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Investigation of surface roughness values of various restorative materials after brushing with blue covarine containing whitening toothpaste by two different methods: AFM and profilometer

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Abstract

The aim of this study is to evaluate the effect of whitening toothpaste on the surface roughness of resin-based restorative materials by different measurement methods. Twenty four specimens from each of human enamel, a microhybrid composite and two nanohybrid composites discs (8.0 diameter $\times$ 4.0 mm thick) were divided into two groups ($n = 12$) according to toothbrushing solution and subjected to simulation toothbrushing (30,000 cycles) with both distilled water and whitening toothpaste containing blue covarine. Surface roughness was examined using atomic force microscopy (AFM), profilometer, and scanning electron microscopy (SEM), and the data obtained were subjected to analysis. Ra values of Tescera (TES) were significantly higher than Sonicfill 2 (SF2) when brushing both toothbrushing solutions for initial or 30,000 cycles. Roughness increased for SF2 and TES when brushed for 30,000 cycles and was higher than enamel and Herculite XRV Ultra (HXU). Human enamel was obtained lower surface roughness values brushed with toothpaste compared with distilled water. Evaluation of the surface roughness of control groups using the AFM revealed no statistically significant difference between the groups, but significant differences were found using a profilometer. The use of abrasive whitening toothpaste containing blue covarine and the number of brushing cycles affect the surface properties of human enamel and the restorative material, and also, the clinical success of the restoration. Toothbrushing for 30,000 cycles increased the surface roughness of all materials. The type of toothbrushing solution partially has affected surface roughness.

KEYWORDS
AFM, bulkfill, profilometer, surface roughness, whitening toothpaste

1 | INTRODUCTION

Consumers and patients in different populations who perceive white teeth as a symbol of beauty and health are not satisfied with the current tooth color and cite tooth color as the main reason for their dissatisfaction with their appearance (Al-Zarea, 2013; Vinita Mary et al., 2020; Xiao et al., 2007).

In recent years, the demand for "whiter teeth" by patients who are dissatisfied with their tooth color and have normal tooth color seems to be continuously growing (Santos, De Carvalho, Cangussu, Barros, & Trindade, 2020; Widodo, Soetjipta, & Palupi, 2020). The increasing interest in the aesthetic benefits that can be obtained from dental treatment has contributed to the development of a wide variety of teeth whitening treatment products and technologies that will meet the expectations and demands. (Kielbassa, Maier, Gieren, & Eliav, 2015; Meireles, de Sousa, Lins, & Sampaio, 2021).

In tooth whitening treatment, which is one of the concepts to improving dental aesthetics and tooth discoloration, include either gels containing hydrogen peroxide or carbamide peroxide, or whitening toothpastes containing specific abrasives or chemicals that can be added to the toothpaste to optimize the removal and control of external tooth stain (Bielfeldt, Foltran, Bohling, Manger, & Wilhelm, 2018; Tao et al., 2017).

The abrasive property of the particles in the toothpaste content should not be high enough to damage the hard or soft tissues and restorations in the oral cavity (Barbieri, Mota, Rodrigues-Junior, & Burnett Jr., 2011; da Rosa et al., 2016). Whitening toothpaste with formulations containing abrasive such as hydrated silica, calcium carbonate, dicalcium phosphate dihydrate, calcium pyrophosphate, sodium metaphosphate, alumina, perlite, nanohydroxyapatite, or sodium bicarbonate mechanically remove pigmented biofilms and chromophores on the surface of the dental enamel (Lippert, 2013; Shamel, Al-Ankily, & Bakr, 2019). Furthermore, the daily utilization of these abrasives modifies the surface of enamel and resin restorations in the mouth, decreasing the adhesion of dental plaque, food debris and chromophores, reducing tooth pigmentation, and changing its discoloration (Joiner, 2010; Vaz et al., 2019). As a new development in teeth whitening, apart from the abrasive cleaning system, using optical principles silica-based toothpaste containing blue covarine pigment has developed (Meireles et al., 2021).

The aesthetic quality of restoration depends on surface texture; if it is the low surface hardness of the material is susceptible to scratches and rough leads to decreased gloss, staining of material, discoloration, also bacterial adhesion and biofilm formation which may lead to secondary caries (Da Costa, Adams-Belusko, Riley, & Ferracane, 2010). Not only are direct resin-based materials used in restorative purposes in posterior restorations, but also indirect composite resins are frequently used. Thus, it is very important to know the effect of abrasive containing whitening toothpaste on the physical properties of restorative materials.

The profilometer uses tracing to determine the profile along three lines and its fluctuations on the surface relative to the baseline, but cannot provide a good visual description of the surface with two-dimensional (2D) measurements. AFM presents some important advantages such as minimal specimen preparation, high-quality images, and visualization of a three-dimensional (3D) image of the surface, convenient for high-resolution morphological description. Furthermore, the information obtained is more comprehensive and descriptive than the 2D measurements provided from the stylus profilometry (Tholt, Miranda-Júnior, Prioli, Thompson, & Oda, 2006).

SEM is widely used to observe the surface structure of materials, particle size, and surface scratches and defects that occur on a surface. The most reliable in evaluating surface roughness atomic force microscopy (AFM), measurements can be made on 2D images, giving numerical values, and allowing a 3D imaging at a nanometric resolution. While 3D images cannot be obtained with SEM, AFM can provide the surface topography in more detail (Kumari, Bhat, & Bansal, 2016).

This study aimed to assess surface characteristics of both indirect resin composite polymerized with additional polymerization method and direct resin composite so-called as bulkfill and compared it to that of human enamel and conventional composite resin, using the following instruments: mechanical profilometer, atomic force, and scanning electron microscopes.

The research hypotheses tested were: (a) the surface roughness of resin composites obtained after toothbrushing procedures are similar to that of human enamel, (b) conventional nanohybrid, nanohybrid bulkfill, and reinforced microfill resin composites present similar surface quality, (c) the methods applied for surface analysis are equally effective to determine the surface quality in resin composites and human enamel, and (d) there is no difference between toothbrushing procedures.

2 | MATERIALS AND METHODS

The study was validated by the non-interventional Clinical Research Ethics Committee under the protocol number 2018/5 (Faculty of Medicine, Afyon Kocatepe University). The study used human enamel and three different types of resin composites: the negative control group; human enamel, the positive control group; a nanohybrid direct conventional resin composite Herculite XRV Ultra, (HXU; Kerr, Orange, California), experimental groups; a nanohybrid direct bulk-fill resin composite SonicFill 2 (SF2; Kerr, Orange, California), and a microhybrid indirect resin composite Tescera (TES, Bisco Dental Products, Seoul, Korea) were selected and then randomly divided into two groups ($n = 12$).

2.1 | Preparation of specimens for negative control group

A total of 24 extracted, intact, human permanent maxillary central incisors with no structural deformities, previous restorations, and caries were collected and then screened for surface cracks and fracture under magnification with a light microscope. Calculus deposits and soft tissues were removed with a hand scaler and the roots were then cut 1 mm below the cementoenamel junction using a hard tissue cutter. The buccal surface of the crown was cleaned with pumice and then flattened using a model trimmer to obtain an even surface. The coronal tooth structure of human enamel specimens attached to by attentively melting wax around their peripheries was embedded in self-cured acrylic resin with the flat surface facing the mold base.

2.2 | Preparation of specimens for positive control and experimental groups

With 24 for each resin composite material (Shade A2), 72 composite discs (8 mm in diameter and 4 mm in thickness) in total were obtained by placing the material in a disc-shaped metal mold. After placing a mylar strip on either side of the mold covered with a 1-mm thick glass slide and pressing tenderly to remove excess material and produce a smooth flat surface. The cured specimen was removed from the mold and excess material was removed by hand.

