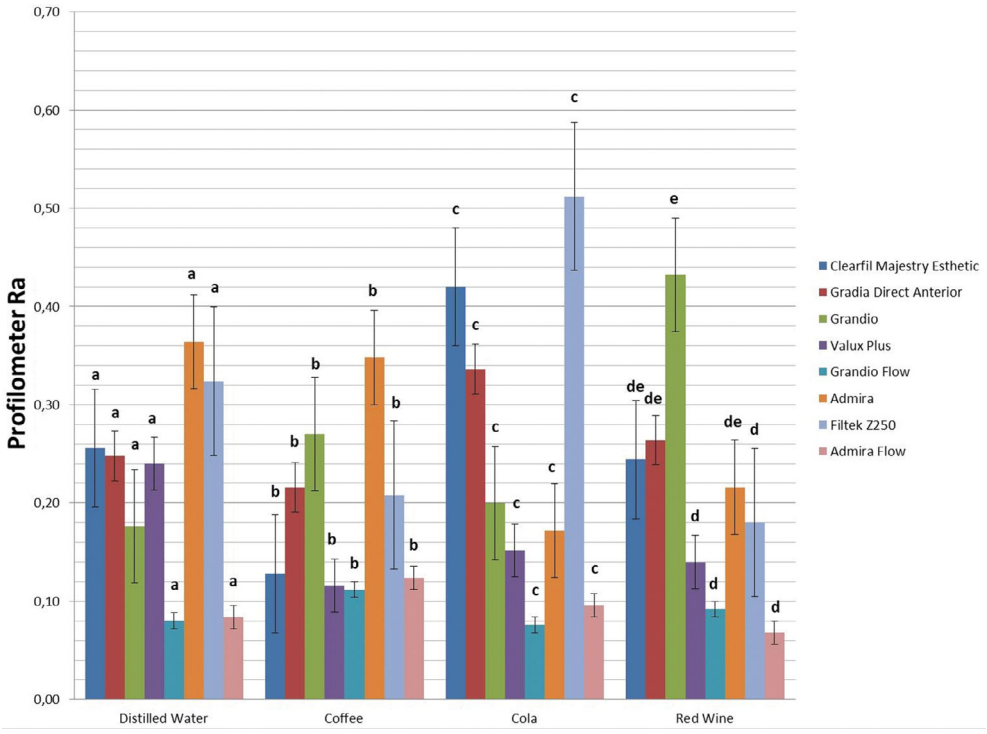

FIGURE 2 Results of surface roughness analysis by storage solutions by using a profilometer. Different letters indicate a statistically significant difference between the groups [Color figure can be viewed at wileyonlinelibrary.com]

properties to those of dental tissues and contact enamel surfaces (Ferreira Rde, Lopes, & Baratieri, 2004). The absolute arithmetic mean of the profile fluctuations that occur upward from the center of the material is known as surface roughness (Valicek et al., 2019). As the surface roughness of the restoration decreases, its cosmetic appearance improves, coloring resistance and abrasion resistance increase, plaque deposition decreases, and the health of the periodontal tissues is maintained. In addition, as the surface smoothness of the restoration increases, microleakage between the tooth and restoration and the risk of secondary caries both decrease. Previous studies have demonstrated that oral hygiene increases in line with the surface smoothness of the restoration (Erdemir, Sancakli, & Yildiz, 2012; Ergucu & Turkun, 2007). Different polishing methods can be applied to reduce the surface roughness of the restoration. The most

TABLE 2 Significance of profilometer values among comparison of restorative materials and staining solutions

Source	Type III sum of squares	*df*	Mean square	*F*	Sig.
Composite	1.060	7	0.151	4.907	.000*
Solution	0.068	3	0.023	0.732	.534
Composite × solution	0.921	21	0.044	1.422	.120
Error	3.948	128	0.031		
Total	13.423	160			
Corrected Total	5.997	159			

*Statistically significant, $p < .05$.

