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Effects of various chair-side surface treatment methods on dental restorative materials with respect to contact angles and surface roughness

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Available chair-side surface treatment methods may adversely affect prosthetic materials and promote plaque accumulation. This study investigated the effects of treatment procedures on three resin restorative materials, zirconium-dioxide and polyetheretherketone in terms of surface roughness and hydrophobicity. Treatments were grinding with silicon carbide paper or white Arkansas stone, blasting with prophylaxis powder and polishing with diamond paste. Surface roughness was assessed using confocal laser scanning. Hydrophobicity as measured by water contact angle was determined by computerized image analysis using the sessile drop technique. All of the specific surface treatments performed led to significant changes in contact angle values and surface roughness (Ra) values. Median contact angle values ranged from 51.6° to 114°. Ra values ranged from 0.008 µm to 2.917 µm. Air-polishing as well as other polishing procedures increased surface roughness values in all materials except zirconium dioxide. Polyetheretherketone displayed greatest change in contact angle values after air-polishing treatment.

Keywords: Plaque, Inorganic fillers, Polyether ether ketone, Contact angle, Air-polish

INTRODUCTION

In recent years, there has been growing interest in the use of dental resin composites as direct restorative materials in clinical dentistry[1,2]. The mechanical and physical properties of composites made up of a resin matrix and filler materials have been improved[2-4]. The application of tooth-colored restorations has greatly increased due to aesthetic demands.

Polyether ether ketone (PEEK) is an advanced biocompatible material used in the field of restorative and prosthetic dentistry that features a natural tooth-colored appearance[5]. As a result of the trend towards fixed full-arch restorations on a reduced number of implants, a stable framework and different dental resin materials for customized veneering are indispensable[6]. PEEK offers high strength, low moisture absorption and similar elasticity as bone and is used for telescopic and precision attachments as well as implant-supported suprastructures[7].

However, plaque accumulation under and around restorations is still a common problem and the main reason for the alteration and replacement of direct restorations[1,2,8,9]. Especially in the field of prosthetic dentistry, the control and removal of dental plaque deposits and subsequent polishing seems to be essential for the longevity of restorations. Air-polishing devices (APDs) have become an effective tool for plaque control and are applied routinely in professional dental cleaning[10-14]. However, adverse effects of APDs on dental restorative materials have been reported. Previous studies have found that the use of APDs increases surface roughness and leads to alteration of the surface integrity of restorative materials[15-19]. The adhesion of oral microorganisms is also significantly influenced by various substratum properties. Surface roughness of intraoral dental materials seems to be of great clinical importance in terms of bacterial retention, and changes in surface roughness might facilitate the prevention of caries, gingivitis, periodontitis, peri-implantitis and stomatitis. Rough surfaces provide opportunities for bacterial adhesion by increasing the surface area[20-25]. In addition, *Candida albicans* adhesion is enhanced if the roughness of the biomaterial is increased, and this biofilm has been shown to potentially initiate inflammatory diseases of the oral mucosa[26]. Low roughness and low energy surfaces have been proven to be fundamental properties of restorative materials for reducing bioadhesion in the oral cavity[24,25,27,28]. Furthermore, surface free energy (SFE), electrical properties and hydrophobicity of the substratum affect the accumulation of biofilm[29,30].

According to Combe *et al.*, SFE is not desirable if plaque resistance is needed, and restorative materials with low SFE are more likely to resist plaque formation[31]. Satou *et al.* state that the surface contact angles can be measured as an index of hydrophobicity. Their findings indicate that hydrophobic interaction plays a more important role than electrostatic interaction in the adherence of bacteria with pronounced hydrophobic

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surface properties[32]. Conflicting reports relate plaque formation to substratum hydrophobicity. Some previous studies conclude that dental plaque formation is greater on hydrophilic restorative materials such as porcelain and metals than on hydrophobic materials such as amalgams and resins[25,29,33-35].

Contrary studies, however, report greater plaque formation on hydrophobic materials and significantly lower adhesion to ceramics than to composite resin surfaces of polymeric origin or amalgams[36-39]. Other studies have indicated that bacterial hydrophobicity must also be investigated[32,40-43]. Dental plaque not only adheres better but also accumulates more quickly on rough surfaces[44]. Thus a smooth surface with low surface free energy is important to modulate biofilm adherence and growth. An improvement in surface conditions could be achieved if resin composites were carefully polished[8,29,37,45]. In addition, biofilm formation is also influenced by the type (chemical composition) of resin biomaterial[3,24,45-47]. Furthermore, it has been stated that the siloxane backbone provides the restorative material with its hydrophobic nature[48,49]. Burgers *et al.* conclude that this leads to reduced bacterial adhesion in comparison to other materials[2].

In addition, bacterial adherence also seems to be affected by the size and position of fillers and the matrix monomer of dental resin composites. Findings indicate that filler particles that are exposed by finishing and polishing procedures after composite restoration play an important role in plaque formation[50,51].

Polished composite surfaces seem to be rather rough due to the loss of superficial filler particles during the polishing process, thus promoting plaque accumulation[4,22,52].

In an effort to prevent caries, gingivitis, periodontitis, peri-implantitis and stomatitis, dental materials with a low susceptibility to bacterial adhesion are preferable for the longevity of restorations and teeth[27,53-56].

In consideration of this information, this study was designed to characterize test surfaces of commonly used composite resins, a zirconium dioxide and a polyether ether ketone in terms of roughness, surface free energy and chemical composition. The aim of the measurement was to evaluate available chair-side surface conditioning methods with regard to changes in contact angles and wettability and to relate the outcomes to possible bacterial adhesion.

In the current study, we hypothesize that the use of different surface treatment methods does have a significant impact on the properties of the materials that were tested.

MATERIALS AND METHODS

In total, 160 specimens were investigated. The materials tested ranged across different classes. Three commercially available resin restorative dental materials, a non- filled polymethyl methacrylate (PMMA-noF), an inorganic nano-filled dimethylacrylate (DMA-nano), an inorganic filled polymethyl methacrylate (PMMA-DMA), an inorganic filled polyether ether ketone (PEEK-IOF) and a zirconium dioxide (ZrO) were assessed in this study. Table 1 lists the five restoratives investigated, including material information. Ten additional zirconium dioxide discs, which were not subjected to any treatment modality, served as reference material. Cylindrical zirconium dioxide specimens (ZrO) and polyether ether ketone (PEEK-IOF) specimens (10 mm in diameter, 2 mm in height) were industrially manufactured and supplied by Bredent Medical.