Both sides of all composite disc specimens were polymerized for 10 s using LED light-curing units (Elipar Deep Cure-S, 3M ESPE, St. Paul, Minnesota) during the preliminary curing phase. Indirect resin composite specimens were supplementary post-cured with their own special curing units according to their manufacturer's instructions. After removing the specimens from the mold, their lateral surfaces were marked to differentiate the top and bottom surfaces. Resin composite specimens were embedded in self-cured acrylic resin with the top surface facing the mold base.

SonicFill 2, was a high viscosity and pasted consistency nanohybrid bulk-fill that uses sonic energy produced by a specially designated handpiece to reduce the viscosity of the material during placement to improve its flow properties. The SonicFill handpiece (Kerr) was set to 3 when dispensing SonicFill 2.

The TES specimens to complete the polymerization process under pressure, light, and heat were placed in a light cycle unit for 2 min (0.5 MPa pressure-light; Tescera ATL Light Cup, Bisco Inc., Schaumburg, Illinois), followed by a 16-min heat cycle in water with oxygen scavenger capsules (135°C—Heat Cup, Bisco Inc., Schaumburg, Illinois) at post-cure.

The exposed surface of all specimens was polished using sandpaper with a straight handpiece set at the same speed in increasing grit of 400, 600, and 800 μm, respectively, to obtain a smooth surface for 20 s and then polished by two dental polishing protocols: multistep dental polishing systems with SuperSnap sandpapers and one-step finishing/polishing systems One-Gloss (Shofu, Inc. Kyoto, Japan).

All specimens were finished and polished unidirectionally using Super Snap Rainbow Technique Kit (Shofu, Kyoto, Japan) Al_2O_3, each polishing discs was used only one time for a single specimen, under water-cooling, for 20 s (1 min for each specimens) at 10,000 rpm. The specimens were finished and polished using purple, green, and pink discs. After each step, the specimens were washed with distilled water and dried with air for 5 s. After polishing each specimen, the discs were replaced with new ones to obtain homogeneous surfaces on specimens of equal dimensions measured using digital calipers. The ultrasonic cleaning to remove residue from polishing between each sandpaper and after polishing was carried out for 10 min and dried. All specimens were stored in distilled water at 37°C for 24 hr before analysis.

2.3 | Simulated toothbrushing

Brushing was performed using a soft toothbrush (Sensitive Expert, Signal, Unilever, Germany) attached to the movable arm of the toothbrushing machine applying a reciprocating linear motion under a constant pressure load of 200 gF After the brushes were placed in their starting positions, the experiment parameters were entered. In forward–backward movement, the vertical movement distance was set to 10 mm and the movement speed was set to 40 mm/s. All brush holders were adjusted to contact the middle of the specimens with adjustments in the x-axis and y-axis. Surface roughness measurements were repeated under appropriate conditions at the end of 30,000 brushing cycles (Esetron MF-100, MOD Dental, Ankara, Turkey) for each specimen in both distilled water groups and whitening toothpaste groups. While surface roughness measurements were made at all cycle times with the mechanical profilometer, AFM was made only in the initial phase and at the end of 30,000 cycles.

The entire surface of specimens in the chamber was coated at least 3 mm with the homogeneous whitening toothpaste slurry, obtained at room temperature by adding 25 g of toothpaste (White Now CC, Signal) and 40 ml of distilled water (5:8) into a magnetic stirrer. Toothpaste solution (White Now CC, Signal) and toothbrushes (Sensitive Expert, Signal) were renewed every 5,000 cycles. The brush head was placed parallel to the enamel surface and resin composite specimens, while the bristles were placed vertically. After each brushing cycle, marking in the brushing direction was made with waterproof pens on each edge to the ensure correct positioning of the specimens in the brushing simulator. At the end of each brushing cycle, the specimens were washed under running water and the paste remaining on the brushed surfaces was cleaned for 10 min using an ultrasonic device. The long axis of the specimens in the custom-made chambers is perpendicular to the long axis of the toothbrushes placed in the holder of the brushing machine.

2.4 | Surface roughness

The surface roughness measurements of the specimens were carried out at the end of 30,000 cycles at an angle of 90° to the line determined in the initial measurements and the bristle traces formed after brushing. After five different measurements were made from the brushed surface of the specimen, the average surface roughness value (Ra, μm) of the specimen was calculated by taking the arithmetic mean of these measurements. The data scanned by mechanical profilometer (Surtronic S-100) was recorded as data in TalyProfile Lite 7.1 program (Leicester, England). All roughness measurements were made by the same researcher. After finishing-polishing and simulated toothbrushing, the average surface roughness of the specimens was measured using a contact type profilometer (Surtronic S-128, Taylor Hobson, Leicester, England, UK) (Figure 1).

The surface roughness tester was periodically calibrated prior to the measurement of each group and the average roughness values obtained as micrometers were converted into nanometer values.

After the profilometer device was calibrated with the help of a reference calibration block with a Ra value of 6.0 μm, the "cutting length" value of the diamond tip with 10 μm radius was 0.80 mm and the stylus speed was set as 0.1 mm/s. Then, in five measurements made in different locations and the same direction of the 8 mm diameter disc-shaped specimens, the surface roughness values were measured by scanning a trace length of 4 mm for resin composites and 3 mm for human enamel specimens at 100 μm intervals with a diamond tip.