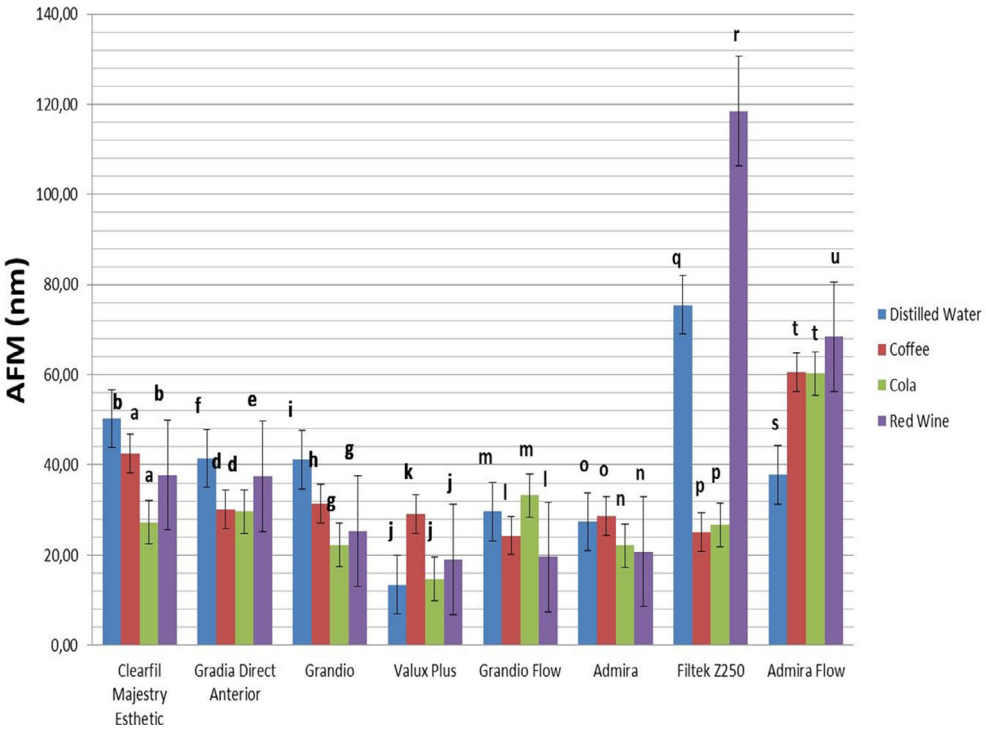

FIGURE 3 Results of surface roughness analysis by composite type by using AFM. Different letters indicate a statistically significant difference between the groups. AFM, anatomic force microscope [Color figure can be viewed at wileyonlinelibrary.com]

successful results in the polishing of composite resin restorations have been reported with flexible aluminum oxide discs (Berastegui, Canalda, Brau, & Miquel, 1992; Lu, Roeder, & Powers, 2003). In this study, the surfaces of all specimens were also polished with aluminum oxide discs. Similarly, smooth surfaces have also been obtained using a celluloid strip matrix during the formation of composite resin restorations (Jung, 2002; Pratten & Johnson, 1988). In this study, the celluloid strip matrix was placed onto the upper and lower surfaces during polymerization of all composite resin specimens.

In this study, the method used by Okte, Villalta, Garcia-Godoy, Lu, and Powers (2006) in their study, was used to mimic the coloring effect of oral beverages on composite materials. According to this method, samples were stored in staining solution for 3 hr per day, and the remaining time was stored in distilled water. As stated in the literature, since composite resins were not exposed to colorant beverages continuously in the oral environment, the materials were kept in distilled water for 3 hr and refreshed every day. Similarly, Celik, Yuzugullu, Erkut, and Yazici (2009) were subjected their specimens to the coloring cycle for 3 hr and 30 days a day in their study. The researchers reported that this time corresponds to 5-year aging with respect to immersion time.

The most commonly used parameter for measuring the surface roughness of composite resins is Ra. Profilometer devices have long been used to determine this value on the material surface in vitro. These devices are capable of calculating the mean surface roughness values of various materials by obtaining 2D images from the sample surface (Ergucu & Turkun, 2007). However, since these devices provide 2D images and do not adequately reflect the surface properties of the material, AFM devices have been employed in the evaluation of surface roughness of dental materials in recent years. AFM devices yield high-resolution 3D nanometric images from the material surface. These devices have the advantages of higher resolution and 3D image acquisition compared to the profilometer, and also provide better evaluation of surface properties than electron microscopes (Varanda et al., 2013). Furthermore, these devices do not require specimen coating and fixation. Due to these advantages, AFMs are used in dentistry to measure fractures and cracks in the material surface, and the roughness thereof (Botta, Duarte Jr., Paulin Filho, & Gheno, 2008).