Thirty specimens were prepared for each of the three resin restoratives (20×10×2 mm). All materials were handled in strict compliance with their manufacturers' instructions and were prepared by light polymerization to ensure the fullest possible conversion of monomer into polymer as residual monomer would affect the property of the tested materials and thus influence the contact angle measurement.

Each specimen was then manually polished using 1000 grit siliciumcarbide paper (Buehler, Duesseldorf, Germany) in order to achieve a similar degree of surface roughness in all specimens. This was done to reduce possible surface roughness effects, so that the differences in subsequent measurements after modification of the surface would only result from the properties and composition of the specific materials and could be clearly evaluated. Baseline confocal laser scanning measurements and contact angle determination were obtained on all samples prior to further surface treatment.

After preparation, the specimens were ultrasonically cleansed with isopropanol (70%) for 15 min to remove any embedded grinding material and washed twice in sterile distilled water before air drying according to the manufacturers instructions of the contact angle measuring device. The specimens were stored overnight in a desiccator to improve the dryness of the material. Confocal laser scanning and contact angle measurements were performed.

All specimens were thereafter divided into 15 equal groups, which were subsequently subjected to different treatments. Sixty contact angle measurements and 10 confocal laser scanning measurements were performed within each group of 10 specimens. The same cleaning and drying procedures were again applied prior to further confocal laser scanning examinations and contact angle measurements.

Treatment modalities

Ten specimens of each group were subjected to each treatment modality. The selected surface treatments were analogous to those routinely applied in lab-technical and clinical settings in chair-side dentistry.

Group 1: Paper-grinded Surface was ground with 1000 grit siliciumcarbide paper (Buehler), performing a one-way, straight-line motion.

Group 2: Stone-grinded Surface was equally ground with a cylindrical white Arkansas stone (4 mm diameter, Meisinger, Hafer &

Table 1 Materials assessed in this study

Abbreviation	Material	Lot #	Brand name	Filler	Content of fillers
PEEK-IOF	PEEK	379805	BioHPP	inorganic ceramics and metal oxides	<30%
PMMA-noF	PMMA, MMA, EGDMA	374873	Breformance	No fillers	No fillers
DMA-nano	Bis-GMA, UDMA and aliphatic Dimethacrylate resins	123765	CreaLign	inorganic ceramic fillers	~50%
PMMA-DMA	High molecular PMMA und Dimethacrylate	3.1/120609	NovoLign	inorganic ceramic fillers	<10%
ZrO	Yttriumoxide, partially stabilized isostatically pressed ZrO_2	378421	Brezirkon	Alumina	0.2–0.5%

Meisinger, Neuss, Germany, batch # Q27492), performing a one-way, straight-line motion with a straight hand piece and 20.000 rpm[57].

Group 3: Air-polished Surface was air-polished with sodium bicarbonate prophylaxis powder (Air Flow Classic, EMS, Nyon, Switzerland, batch # 1210051) using a standard air-polishing unit (EMS handy). The application time was 10 s at an approximate distance of 5 mm. The working pressure was kept at 60 psi. The mean particle size of the sodium bicarbonate particles ejected was 65 µm. The nozzle of the instrument was kept at a 45 degree angle to the specimen surface, and a constant straight line motion was performed. To ensure maximum reproducibility, the instrument powder chamber was refilled after each air-polishing period.

Group 4: High polished Surface was polished to high gloss with a 1 µm diamond paste (ZirPolish, Bredent, Senden, Germany, Ref. # 36010025) using a cotton buff.

All surface treatments were performed by the same trained operator to achieve a homogenous surface.

Surface roughness analysis

Surface roughness and surface area were determined on the prepared surfaces using a confocal laser scanning microscope (µsurf explorer, NanoFocus, Oberhausen, Germany). Analysis was performed on all test objects using the µsoft analysis premium program (NanoFocus), and an area of 320×320 µm was measured on each surface. In this context, roughness does not refer to macroscopic grooves and pits, which might be present on the materials tested, but to microscopic irregularities in the surface structure. To describe the surface structure numerically, Ra, Rz and Sa were used.

Sa gives a three-dimensional description of the arithmetic height deviation from a mean plane and is the parameter corresponding to the two-dimensional parameter Ra, describing the average surface roughness by reading the maximum peak to valley heights of a certain surface profile[22]. Rz describes the mean roughness depth and is calculated by measuring the vertical distance from the highest peak to the lowest valley within five sampling lengths and then averaging these distances.

Contact angle measurement

The contact angle reflects the interactions of fluids with solid surfaces, which depends on the polarity, hydrophobicity and wettability of the involved components[27,58]. The hydrophobicity of all test and reference materials was evaluated by measuring water contact angles. The computerized contact angle system EasyDrop DSA 100 (Krüss, Hamburg, Germany) was used in combination with Easy Drop Shape Analysis DSA1 v 1.90 software (Krüss) for image analysis and contact angle calculation. A time frame of 30 s for each measurement was recorded and evaluated. Two measurements (right and left contact angle) were carried out for each droplet. Droplets were generated manually, and contact angles were determined at 23°C using the sessile drop technique. The deionized distilled water used for the measurements was of HPLC (High Pressure Liquid Chromatography) quality (Sigma-Aldrich, St. Louis, USA, Lot # BCBH4122V). The procedure for measuring the contact angle was the same for all groups of specimens and was performed by the same trained operator.

Statistical analysis

The data were analyzed by using descriptive statistics including means and standard deviations. One-way analysis of variance (ANOVA) and *post hoc* Bonferroni tests were used to determine differences among the material groups and obtained surface treatments. The same level of significance (α=0.05) was used throughout the study. Continuous data were summarized by using medians and interquartile ranges (25th to 75th

percentile). Calculations were done using statistical software SPSS 23.0 for Windows (SPSS, Chicago, IL, USA).