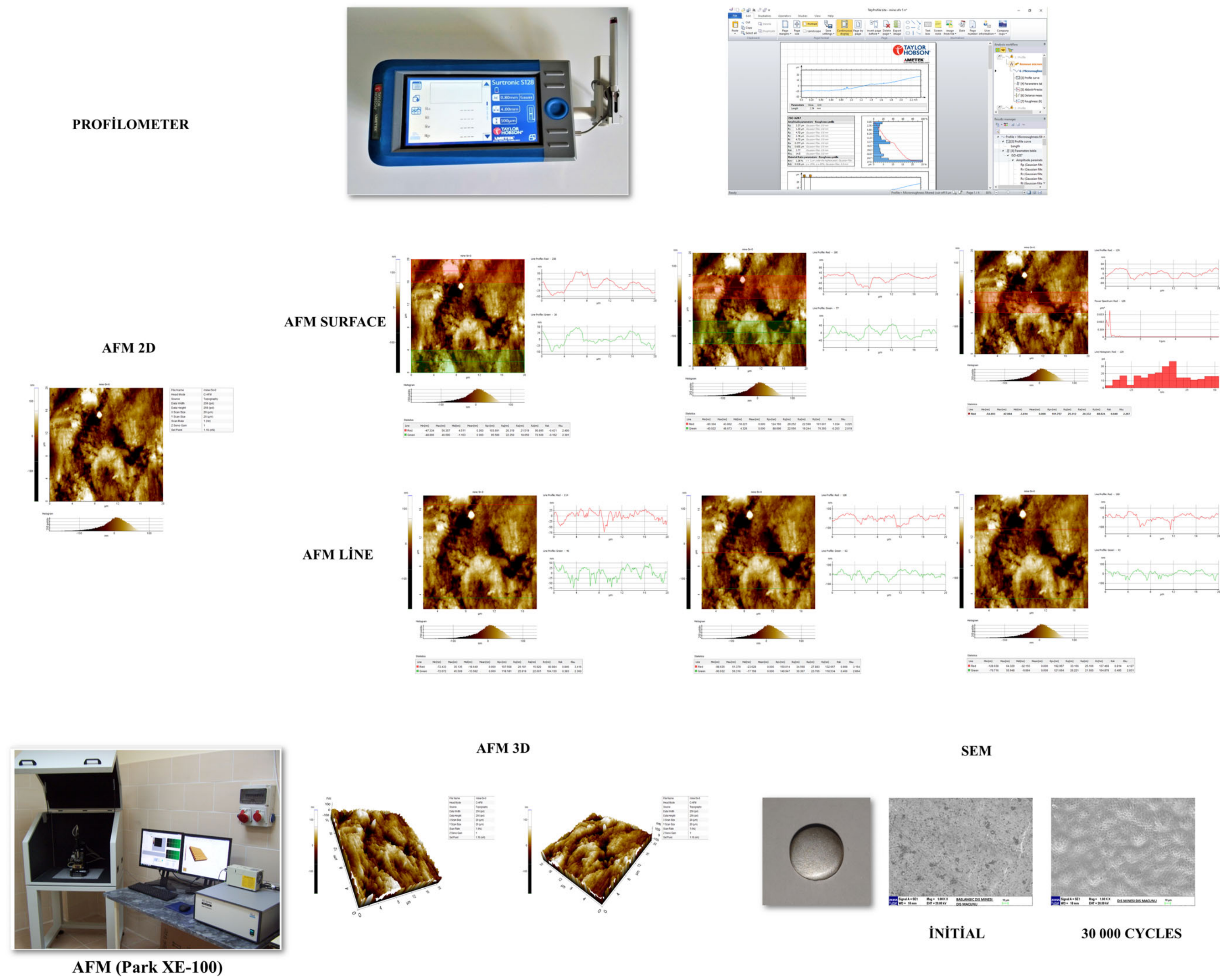

FIGURE 1 Surface roughness measurements with AFM (AFM surface and AFM line) on 2D images and Taylor Hobson Profilometer. SEM images (×500 magnification) and Atomic force micrography showing 20 × 20 μm^2 surface area 2D and 3D topography with a resolution of 256 × 256 pixels of the human enamel surface

The surface of the specimen were scanned in contact mode, 1 Hz scan rate, on 20 × 20 μm^2 scanning area by AFM (Park XE-100, Park Systems Inc., South Korea), and images with a resolution of 256 × 256 pixels were recorded in three dimensions. Nanosensors PPP-CONTSCR 10M (Park Systems, South Korea) with nominal force constant 0.2 N/m and resonance frequency 23 kHz were used to image specimens. The cantilever was 225-μm long, 1-μm thick, and 48-μm mean width and the backside Al reflective coating for contact mode. Data were obtained from five different regions for AFM Line on 2D images obtained by scanning an area of 20 × 20 μm^2. For the measurements made with AFM surface, after the image was divided into five equal areas, the average surface roughness values (Ra, nm) were calculated by taking the arithmetic averages of the five data obtained by scanning the 80 μm^2 area (Figures 1 and 3).

AFM was calibrated by taking measurements in 3D of the reference sample with software calibration. This is a reasonably simple and inexpensive procedure that involves imaging a standard sample (usually a grid structure with a known pitch), in order to create a calibration file that will be used to control the scanner's movements when control and experimental groups' samples are imaged. However, calibration using software becomes accurate only when the center of the scan range used to measure an unknown sample exactly coincides with the center of the scan range used to view the reference sample and generate the calibration file.

The XEP Data Analysis Program, version 1.8.0 Build 48 (Park Systems Corp., South Korea) analysis software was used to obtain surface topography from AFM images and for roughness calculations.

2.5 | Surface morphology

The surface of two randomly selected specimens mounted on an aluminum plate, sputter-coated with carbon, from each group, was examined using a SEM (LEO 1430 VP, Cambridge, UK) with an operating voltage of 20 kV in secondary electron mode to investigate the surface morphology and qualitatively compare surface characteristics before and after simulated toothbrushing at a magnification of ×500, ×1,000, ×2,000, and ×5,000.

After the surface roughness measurement, specimens were prepared for SEM to investigate a visual correlation with numerical surface roughness (Ra) values (Figure 1).

2.6 | Analysis

The surface roughness data were examined by one-way ANOVA. AFM line and AFM surface, dual comparisons at the level of brushing solution in distilled water and toothpaste solution groups were made using the independent sample *T*-test. Post hoc LSD test was applied for multigroup comparisons at the level of material groups, measurement methods, number of cycles, and differences.

3 | RESULTS

Results of surface roughness measurement of specimens of all groups are tabulated in Tables 1 and 2. Roughness differences within the same material and between materials were calculated in the same toothbrushing solution at initially and after 30,000 cycles simulation toothbrushing using different measurement methods.

There was an increase in surface Ra after the simulated toothbrushing in all groups. The type of toothbrushing solution used partially affected the difference in surface roughness.

İn measurements with Taylor Hobson profilometer obtained significantly higher surface roughness values before and after simulated toothbrushing in both solutions compared with AFM line and AFM surface (Graphic 1). Besides, surface roughness decreased following brushing with the toothpaste compared with brushing with distilled water at the end of 30,000 cycles. The human enamel was the least affected by toothbrushing at AFM measurements.

The resin composites specimens brushed with toothpaste were showed higher roughness values compared with distilled water groups. However, surface roughness values were found to be lower in human enamel specimens brushed with toothpaste.

Post-hoc LSD multiple comparison tests showed that toothbrushing had the least effect on the control groups, human enamel and HXU, where there was no statistical difference in the roughness value in Taylor Hobson profilometer (except enamel brushing with toothpaste), AFM line, and AFM surface measurements.

TES exhibited the highest surface roughness in all other methods, except for the Taylor Hobson method, which obtained the greater surface roughness in the SF2 group.

The lowest surface roughness value for both toothbrushing solutions was obtained for the AFM surface measurements performed HXU group at initial. Before the toothbrushing there was no significant difference between the resin SF2 and the other materials (Human Enamel and HXU), but, after the toothbrushing, it was observed a significant statistical difference between the groups Human Enamel and HXU with the groups SF2 and TES, which presented greater values of final Ra (Table 2).

TABLE 1 Mean and *SD* of roughness (Ra, μm) of human enamel before and after simulated toothbrushing

		Initial		30,000 cycles	
Material	Measurement method	Distilled water	Toothpaste	Distilled water	Toothpaste
Human enamel (negative control group)	Taylor Habson	$122.3 \pm 11.5^{Aa/f}$	$116.2 \pm 14.7^{Aa/h}$	$243.7 \pm 71.4^{Aa/e}$	$212.6 \pm 54.1^{Aa/g}$
	AFM line	$20.1 \pm 4.0^{Bb/f}$	$16.3 \pm 4.4^{Bb/g}$	$25.0 \pm 2.4^{Bb/e}$	$22.6 \pm 5.8^{Bc/g}$
	AFM surface	$13.1 \pm 2.7^{Cc/e}$	$12.8 \pm 4.6^{Bc/g}$	$17.8 \pm 3.8^{Bd/e}$	$16.9 \pm 5.2^{Bd/g}$

Note: The upper case superscripts refer to the columns (measure method at toothbrushing solution). First lower case superscripts refer to the rows (toothbrushing solutions within measure method), and the second lower case superscripts refer to the columns (initial and 30,000 cycles within solutıon). *$p < .05$ was accepted as significance level.