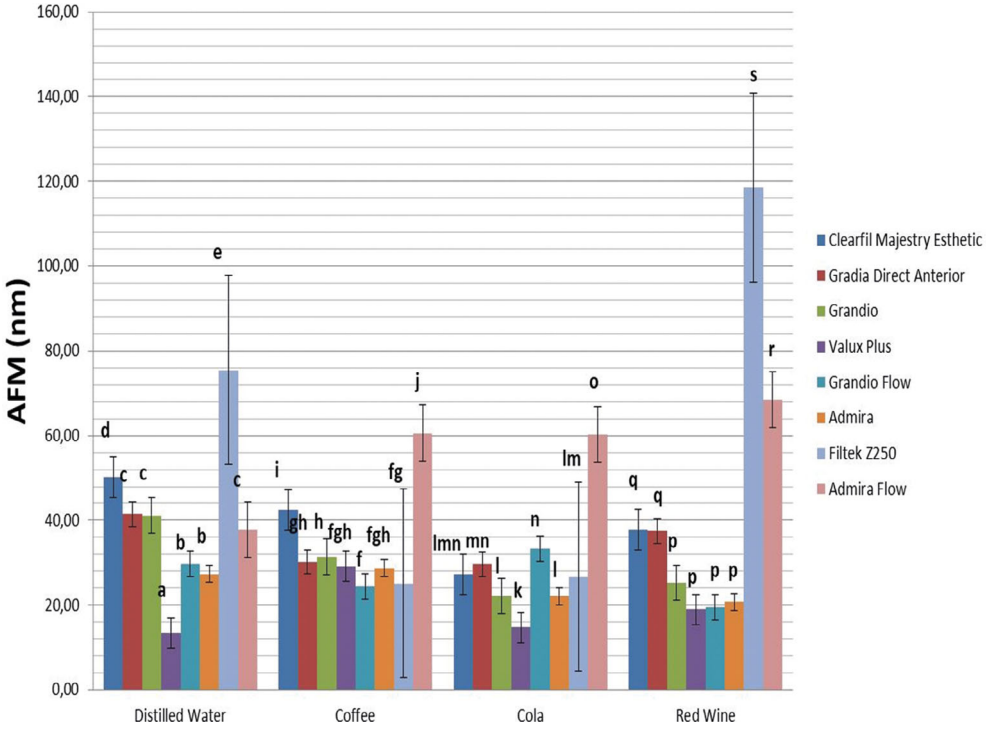

FIGURE 4 Results of surface roughness analysis by storage solutions using AFM. Different letters indicate a statistically significant difference between the groups. AFM, anatomic force microscope [Color figure can be viewed at wileyonlinelibrary.com]

Source	Type III sum of squares	*df*	Mean square	*F*	Sig.
Composite	32,508.852	7	4,644.122	525.828	0.000*
Solution	4,456.313	3	1,485.438	168.188	0.000*
Composite * solution	32,505.165	21	1,547.865	175.256	0.000*
Error	1,130.498	128	8.832		
Total	285,112.735	160			
Corrected Total	70,600.828	159			

TABLE 3 Significance of AFM values among comparison of restorative materials and staining solutions

*Statistically significant, $p < .05$.
Abbreviation: AFM, anatomic force microscope.

In this study, surface roughness values of composite resin specimens were measured using both a profilometer and an AFM, and the results yielded by the two different devices are presented and compared. Differences between the measurement sensitivity and operating mechanisms of the two instruments may result in different surface roughness values being obtained from the same specimens. Comparisons of composites stored in the same solutions in this study revealed no statistically significant difference between the surface roughness values of the composite resin specimens obtained with the profilometer ($p > .05$),but significant differences were detected using the AFM ($p < .05$). Similarly, after the composite specimens had been stored in different solutions, no statistically significant differences were found between the surface roughness values of the specimens obtained with the profilometer ($p > .05$),but differences were detected by the AFM ($p < .05$). The null hypothesis in this study was partially rejected due to differences in surface roughness values between the groups at evaluation with the AFM. Differences in AFM measurements may be attributed to the fact that the AFM device provides a higher resolution image and performs nanometric measurement, and a more sensitive measurement may result in more distinctive results.