RESULTS

For surface roughness analysis, 310 measurements were carried out in total, providing 930 individual values for analysis from the confocal laser morphological image analysis (Figs. 1–20). Paper-grinded PMMA-DMA displayed the lowest Ra value (0.008 μm±0.0025), whereas air-polished PMMA-noF displayed the highest Ra value (2.917 μm±0.4709).The arithmetic means and standard deviations of the surface roughness values (Ra) for the five materials tested, treated with different surface procedures, are reported in Table 2. The results demonstrate a significant increase in surface roughness after polishing procedures in all groups except for the ZrO group, which displayed a significant reduction in surface roughness. PMMA-noF samples and PMMA-DMA samples displayed a highly significant (p<0.001 one-way ANOVA) change in surface roughness (Ra values) after exposure to APD (Figs. 21–24). The highest Sa values were recorded for PMMA-noF samples after APD application (6.197 μm±0.9268). The summary of the ANOVA for surface roughness measurements is presented in Tables 3 and 4. Statistical analysis of the data indicated significant differences in surface roughness between the groups of restoratives (p< 0.001) and the surface treatments (p<0.001).

Table 3a shows differences in surface treatment methods (Ra) within the groups. *Post hoc* Bonferroni test results for Ra, Rz and Sa are displayed in Tables 4a–c.

For the determination of hydrophobicity, 1,210 measurements were carried out in total, providing 3,630 individual values for analysis. The median values from

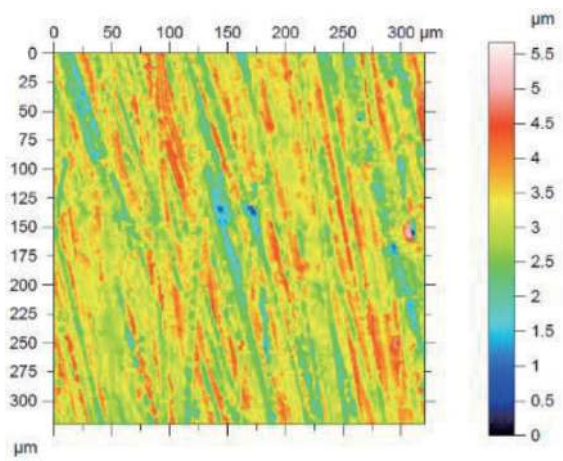

Fig. 1 Confocal laser scanning image of a paper-grinded Peek-IOF sample (Ra value 0.366 μm).

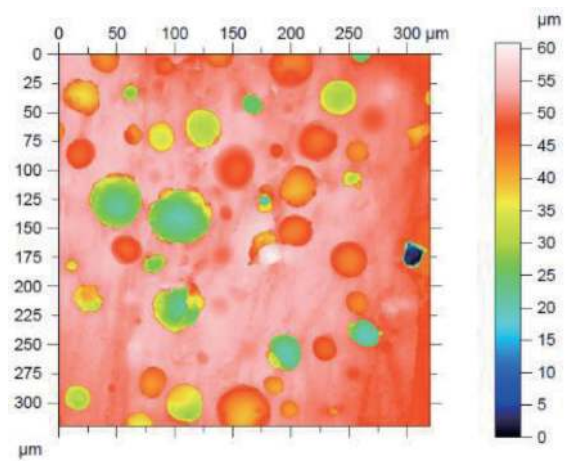

Fig. 2 Confocal laser scanning image of a paper-grinded PMMA-noF sample (Ra value 1.22 μm).

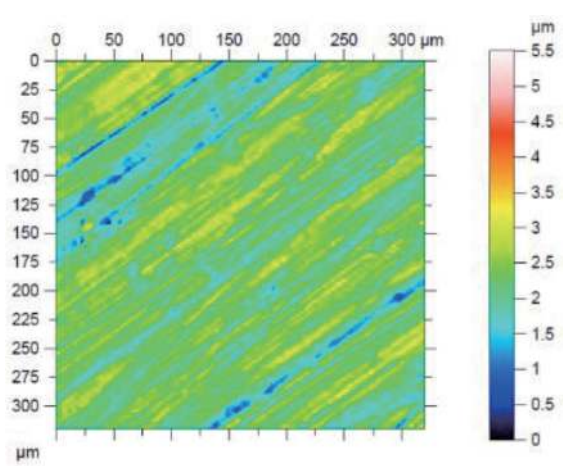

Fig. 3 Confocal laser scanning image of a paper-grinded DMA-nano sample (Ra value 0.223 μm).

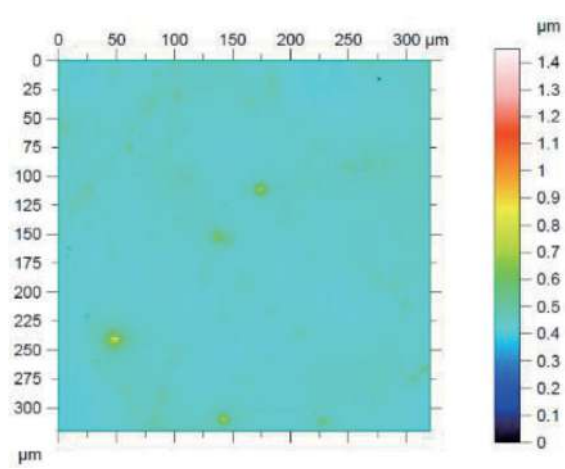

Fig. 4 Confocal laser scanning image of a paper-grinded PMMA-DMA sample (Ra value 0.00565 μm).

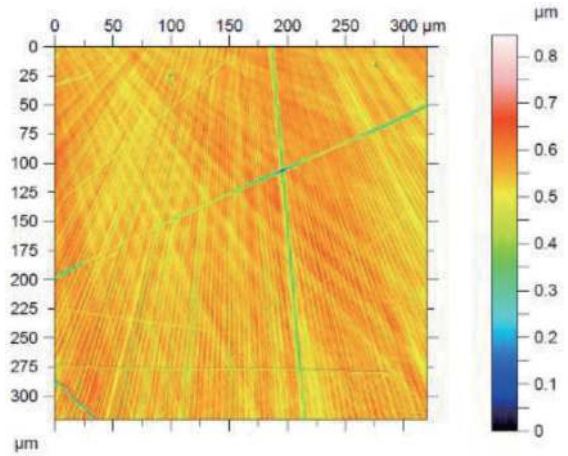

Fig. 5 Confocal laser scanning image of a paper-grinded ZrO sample (Ra value 0.0307 µm).