TABLE 2 Mean and *SD* of AFM surface (Ra, μm) of different composite resins before and after simulated toothbrushing

		Distilled water			Toothpaste		
Material		Initial	30,000 cycles	30,000–initial	Initial	30,000 cycles	30,000–initial
AFM surface	Human enamel	$13.1 \pm 2.7^{Bb/c}$	$17.8 \pm 3.8^{Ca/d}$	4.7 ± 2.6^{B}	$12.8 \pm 4.6^{Ba/c}$	$16.9 \pm 5.2^{Ca/d}$	4.1 ± 2.3^{B}
	Herculite XRV ultra	$10.1 \pm 2.8^{Bb/c}$	$16.1 \pm 1.8^{Ca/e}$	6.0 ± 2.3^{B}	$9.7 \pm 2.7^{Bb/c}$	$22.1 \pm 5.5^{BCa/d}$	12.4 ± 5.1^{A}
	Sonicfill2	$19.5 \pm 8.0^{Bb/c}$	$33.2 \pm 10.8^{Ba/d}$	13.7 ± 11.0^{A}	$20.9 \pm 6.2^{Bb/c}$	$33.4 \pm 9.2^{Ba/d}$	12.5 ± 8.9^{A}
	TESCERA	$61.0 \pm 23.1^{Aa/c}$	$76.4 \pm 26.4^{Aa/d}$	15.4 ± 6.4^{A}	$66.9 \pm 32.1^{Aa/c}$	$81.6 \pm 37.6^{Aa/d}$	14.7 ± 6.2^{A}

Note: The upper case superscripts refer to the columns (human enamel and composite at initial, 30,000 cycles, and 30,000–initial). First lower case superscripts refer to the rows (cycles within human enamel and composite), and the second lower case superscripts refer to the columns (solutıon within initial and 30,000 cycles). *$p < .05$ was accepted as significance level.

3.1 | Roughness

3.1.1 | Atomic force microscope results

Qualitative analysis of the 2D (AFM line and AFM surface) and 3D AFM images showed that the toothbrushing protocol produced lines and scratches on the tested materials' surfaces, which were results of the brushing process with whitening toothpaste (Figures 2 and 3). The measured AFM roughness parameters (Ra) values were satisfying and showed relatively low surface roughness for all materials brushed by toothpaste. Figure 2 shows 3D microphotography of the control and tested groups before and after brushing for 30,000 cycles with distilled water and hydrated silica blue covarine containing toothpaste. HXU reveals a nearly uniform surface texture with smooth elevations and few numbers of shallow valleys. SF2 shows broad elevations and uniform ground. Figure 2 shows AFM 3D image of groups with toothpaste brushing, and it reveals that surface texture had irregularities, prominent sharpened peaks, and deep valleys.

The topographical analysis (Figure 2), showed that the smoothest surfaces were associated with HXU, while the highest surface irregularities were observed with TES among all groups. For microhybrid composite (TES) specimens, more irregularities were detected on the surface in comparison with nanohybrid (HXU and SF2). In contrast, irregular surfaces are exhibited higher after the brushing procedure with blue covarine containing toothpaste than distilled water. The current study (Figure 3), shows that the AFM surface exhibited lower values in the measuring of surface roughness for tested composites and human enamel compared with the AFM line among measurement methods.

3.1.2 | Scanning electron microscopy

Figure 4 shows that all polished enamel and composite surfaces were roughened following simulated toothbrushing. From the analysis performed by SEM, in amplification of ×5,000, it may be observed that

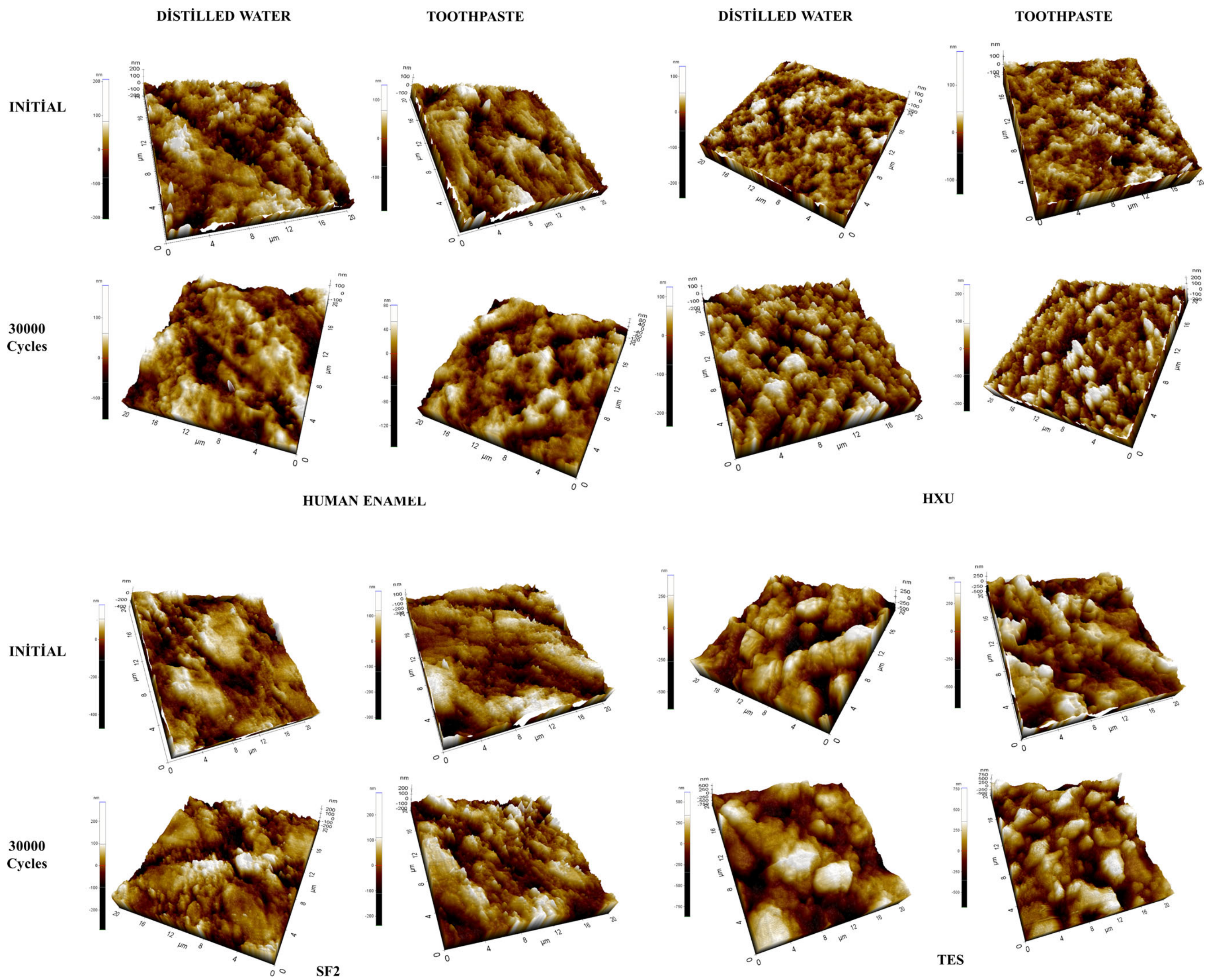

FIGURE 2 The 3D microphotograph of human enamel and resin composites before and after simulated toothbrushing with distilled water and whitening toothpaste