The surface roughness of composite resins depends on the inorganic filler content and the organic matrix structure. In general, composite resins with a smaller filler content exhibit smoother surface properties (Ereifej et al., 2013). In this study, the mean surface roughness values of the nanohybrid Grandio specimens were lower than those of the microhybrid composite resin specimens. Some studies have indicated that the surface properties of the composite resin develop when the filler particle size decreases (Nagem Filho, D'Azevedo, Nagem, & Marsola, 2003; Yap, Low, & Ong, 2000). Nanoscale particles produce a smoother surface than conventional microhybrid composites. Ereifej et al. (2013) found that nanofiller composites provided lower surface roughness and better polishability. Similarly, Ergucu and Turkun (2007) reported smaller defects on the surface of composite resins after polymerization and polishing, and that smoother surfaces thus resulted. In addition to filler size, the filler type and amount are other factors that affect the surface roughness of composite resins. In this study, based on AFM evaluation, the

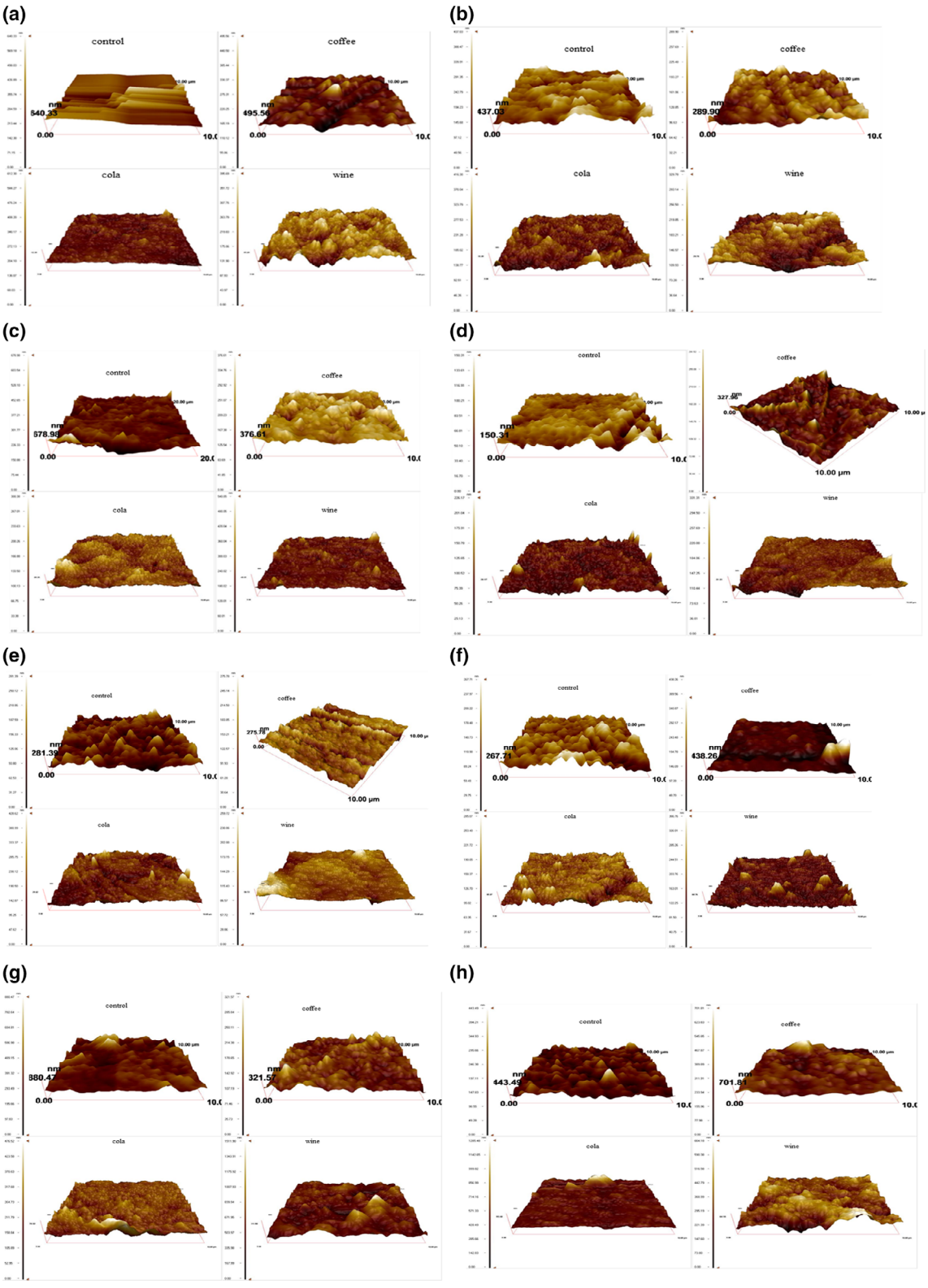

FIGURE 5 (a) AFM surface image specimens of Clearfil Majesty Esthetic composite resin stored in different solutions. (b) AFM surface image specimens of Gradia Direct Anterior composite resin stored in different solutions. (c) AFM surface image specimens of Grandio composite resin stored in different solutions. (d) AFM surface image specimens of Valux Plus composite resin stored in different solutions. (e) AFM surface image specimens of Grandio Flow composite resin stored in different solutions. (f) AFM surface image specimens of Admira composite resin stored in different solutions. (g) AFM surface image specimens of Filtek Z250 composite resin stored in different solutions. (h) AFM surface image specimens of Admira Flow composite resin stored in different solutions. AFM, anatomic force microscope [Color figure can be viewed at wileyonlinelibrary.com]

surface roughness values of Valux Plus specimens stored in distilled water and cola were significantly lower than those of other composites ($p < .05$). Valux Plus composite resin contains a high amount of inorganic filler (71% vol.). Very small filler particles (0.01 μm) and silanized ceramic are also present in this composite resin. These structural properties can be effective in reducing the surface roughness of Valux Plus specimens. The filler type also affects the surface properties of the composite resin. Ceramic fillers exhibit high abrasion resistance and bestow better polishability on the composite resin (Scheibe, Almeida, Medeiros, Costa, & Alves, 2009). In this study, Admira composite resin specimens exhibited similar surface roughness values to those of nanohybrid composites, which may be due to its ormocer-based composite resin structure. In addition to inorganic fillers, the organic matrix structure is another important factor affecting the surface properties of the composite resin. The high surface roughness values of Filtek Z250 specimens in some solutions despite the high inorganic filler content (78% vol.) may be due to the organic matrix structure of this composite resin.