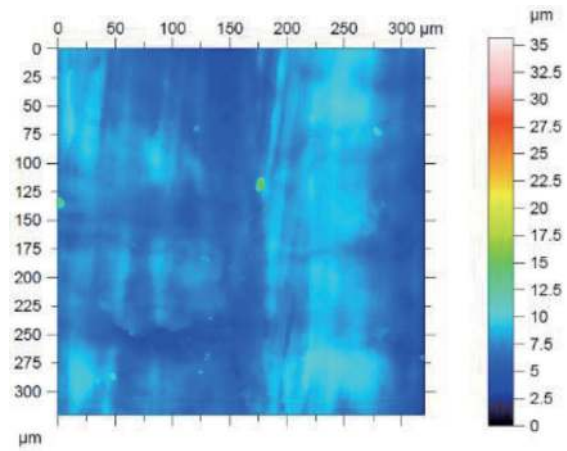

Fig. 6 Confocal laser scanning image of a stone-grinded PEEK-IOF sample (Ra value 0.378 µm).

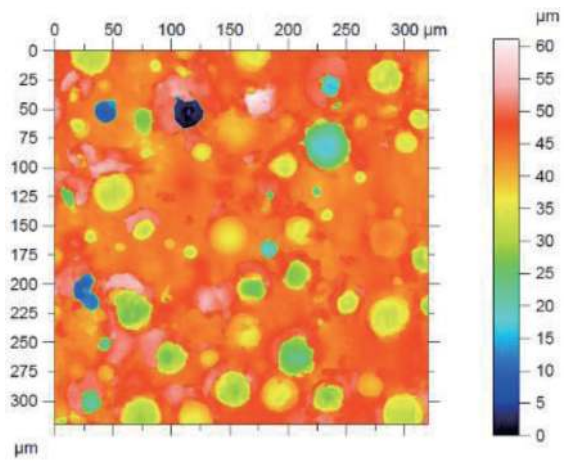

Fig. 7 Confocal laser scanning image of a stone-grinded PMMA-noF sample (Ra value 2.41 µm).

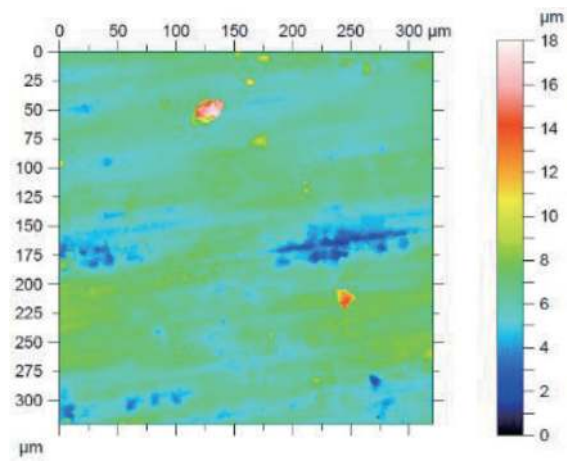

Fig. 8 Confocal laser scanning image of a stone-grinded DMA-nano sample (Ra value 0.248 µm).

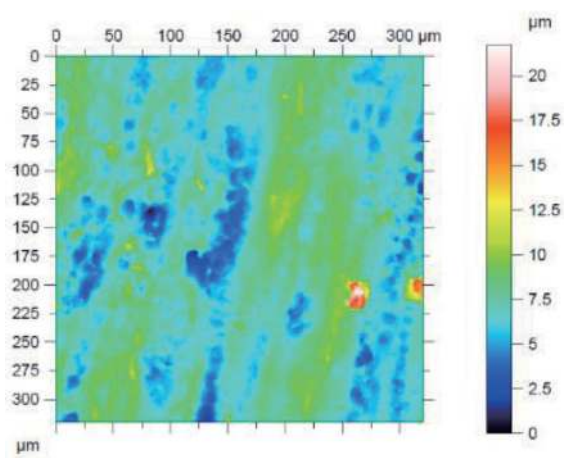

Fig. 9 Confocal laser scanning image of a stone-grinded PMMA-DMA sample (Ra value 0.578 µm).

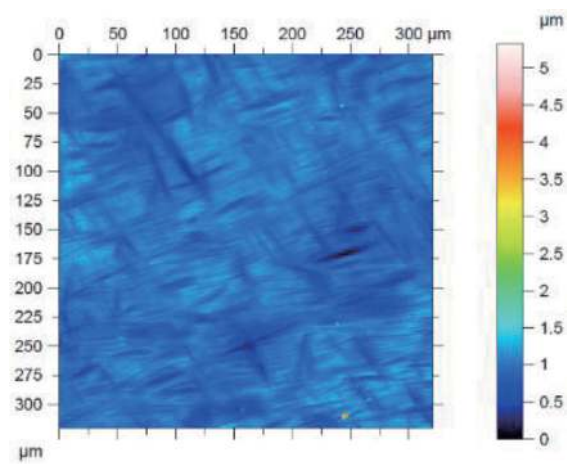

Fig. 10 Confocal laser scanning image of a stone-grinded ZrO sample (Ra value 0.077 µm).

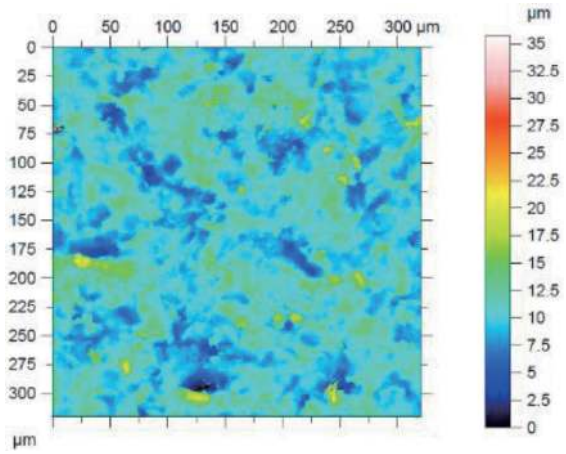

Fig. 11 Confocal laser scanning image of an air-polished PEEK-IOF sample (Ra value 0.995 µm).