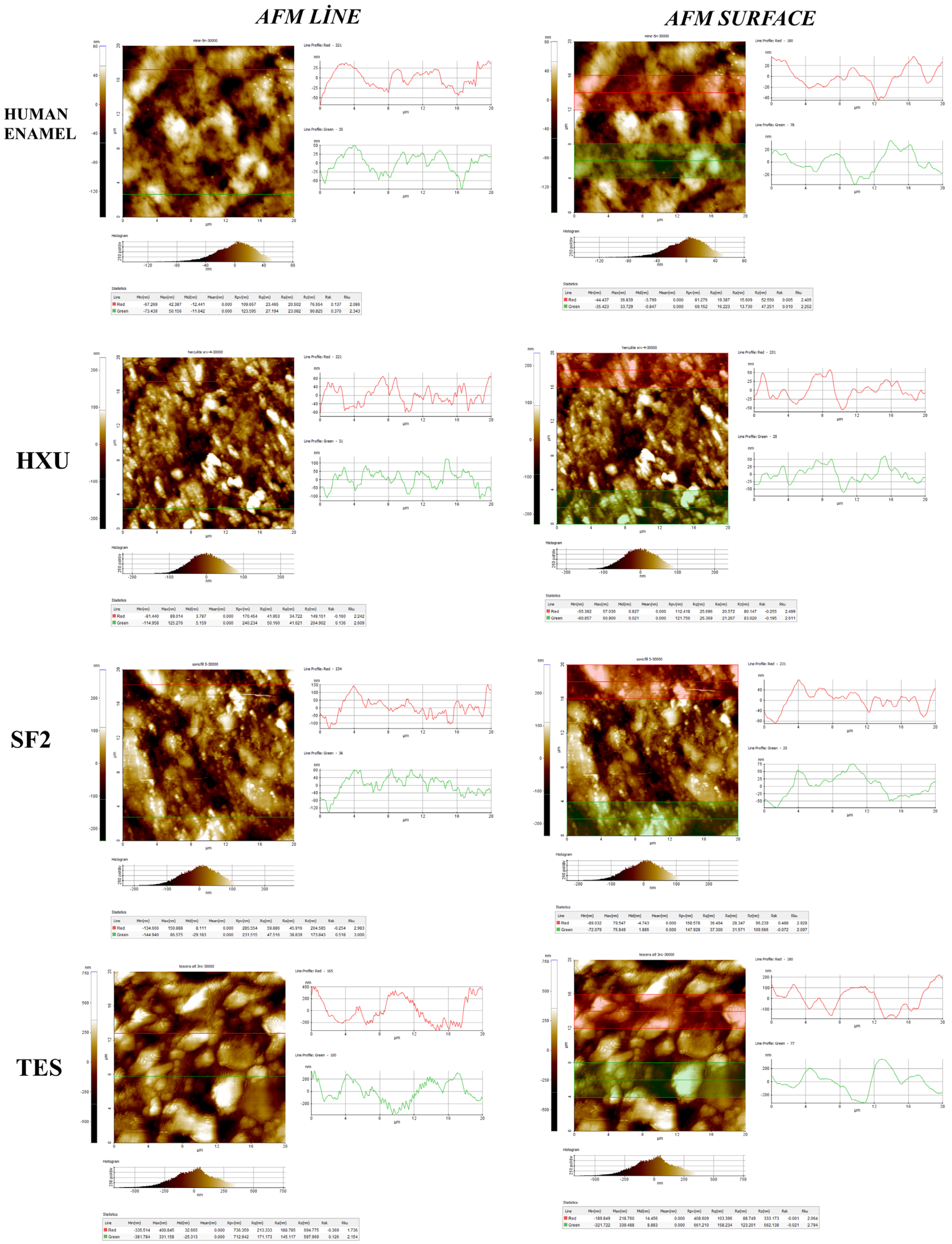

FIGURE 3 AFM line and AFM surface measurements on 2D microphotograph of human enamel and resin composites (HXU, SF2, and TES) after simulated toothbrushing with whitening toothpaste

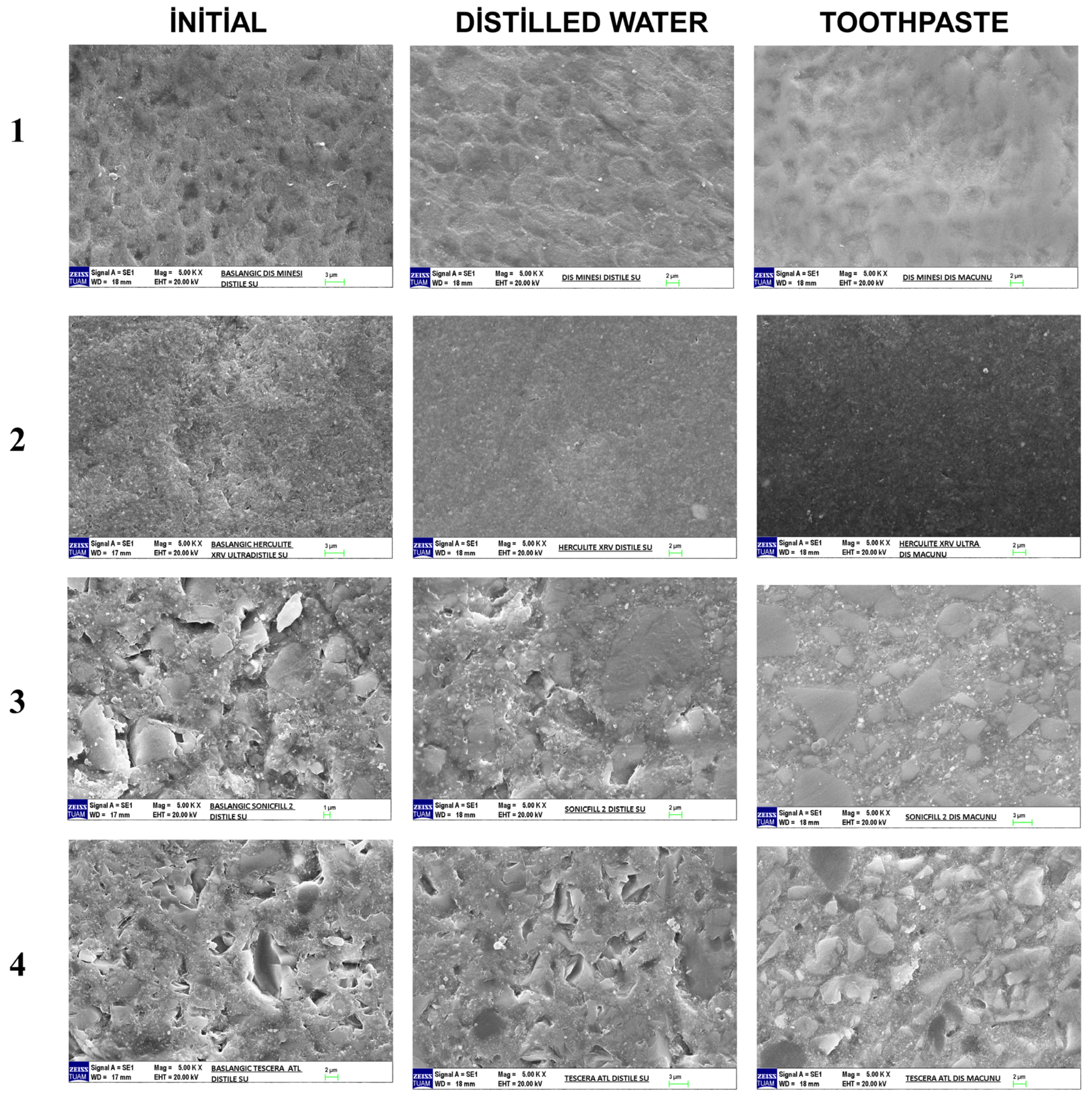

FIGURE 4 Scanning electron micrographs of the surfaces at ×5,000 of human enamel and composite specimens brushed with distilled water and blue covarine containing toothpaste at the end of 30,000 cycles (1: Human Enamel; 2: HXU; 3: SF2; 4: TES)

the surface after the polishing (before simulated toothbrushing—left column) was smoother and uniform. On the other hand, after the simulated toothbrushing (right column), it is observed the presence of protuberant particles of medium and small size at the surface of the resinous matrix, being this fact less evident on the conventional nanohybrid resin, that is, HXU group. The filler particles of HXU tend to be more regular, whereas porous structure and irregularly shaped particles are visible in SF2 and TES.