Exposure of composite resin restorations to different solutions in the oral environment may affect surface properties. In this study, composite resin specimens were stored in commonly consumed alcoholic (red wine) and nonalcoholic (coffee and cola) solutions, and surface roughness values were compared with those of the control group. A number of previous studies have shown that alcoholic beverages increase the surface roughness of composite resins (Bansal et al., 2012; MA et al., 2016). In this study, the surface roughness values of Filtek Z250 and Admira Flow specimens stored in red wine were higher than in the control group ($p < .05$). Alcohol derivatives such as ethanol in red wine can increase the surface roughness of the composite resin by penetrating the resin matrix structure. Bansal et al. (2012) investigated the effect of different solutions on the surface roughness of composite resins and observed the highest surface roughness values in specimens stored in cola. The researchers attributed this to the low pH of cola, leading to surface wear and roughness. The pH values of the coffee and cola used in this study are lower than 7. However, the mean surface roughness values of some composites stored in coffee and cola were lower than those of the control group. Depending on the composite resin matrix structure, low pH solutions can remove burrs and residual monomers from the resin surface and lead to a smoother surface (Reddy et al., 2013; Tavangar, Bagheri, Kwon, Mese, & Manton, 2018). In addition, since measurements were taken only from certain regions on the surface and not from the entire specimen surface, the results may not exactly reflect the roughness values of the entire surface. In this study, Admira Flow and Valux Plus specimens stored in cola and coffee exhibited higher average surface roughness than the control group. The effect of different solutions on the composite resin surface may vary depending on the matrix structure of the composite resin.

5 | CONCLUSION

Within the limitations of this in vitro study, it may be concluded that

- Since both measurement methods show similar and different results, they support each other partially. Generally, AFM performs more detailed surface analysis than profilometer. However, since both methods measure the limited areas on the sample surfaces, the measurements on which the entire surface is evaluated will give more accurate results.
- Inorganic filler content and the organic matrix structure of composite resins affect the surface roughness.
- Alcoholic and nonalcoholic beverages may affect the surface roughness of composite resins, depending on the pH of the beverage, its alcohol content, and the organic and inorganic structure of the resin. Further studies are now needed to evaluate our findings. We also think that more accurate data can be obtained by developing techniques to measure the roughness of the entire material surface.