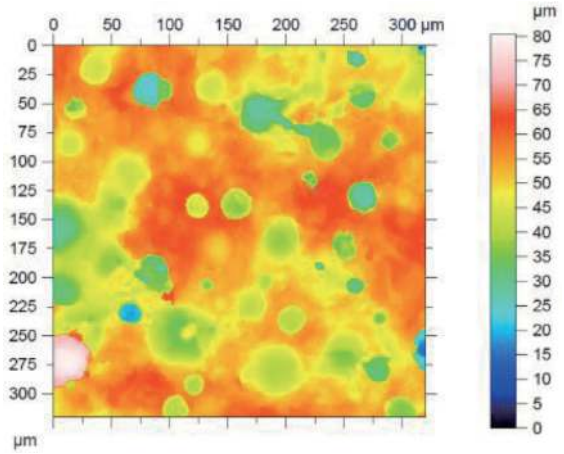

Fig. 12 Confocal laser scanning image of an air-polished PMMA-noF sample (Ra value 3.4 µm).

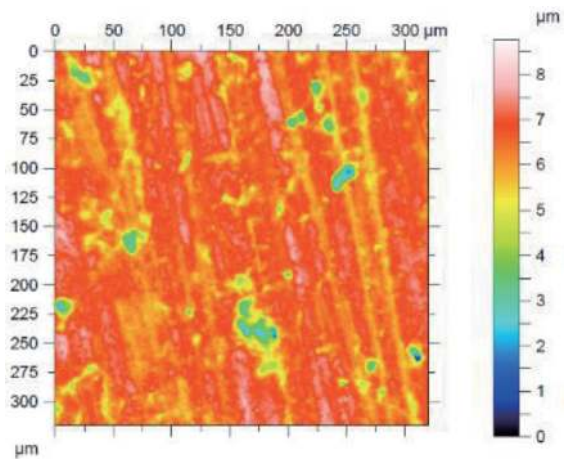

Fig. 13 Confocal laser scanning image of an air-polished DMA-nano sample (Ra value 0,376 µm).

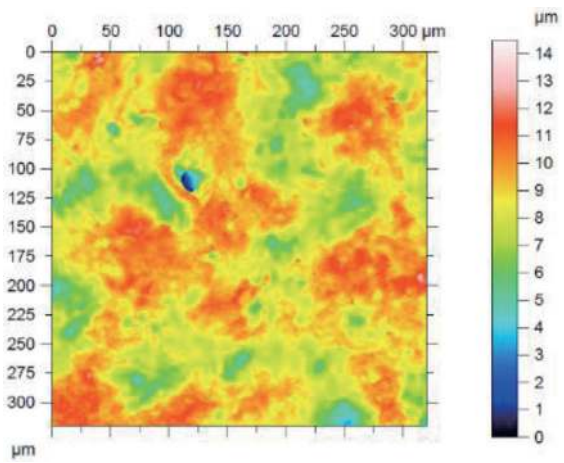

Fig. 14 Confocal laser scanning image of an air-polished PMMA-DMA sample (Ra value 0.562 µm).

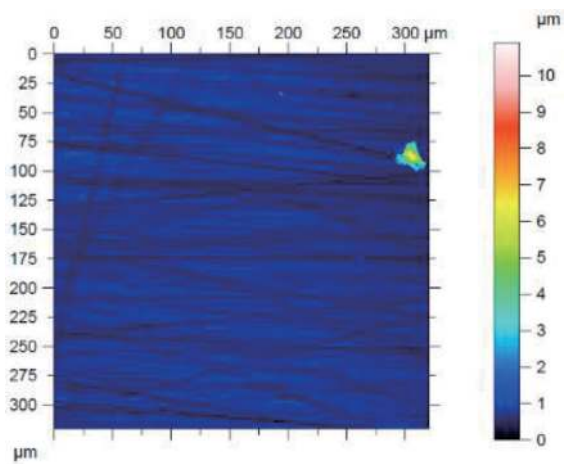

Fig. 15 Confocal laser scanning image of an air-polished ZrO sample (Ra value 0.059 µm).

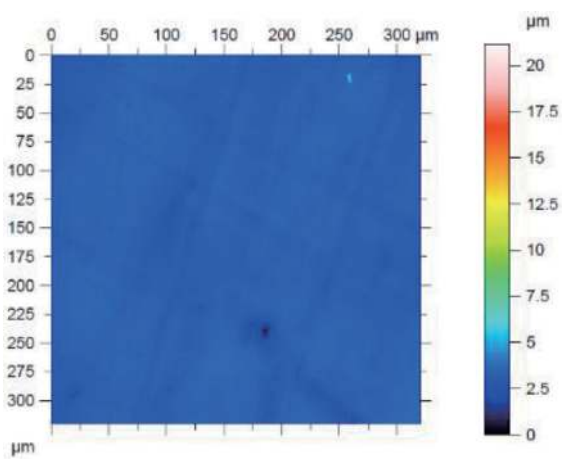

Fig. 16 Confocal laser scanning image of a high-polished PEEK-IOF sample (Ra value 0.065 µm).

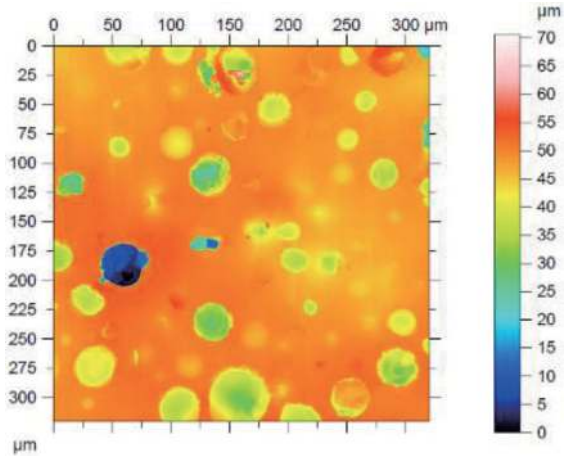

Fig. 17 Confocal laser scanning image of a high-polished PMMA-noF sample (Ra value 1.49 µm).