4 | DISCUSSION

In the oral cavity, the surfaces of restorative materials are exposed to various factors that can alter the quality of the surface. Oral hygiene procedures, among other factors, play an important role. Frequent use of oral and dental care products causes an increase in the surface roughness of the restorative materials (da Cas, Ruat, Bueno, Pachaly, & Pozzobon, 2013). In the study, it was investigated whether oral hygiene materials, which play an active role in our lives, can change the dynamics in the mouth in long-term use, except finishing and polishing processes to minimize the accumulation of plaque in the existing restorations in the mouth.

Scientific studies have reported that alone brushing with a toothbrush could not the capability to promote a significant increase in surface roughness, but that brushing with toothpaste owing to retention of the abrasive agents in the toothpaste ingredient could affect the surface structure (da Cas et al., 2013; Tellefsen, Liljeborg, Johannsen, & Johannsen, 2011). Contrary to previous studies, specimens of brushed with distilled water statistically significant surface roughness increases were observed human enamel in all measurement methods in this study.

The surface roughness of intact enamel is determined by a height difference between enamel prisms and planar faces of hydroxyapatite. The placement of enamel prisms and the organic content of the interprismatic gap may vary depending on individual variations in human tooth enamel (Botta, Duarte Jr., Paulin Filho, Gheno, & Powers, 2009).

Amaral, Miranda, Correa, and Silva (2014) showed that fluoride-containing toothpaste leads to a CaF_2 layer formation that supplies available fluoride and minimizes demineralization on the dental surface. Besides, they have also been suggested that a high concentration of NaF can cause some precipitation of fluorhydroxyapatite within the previously softened surface enamel and, which can result in both an increase of hardness and reduced susceptibility to subsequent dissolution.

Previously studies demonstrated that highly concentrated fluoride agents for topical application can protect enamel against erosion and toothbrushing abrasion (Lagerweij et al., 2006; Wegehaupt & Attin, 2010). İn this study, it is found that human enamel brushed with distilled water creates higher surface roughness than in brushing with toothpaste. We think that the component in the toothpaste provides a smoother surface by placing both in the interprismatic spaces and the heads of the enamel prisms to ensure the balance of remineralization and demineralization. The slightly higher roughness values of the specimens brushed with distilled water can be attributed to this and it was supported by the SEM method. In the images after brushing, the prismatic structure before toothbrushing can still be defined; however, it has been found that the rough surface looks smoother after toothbrushing. Consistent with the results of our study were found in a review by Joiner, Philpotts, Ashcroft, Laucello, and Salvaderi (2008), which concluded that the blue covarine containing whitening toothpaste has no harmful effects on enamel roughness and is an effective resource that delivered demineralization, remineralization and fluoride-uptake performance.

Depending on technological advances in resin composite materials, the maximum acceptable threshold value for surface roughness has been determined as 0.2 μm (200 nm). However, increased bacterial accumulation on material surfaces becomes a problem for biomaterials above this threshold (O'Neill et al., 2018). In the present study, the critical roughness value previously reported as 0.2 μm was exceeded in the Taylor Hobson profilometer; by SF2 and TES in test groups at all toothbrushing solutions for all brushing cycles and at the end of 30,000 cycles by the negative control group and positive control group.

da Cas et al. (2013) obtained that regardless of the type of toothpaste, the final Ra values were found to be considerably higher than the initial values. However, they reported that scrubbing with distilled water was not significantly change the Ra value. The average roughness values of the resin composite groups brushed with distilled water were found to be partially consistent with the results of this study.

The inorganic matrix known as the filler system is usually added to the organic phase to provide a direct effect on the surface properties of the composite such as high wear resistance, hardness, improve the aesthetic appearance of the biomaterial and protect the organic matrix from wear directly against the force applied to the restoration. Simulated toothbrushing modifies the balance between organic matrix and filler particles. The Ra also depends on the microstructure of the resin composites used (Ruivo, Pacheco, Sebold, & Giannini, 2019; Soliman et al., 2020).

Al-Angari, Eckert, and Sabrah (2021) found that, while microhybrid resin composite had significantly higher roughness values compared with nanohybrid resin composite (HXU), no significant differences in surface roughness were found between enamel and nanohybrid resin composite (HXU). Similar to the results of this study, our study founded that enamel, HXU and SF2 have less surface roughness compared with TES. However, it is also remarkable that TES consists of silica glass particles, which increase the porosity of the biomaterial and, therefore, produce a higher level of surface roughness. This result can be explained by the less homogenous distribution of inorganic filler in microhybrid composites than in nanohybrid composites. This result was in accordance with Soliman et al. (2020), who stated that surface roughness was the linear relationship with the filler particle size of resin composite.

In the studies of Chesterman, Jowett, Gallacher, and Nixon (2017) reported that the SonicFill system (especially SonicFill 2) combined the advantages of a conventional resin composite with a flowable resin composite material using sonic activation, thereby achieving the highest degree of polymerization even in specimens with a thickness of 5 mm. Considering the generic properties of both resins in test group, the expectation is that the SF2 will evidence a lower performance in terms of surface roughness. It is considerable to note that this resin is composed of agglomerates of particles of zirconia–silica (nanoclusters), which also have a higher percentage of particulate inorganic filler. Thus, removing the clusters, which can act as a single unit on the composite surface, induces smaller irregularities lower in point of surface roughness (Mitra, Wu, & Holmes, 2003). As additional polymerization methods are applied in indirect composite systems, the conversion rate of monomers to polymer increases, and this increase has a positive effect on the mechanical properties of the resin composite (Azeem & Sureshbabu, 2018). In this case, sonic activation and the additional polymerization method are expected to reduce surface roughness. In the study, the surface roughness values were observed to be higher than expected compared with the control groups. In this study, the presence of sonic activation and an additional polymerization method did not significantly reduce the surface roughness of the resin composites as expected.

Inorganic filler particles are harder than the organic matrix. Therefore some particles protrude on the surface during finishing and polishing processes, while some leave gaps by breaking off the surface (Antonson et al., 2011; Zimmerli et al., 2011).

Some inorganic filler particles, which initially protruded from the aesthetic restorative materials, may have been displaced or wholly removed from the surface with the toothbrushing cycle up to 30,000 cycles. Therefore, it can be expected that TES, a microhybrid resin composite, will show higher surface roughness.

In the study, while the TES compared with the control groups in the Taylor Hobson profilometer always gave higher values, it showed lower values compared with the SF2. However, in the AFM line and

AFM surface measurements, the TES showed the highest surface roughness values and showed a statistically significant difference from the SF2. This difference in AFM measurements can be due to the precision of the measurement method, which perceivable the difference in inorganic filler particle sizes and the high filler ratio.

The first null hypothesis was partially rejected that the surface roughness of resin composites in different toothbrushing procedures is similar to human enamel.