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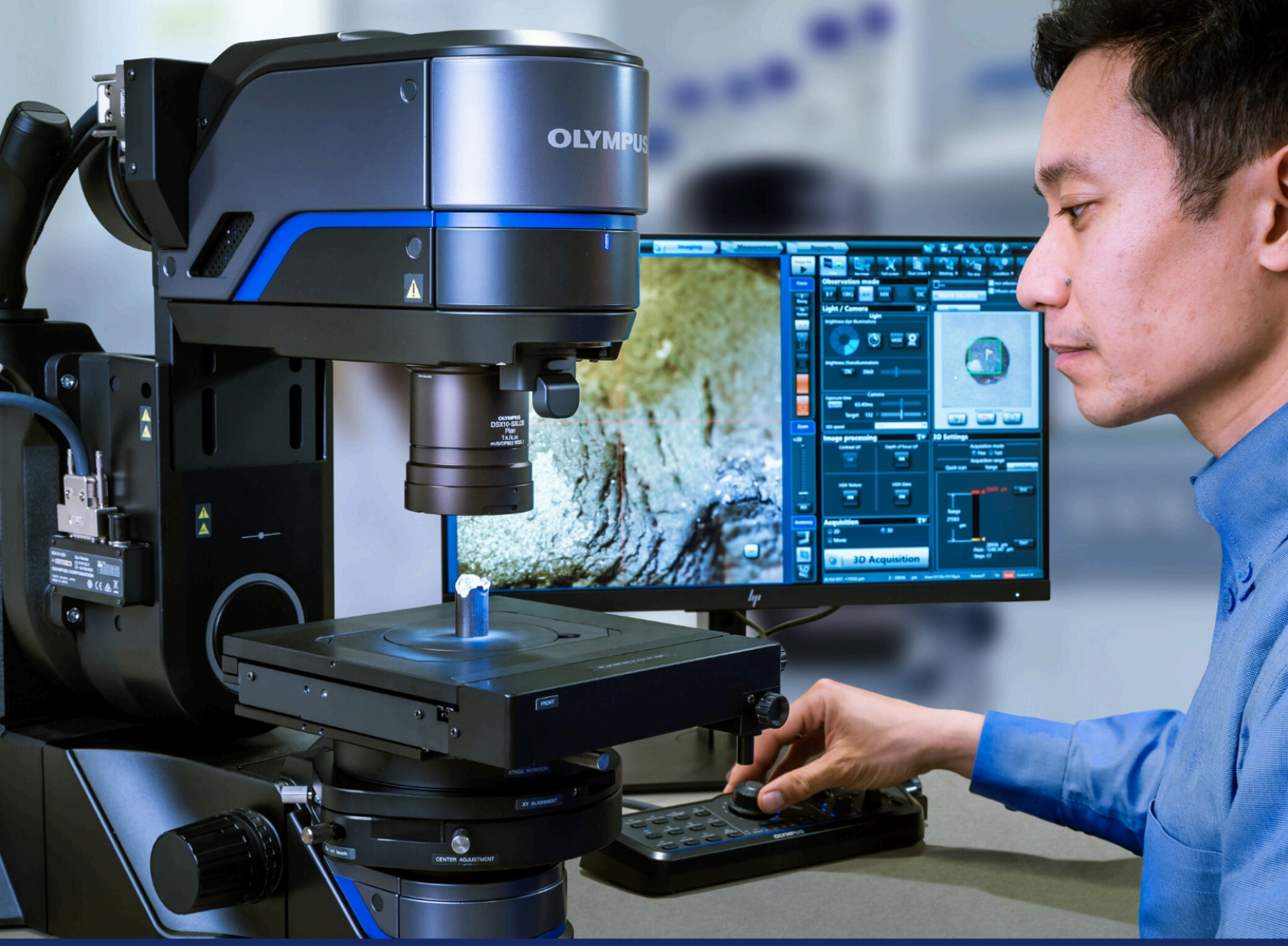

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