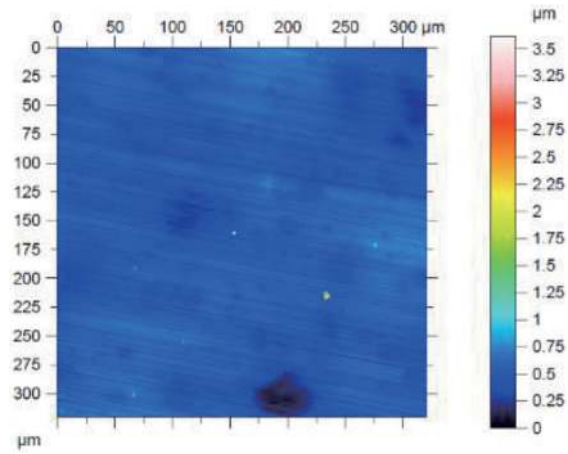

Fig. 18 Confocal laser scanning image of a high-polished DMA-nano sample (Ra value 0.045 µm).

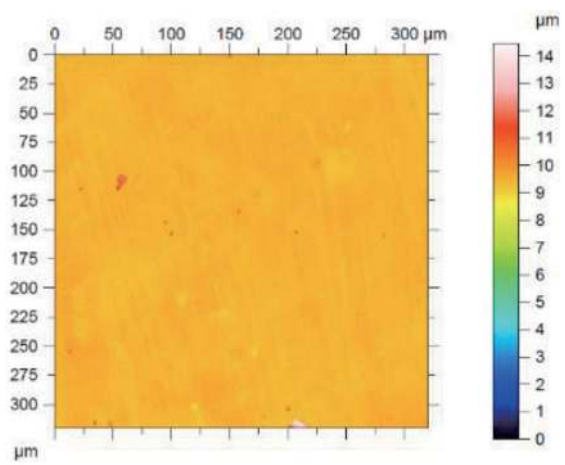

Fig. 19 Confocal laser scanning image of a high-polished PMMA-DMA sample (Ra value 0.042 µm).

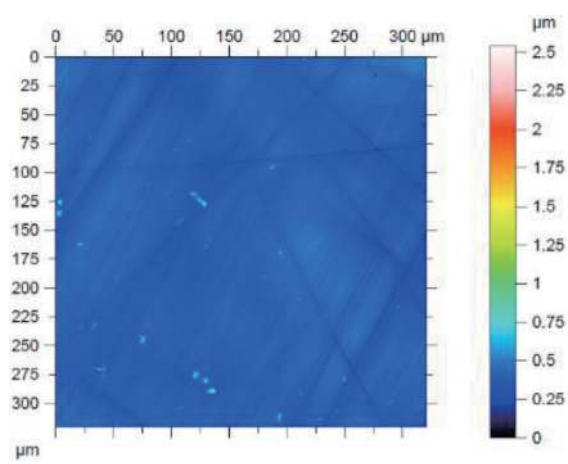

Fig. 20 Confocal laser scanning image of a high-polished ZrO sample (Ra value 0.017 µm).

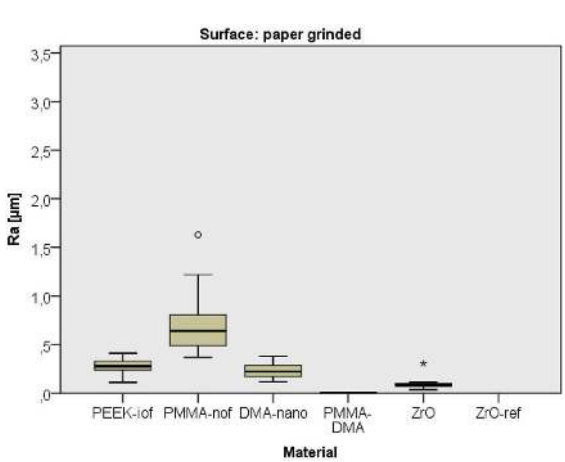

Fig. 21 Surface roughness of the five tested materials after paper grinding.

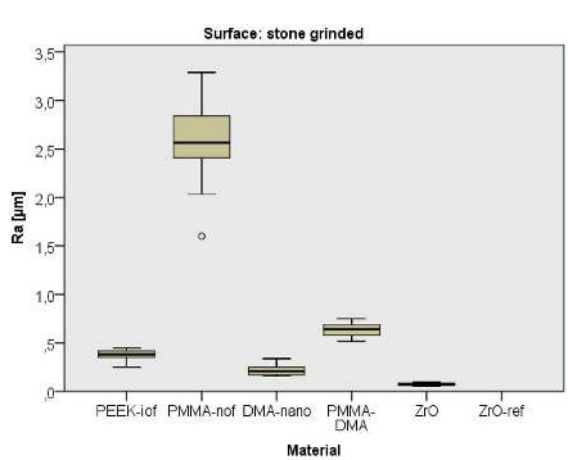

Fig. 22 Surface roughness of the five tested materials after stone grinding.

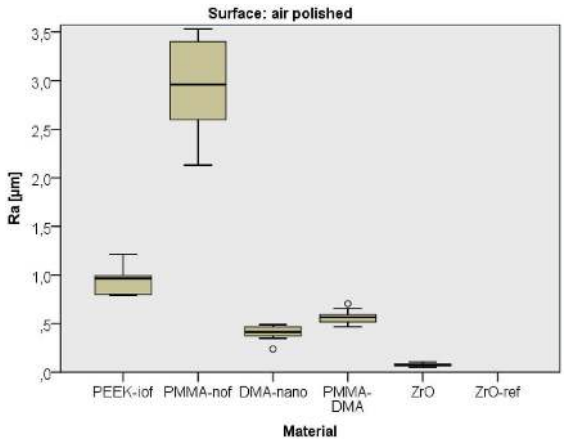

Fig. 23 Surface roughness of the five tested materials after APD treatment.

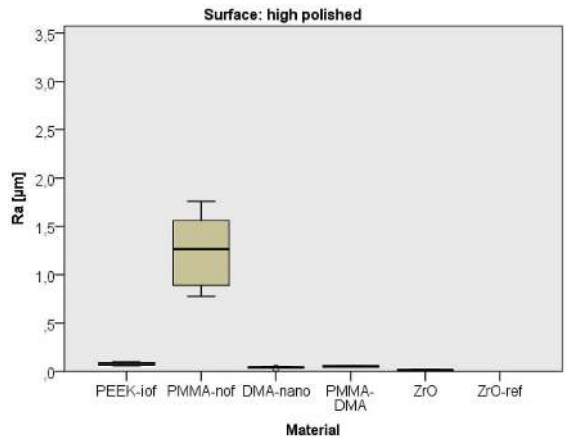

Fig. 24 Surface roughness of the five tested materials after high polishing.