Kakaboura, Fragouli, Rahiotis, and Silikas (2007) reported that a 2D surface profilometer determines line roughness in the linear or vertical direction, on the other hand, it describes the area roughness of the AFM viewed on an entire surface. For this reason, they thought that it was not appropriate to compare the AFM analogs (Sa) and 2D profilometer arithmetic Ra values. The 2D and 3D images were obtained similarly with this study and used for qualitative evaluations. Different from this study; In order to be comparable to the measurements made with the Taylor Hobson profilometer, surface roughness measurements were made with the AFM line method in the contact style of the AFM. In addition, they proved that AFM is a more reliable method for determining the surface quality of resin composites. The surface parameter value depends on the size of the area being examined. It has been reported in this study that lower Ra values can be obtained because the areas scanned with the AFM are small.

In this study, the surface characteristics were defined by both qualitative evaluations, assessed by AFM and SEM, and by quantitative measurements conducted by 2D and 3D profilometry. Similar to this study, AFM surface (Sa) and AFM line (Ra) measurements were made with the AFM method, and lower roughness values were obtained for all groups than the Taylor Hobson profilometer (2D profilometer). Based on the overall evaluation and ranking of the different measurement methods, the Taylor Hobson profilometer is underlined as the one that induces significant and higher surface roughness values. This shows that there is a difference in sensitivity between measurement methods. AFM needs precise measurements to visualize the surface topography of composite resins at the high spatial resolution; therefore, it is more sensitive to small surface variations.

The alternative hypotheses of the present study can be dismissed when the differences between various measurement methods, material groups, and toothbrushing solutions at the beginning and at the end of 30,000 cycles are evaluated. Assuming that there are statistically significant differences between toothbrushing solutions, for surface roughness measured by different measurement methods of human enamel, nanohybrid, and microfilled resin composites at the beginning and at the end of 30,000 cycles.

Fan, Chen, and Huang (2017) reported that it had been observed in micromorphological images that an aluminum-oxide-coated abrasive disc produced surfaces with the closest appearance to the original enamel surface but showed some deep scratches as well as shallow scratches. One-stage OneGloss polishing system, on the other hand, has been found to create the smoothest enamel surface by creating very few shallow scratches compared with abrasive disc systems. Previous studies reported that an aluminum-oxide-coated abrasive disc produced the smoothest surfaces for composite finishing and polishing procedures (Barbosa, Zanata, Navarro, & Nunes, 2005; Janus, Fauxpoint, Arntz, Pelletier, & Etienne, 2010; Venturini, Cenci, Demarco, Camacho, & Powers, 2006). When the literature is examined, there is no consensus on which of the multistage aluminum-oxide-coated abrasive disc systems and single-stage polishing systems provide a smoother surface. Based on the results of these studies, the flattest and smoothest surfaces were obtained by using both polishing systems sequentially. In the current study, it was determined that the scratches seen in the initial SEM images were caused by the finishing and polishing processes.

Lastly, the results refer to 3-year cycles of the brushed, so short-term results (such as 6 months to 1 year) could also be investigated. Further, more research work should be conducted to evaluate the effect of different cycles time, the toothbrush of diverse hardness, and type of bristle tips, and the consequence of dissimilar types of roughness measurement methods on the surface roughness of human enamel and various resin composites surface with different ingredient whitening toothpastes.

5 | CONCLUSION

Amongst the roughness measurement methods investigated in this study, AFM is the most susceptible technique in determining surface roughness. In addition, the results demonstrate that the silica-based blue covarine whitening toothpaste did not give rise to a concomitant statistically significant increase in the level of roughness to the enamel in AFM and can be an effective source of aiding remineralization. From the results of the current study, it can be considered that utilize toothpaste with blue covarine are a safe method to improve the whiteness of teeth in routine home tooth brushing. Thus, the present study reveals suitable material options for patients using the whitening toothpastes.

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DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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REFERENCES

Al-Angari, S. S., Eckert, G. J., & Sabrah, A. H. A. (2021). Color stability, roughness, and microhardness of enamel and composites submitted to staining/bleaching cycles. *Saudi Dental Journal*, *33*(4), 215–221. https://doi.org/10.1016/j.sdentj.2020.08.003

Al-Zarea, B. K. (2013). Satisfaction with appearance and the desired treatment to improve aesthetics. *International Journal of Dentistry*, *2013*, 912368. https://doi.org/10.1155/2013/912368

Amaral, C. M., Miranda, M. E., Correa, D. S., & Silva, E. M. (2014). Sodium fluoride and casein phosphopeptide-amorphous calcium phosphate cream plus sodium fluoride efficacy in preventing enamel erosion in a simulated oral environment study model. *Indian Journal of Dental Research*, *25*(4), 464–469. https://doi.org/10.4103/0970-9290.142536

Antonson, S. A., Yazici, A. R., Kilinc, E., Antonson, D. E., & Hardigan, P. C. (2011). Comparison of different finishing/polishing systems on surface roughness and gloss of resin composites. *Journal of Dentistry*, *39*, e9–e17. https://doi.org/10.1016/j.jdent.2011.01.006

Azeem, R. A., & Sureshbabu, N. M. (2018). Clinical performance of direct versus indirect composite restorations in posterior teeth: A systematic review. *Journal of Conservative Dentistry*, *21*(1), 2–9. https://doi.org/10.4103/JCD.JCD_213_16

Barbieri, G. M., Mota, E. G., Rodrigues-Junior, S. A., & Burnett, L. H., Jr. (2011). Effect of whitening dentifrices on the surface roughness of commercial composites. *Journal of Esthetic and Restorative Dentistry*, *23*(5), 338–345. https://doi.org/10.1111/j.1708-8240.2011.00426.x

Barbosa, S. H., Zanata, R. L., Navarro, M. F., & Nunes, O. B. (2005). Effect of different finishing and polishing techniques on the surface roughness of microfilled, hybrid and packable composite resins. *Brazilian Dental Journal*, *16*(1), 39–44. https://doi.org/10.1590/s0103-64402005000100007

Bielfeldt, S., Foltran, I., Bohling, A., Manger, C., & Wilhelm, K. P. (2018). The combined use of a nonabrasive, activator-containing toothpaste and a light emitting diode device improves the onset time of tooth whitening. *European Journal of Dentistry*, *12*(3), 329–333. https://doi.org/10.4103/ejd.ejd_167_18

Botta, A. C., Duarte, S., Jr., Paulin Filho, P. I., Gheno, S. M., & Powers, J. M. (2009). Surface roughness of enamel and four resin composites. *American Journal of Dentistry*, *22*(5), 252–254.

Chesterman, J., Jowett, A., Gallacher, A., & Nixon, P. (2017). Bulk-fill resin-based composite restorative materials: A review. *British Dental Journal*, *222*(5), 337–344. https://doi.org/10.1038/sj.bdj.2017.214

da Cas, N. V., Ruat, G. R., Bueno, R. P., Pachaly, R., & Pozzobon, R. T. (2013). Effect of whitening toothpaste on superficial roughness of composite resin. *General Dentistry*, *61*(4), e8–e11.