Table 2 Surface roughness values (µm), mean ±standard deviations

Material	Surface treatment	Ra mean	SD	Rz mean	SD	Sa mean	SD
PEEK-IOF	Paper-grinded	0.277	0.0664	1.589	0.2957	0.547	0.1023
	Stone-grinded	0.364	0.0657	1.959	0.1854	1.114	0.1356
	Air-polished	0.952	0.1359	5.613	0.2558	1.505	0.1705
	High-polished	0.073	0.0128	0.501	0.0448	0.148	0.0384
PMMA-noF	Paper-grinded	0.703	0.2867	4.003	1.3486	4.743	1.0355
	Stone-grinded	2.567	0.4929	13.050	0.9857	5.103	0.7687
	Air-polished	2.917	0.4709	13.930	1,1547	6.197	0.9268
	High-polished	1.260	0.3529	6.733	0.7229	3.303	0.6909
DMA-nano	Paper-grinded	0.236	0.0727	1.349	0.3917	0.357	0.0712
	Stone-grinded	0.218	0.0588	1.261	0.2709	0.907	0.2020
	Air-polished	0.405	0.0742	2.249	0.1588	0.632	0.1852
	High-polished	0.399	0.0038	0.245	0.0243	0.108	0.0585
PMMA-DMA	Paper-grinded	0.008	0.0025	0.800	0.0280	0.020	0.0070
	Stone-grinded	0.633	0.0739	3.543	0.3182	1.378	0.3055
	Air-polished	0.567	0.0725	3.200	0.1053	1.076	0.1495
	High-polished	0.050	0.0064	0.328	0.0255	0.075	0.0117
ZrO	Paper-grinded	0.091	0.0449	0.519	0.1299	0.097	0.0243
	Stone-grinded	0.073	0.0127	0.419	0.0426	0.106	0.0157
	Air-polished	0.076	0.0148	0.464	0.0954	0.095	0.0088
	High-polished	0.103	0.0036	0.108	0.0427	0.023	0.0079
ZrO reference		0.058	0.0173	0.352	0.1238	0.073	0.0179

Table 3 Summary of ANOVA mean roughness values Ra (μm)

Group (Material)		Sum of squares	df*	Mean square	*F***	Significance
PEEK-IOF	Between Groups	4.527	3	1.509	252.513	<0.001***
	Within Groups	0.335	56	0.006		
	Total	4.862	59			
PMMA-noF	Between Groups	51.052	3	17.017	123.928	<0.001***
	Within Groups	7.690	56	0.137		
	Total	58.741	59			
DMA-nano	Between Groups	0.671	3	0.224	53.431	<0.001***
	Within Groups	0.234	56	0.004		
	Total	0.906	59			
PMMA-DMA	Between Groups	4.551	3	1.517	874.641	<0.001***
	Within Groups	0.097	56	0.002		
	Total	4.648	59			
ZrO	Between Groups	0.046	3	0.015	13.762	<0.001***
	Within Groups	0.062	56	0.001		
	Total	0.108	59			

* df: degrees of freedom
** *F*: Variance ratio value
*** statistically significant

Table 3a *Post hoc* Bonferroni tests for Ra

Material	Surface	Surface	Mean difference	Standard error	Significance
PEEK-IOF	Paper-grinded	Stone-grinded	−0.087	0.028	0.018*
		Air-polished	−0.675	0.028	0.000*
		High-polished	0.203	0.028	0.000*
	Stone-grinded	Paper-grinded	0.087	0.028	0.018*
		Air-polished	−0.587	0.034	0.000*
		High-polished	0.291	0.034	0.000*
	Air-polished	Paper-grinded	0.675	0.028	0.000*
		Stone-grinded	0.587	0.034	0.000*
		High-polished	0.878	0.034	0.000*
	High-polished	Paper-grinded	−0.203	0.028	0.000*
		Stone-grinded	−0.291	0.034	0.000*
		Air-polished	−0.878	0.034	0.000*
PMMA-noF	Paper-grinded	Stone-grinded	1.863	0.135	0.000*
		Air-polished	−2.213	0.135	0.000*
		High-polished	−0.557	0.135	0.001*
	Stone-grinded	Paper-grinded	1.863	0.135	0.000*
		Air-polished	−0.350	0.165	0.235
		High-polished	1.306	0.165	0.000*
	Air-polished	Paper-grinded	2.213	0.135	0.000*
		Stone-grinded	0.350	0.165	0.235
		High-polished	1.656	0.165	0.000*
	High-polished	Paper-grinded	0.557	0.135	0.001*
		Stone-grinded	−1.306	0.165	0.000*
		Air-polished	−1.656	0.165	0.000*

Table 3a continued

Material	Surface	Surface	Mean difference	Standard error	Significance
DMA-nano	Paper-grinded	Stone-grinded	0.018	0.023	1.000
		Air-polished	−0.168	0.023	0.000*
		High-polished	0.196	0.023	0.000*
	Stone-grinded	Paper-grinded	−0.018	0.023	1.000
		Air-polished	−0.187	0.028	0.000*
		High-polished	0.178	0.028	0.000*
	Air-polished	Paper-grinded	0.168	0.023	0.000*
		Stone-grinded	0.187	0.028	0.000*
		High-polished	0.365	0.028	0.000*
	High-polished	Paper-grinded	−0.196	0.023	0.000*
		Stone-grinded	−0.178	0.028	0.000*
		Air-polished	−0.365	0.028	0.000*
PMMA-DMA	Paper-grinded	Stone-grinded	−0.625	0.015	0.000*
		Air-polished	−0.559	0.015	0.000*
		High-polished	−0.041	0.015	0.047*
	Stone-grinded	Paper-grinded	0.625	0.015	0.000*
		Air-polished	0.066	0.018	0.005*
		High-polished	0.583	0.018	0.000*
	Air-polished	Paper-grinded	0.559	0.015	0.000*
		Stone-grinded	−0.066	0.018	0.005*
		High-polished	0.517	0.018	0.000*
	High-polished	Paper-grinded	0.041	0.015	0.047*
		Stone-grinded	−0.583	0.018	0.000*
		Air-polished	−0.517	0.018	0.000*
ZrO	Stone-grinded	Paper-grinded	−0.018	0.012	0.816
		Air-polished	−0.002	0.014	1.000
		High-polished	0.059	0.014	0.001*
	Air-polished	Paper-grinded	−0.015	0.012	1.000
		Stone-grinded	0.002	0.014	1.000
		High-polished	0.062	0.014	0.001*
	High-polished	Paper-grinded	−0.078	0.012	0.000*
		Stone-grinded	−0.059	0.014	0.001*
		Air-polished	−0.062	0.014	0.001*
	Paper-grinded	Stone-grinded	−0.009	0.007	1.000
		Air-polished	0.001	0.007	1.000
		High-polished	0.073	0.007	0.000*