Da Costa, J., Adams-Belusko, A., Riley, K., & Ferracane, J. L. (2010). The effect of various dentifrices on surface roughness and gloss of resin composites. *Journal of Dentistry*, *38*, e123–e128.

da Rosa, G. M., da Silva, L. M., de Menezes, M., do Vale, H. F., Regalado, D. F., & Pontes, D. G. (2016). Effect of whitening dentifrices on the surface roughness of a nanohybrid composite resin. *European Journal of Dentistry*, *10*(2), 170–175. https://doi.org/10.4103/1305-7456.178305

Fan, X. C., Chen, L., & Huang, X. F. (2017). Effects of various debonding and adhesive clearance methods on enamel surface: An in vitro study. *BMC Oral Health*, *17*(1), 58. https://doi.org/10.1186/s12903-017-0349-6

Janus, J., Fauxpoint, G., Arntz, Y., Pelletier, H., & Etienne, O. (2010). Surface roughness and morphology of three nanocomposites after two different polishing treatments by a multitechnique approach. *Dental Materials*, *26*(5), 416–425. https://doi.org/10.1016/j.dental.2009.09.014

Joiner, A. (2010). Whitening toothpastes: A review of the literature. *Journal of Dentistry*, *38*, e17–e24. https://doi.org/10.1016/j.jdent.2010.05.017

Joiner, A., Philpotts, C. J., Ashcroft, A. T., Laucello, M., & Salvaderi, A. (2008). In vitro cleaning, abrasion and fluoride efficacy of a new silica based whitening toothpaste containing blue covarine. *Journal of Dentistry*, *36*, 32–37. https://doi.org/10.1016/j.jdent.2008.02.003

Kakaboura, A., Fragouli, M., Rahiotis, C., & Silikas, N. (2007). Evaluation of surface characteristics of dental composites using profilometry, scanning electron, atomic force microscopy and gloss-meter. *Journal of Materials Science. Materials in Medicine*, *18*(1), 155–163. https://doi.org/10.1007/s10856-006-0675-8

Kielbassa, A. M., Maier, M., Gieren, A. K., & Eliav, E. (2015). Tooth sensitivity during and after vital tooth bleaching: A systematic review on an unsolved problem. *Quintessence International*, *46*(10), 881–897. https://doi.org/10.3290/j.qi.a34700

Kumari, C. M., Bhat, K. M., & Bansal, R. (2016). Evaluation of surface roughness of different restorative composites after polishing using atomic force microscopy. *Journal of Conservative Dentistry*, *19*(1), 56–62. https://doi.org/10.4103/0972-0707.173200

Lagerweij, M. D., Buchalla, W., Kohnke, S., Becker, K., Lennon, A. M., & Attin, T. (2006). Prevention of erosion and abrasion by a high fluoride concentration gel applied at high frequencies. *Caries Research*, *40*(2), 148–153. https://doi.org/10.1159/000091062

Lippert, F. (2013). An introduction to toothpaste: Its purpose, history and ingredients. *Monographs in Oral Science*, *23*, 1–14. https://doi.org/10.1159/000350456

Meireles, S. S., de Sousa, J. P., Lins, R. B. E., & Sampaio, F. C. (2021). Efficacy of whitening toothpaste containing blue covarine: A double-blind controlled randomized clinical trial. *Journal of Esthetic and Restorative Dentistry*, *33*(2), 341–350. https://doi.org/10.1111/jerd.12605

Mitra, S. B., Wu, D., & Holmes, B. N. (2003). An application of nanotechnology in advanced dental materials. *Journal of the American Dental Associatio*, *134*(10), 1382–1390. https://doi.org/10.14219/jada.archive.2003.0054

O'Neill, C., Kreplak, L., Rueggeberg, F. A., Labrie, D., Shimokawa, C. A. K., & Price, R. B. (2018). Effect of tooth brushing on gloss retention and surface roughness of five bulk-fill resin composites. *Journal of Esthetic and Restorative Dentistry*, *30*(1), 59–69. https://doi.org/10.1111/jerd.12350

Ruivo, M. A., Pacheco, R. R., Sebold, M., & Giannini, M. (2019). Surface roughness and filler particles characterization of resin-based composites. *Microscopy Research and Technique*, *82*(10), 1756–1767. https://doi.org/10.1002/jemt.23342

Santos, M. R., De Carvalho, F. B., Cangussu, M. C. T., Barros, L. R., & Trindade, R. F. (2020). Spectrophotometric analysis of the effectiveness of bleaching agents used for non-vital teeth bleaching. *Journal of Health & Biological Sciences*, *8*(1), 1–6. https://doi.org/10.12662/2317-3076jhbs.v8i1.2936.p1-6.2020

Shamel, M., Al-Ankily, M. M., & Bakr, M. M. (2019). Influence of different types of whitening tooth pastes on the tooth color, enamel surface roughness and enamel morphology of human teeth. *F1000Research*, *8*, 1764. https://doi.org/10.12688/f1000research.20811.1

Soliman, H. A., Elkholany, N. R., Hamama, H. H., El-Sharkawy, F. M., Mahmoud, S. H., & Comisi, J. C. (2020). Effect of different polishing systems on the surface roughness and gloss of novel nanohybrid resin composites. *European Journal of Dentistry*, *15*(2), 259–265. https://doi.org/10.1055/s-0040-1718477

Tao, D., Smith, R. N., Zhang, Q., Sun, J. N., Philpotts, C. J., Ricketts, S. R., … Joiner, A. (2017). Tooth whitening evaluation of blue covarine containing toothpastes. *Journal of Dentistry*, *67*, S20–S24. https://doi.org/10.1016/j.jdent.2017.10.014

Tellefsen, G., Liljeborg, A., Johannsen, A., & Johannsen, G. (2011). The role of the toothbrush in the abrasion process. *International Journal of Dental Hygiene*, *9*(4), 284–290. https://doi.org/10.1111/j.1601-5037.2011.00505.x

Tholt, B., Miranda-Júnior, W. G., Prioli, R., Thompson, J., & Oda, M. (2006). Surface roughness in ceramics with different finishing techniques using atomic force microscope and profilometer. *Operative Dentistry*, *31*(4), 442–449.

Vaz, V. T. P., Jubilato, D. P., Oliveira, M. R. M., Bortolatto, J. F., Floros, M. C., Dantas, A. A. R., & Oliveira Junior, O. B. (2019). Whitening toothpaste containing activated charcoal, blue covarine, hydrogen peroxide or microbeads: Which one is the most effective? *Journal of*

Applied Oral Science, *27*, e20180051. https://doi.org/10.1590/1678-7757-2018-0051

Venturini, D., Cenci, M. S., Demarco, F. F., Camacho, G. B., & Powers, J. M. (2006). Effect of polishing techniques and time on surface roughness, hardness and microleakage of resin composite restorations. *Operative Dentistry*, *31*(1), 11–17. https://doi.org/10.2341/04-155

Vinita Mary, A., Kesavan, R., Bhavani, M., Blessy Melba, D., Deepiha, D., & Monisha Shrinidhi, S. (2020). Awareness about teeth whitening among young adults in south Indian population. *IJARIIT*, *6*(6), 20–24.

Wegehaupt, F. J., & Attin, T. (2010). The role of fluoride and casein phosphopeptide/amorphous calcium phosphate in the prevention of erosive/abrasive wear in an in vitro model using hydrochloric acid. *Caries Research*, *44*(4), 358–363. https://doi.org/10.1159/000316542

Widodo, P. N., Soetjipta, N. L., & Palupi, R. (2020). Perception and knowledge of patients towards teeth whitening treatment. *EurAsian Journal of BioSciences*, *14*(2), 3759–3764.

Xiao, J., Zhou, X. D., Zhu, W. C., Zhang, B., Li, J. Y., & Xu, X. (2007). The prevalence of tooth discolouration and the self-satisfaction with tooth colour in a Chinese urban population. *Journal of Oral Rehabilitation*, *34*(5), 351–360. https://doi.org/10.1111/j.1365-2842.2007.01729.x

Zimmerli, B., Lussi, A., & Flury, S. (2011). Operator variability using different polishing methods and surface geometry of a nanohybrid composite. *Operative Dentistry*, *36*(1), 52–59. https://doi.org/10.2341/10-096-LR1

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