* statistically significant

the contact angle measurement range from 51.6° to 114° (Figs. 25–28). Air-polished ZrO samples displayed the lowest contact angle values (51.6°±1.16), whereas air-polished PMMA-noF samples displayed the highest contact angle values (114.0°±6.46).

The arithmetic means and standard deviations of the contact angle measurements for the five restorative materials tested, treated with different surface procedures, are presented in Table 5. The air-polished surface treatment revealed the highest contact angles in all groups except ZrO. Furthermore, results show a clear correlation between surface roughness values and

Table 4 Summary of ANOVA

Group (Roughness)		Sum of squares	df*	Mean square	*F***	Significance
Ra (μm)	Between Groups	27.450	4	6.863	17.203	<0.001***
	Within Groups	121.672	305	0.399		
	Total	149.123	309			
Rz (μm)	Between Groups	690.723	4	172.681	18.290	<0.001***
	Within Groups	2879.603	305	9.441		
	Total	3570.326	309			
Sa (μm)	Between Groups	60.966	4	15.242	4.576	<0.001***
	Within Groups	1015.836	305	3.331		
	Total	1076.803	309			

* df: degrees of freedom
** *F*: Variance ratio value
*** statistically significant

Table 4a *Post hoc* Bonferroni tests for Ra

Materials	Surface	Surface	Mean difference	Standard error	Significance
all	No treatment	Paper-grinded	−0.204	0.206	1.000
		Stone-grinded	−0.712	0.218	0.013*
		Air-polished	−0.924	0.218	0.000*
		High-polished	−0.228	0.218	1.000
all	Paper-grinded	No treatment	0.204	0.206	1.000
		Stone-grinded	−0.508	0.103	0.000*
		Air-polished	−0.720	0.103	0.000*
		High-polished	−0.024	0.103	1.000
all	Stone-grinded	No treatment	0.712	0.218	0.130
		Paper-grinded	0.508	0.103	0.000*
		Air-polished	−0.212	0.126	0.940
		High-polished	0.483	0.126	0.002*
all	Air-polished	No treatment	0.924	0.218	0.000*
		Paper-grinded	0.720	0.103	0.000*
		Stone-grinded	0.212	0.126	0.940
		High-polished	0.695	0.126	0.000*
all	High-polished	No treatment	0.228	0.218	1.000
		Paper-grinded	0.024	0.103	1.000
		Stone-grinded	−0.483	0.126	0.002*
		Air-polished	−0.695	0.126	0.000*

* statistically significant

contact angle values after treatment with an APD for all materials except ZrO. In contrast, however, other applied surface treatments did not display a generally conclusive association between surface roughness values and contact angle values. Polishing procedures led to a considerable increase in the contact angle values for PEEK-IOF, PMMA-noF and ZrO. Nano-filled PMMA-DMA displays lower contact angle values than nano-filled DMA-nano due to its considerably lower filler fraction. Polishing resulted in a decrease of contact angle values only for DMA-nano and PMMA-DMA.

The summary of the ANOVA for contact angle measurement is presented in Tables 6 and 7. Statistical analysis of the data with a one-way analysis of variance indicated significant differences in contact angle values between the groups of restoratives ($p<0.001$) and the surface treatments ($p<0.001$).

Table 4b *Post hoc* Bonferroni tests for Rz

Materials	Surface	Surface	Mean difference	Standard error	Significance
all	No treatment	Paper-grinded	−1.156	1.003	1.000
		Stone-grinded	−3.694	1.064	0.006*
		Air-polished	−4.738	1.064	0.000*
		High-polished	−1.230	1.064	1.000
all	Paper-grinded	No treatment	1.156	1.003	1.000
		Stone-grinded	−2.538	0.501	0.000*
		Air-polished	−3.582	0.501	0.000*
		High-polished	−0.074	0.501	1.000
all	Stone-grinded	No treatment	3.694	1.064	0.006*
		Paper-grinded	2.538	0.501	0.000*
		Air-polished	−1.044	0.614	0.902
		High-polished	2.463	0.614	0.001*
all	Air-polished	No treatment	4.738	1.064	0.000*
		Paper-grinded	3.582	0.501	0.000*
		Stone-grinded	1.044	0.614	0.902
		High-polished	3.507	0.614	0.000*
all	High-polished	No treatment	1.230	1.064	1.000
		Paper-grinded	0.074	0.501	1.000
		Stone-grinded	−2.463	0.614	0.001*
		Air-polished	−3.507	0.614	0.000*

* statistically significant

Table 4c *Post hoc* Bonferroni tests for Sa

Materials	Surface	Surface	Mean difference	Standard error	Significance
all	No treatment	Paper-grinded	−1.079	0.596	0.711
		Stone-grinded	−1.648	0.632	0.096
		Air-polished	−1.827	0.632	0.041*
		High-polished	−0.657	0.632	1.000
all	Paper-grinded	No treatment	1.079	0.596	0.711
		Stone-grinded	0.568	0.298	0.573
		Air-polished	0.747	0.298	0.126
		High-polished	0.421	0.298	1.000
all	Stone-grinded	No treatment	1.648	0.632	0.096
		Paper-grinded	0.568	0.298	0.573
		Air-polished	−0.179	0.364	1.000
		High-polished	0.990	0.364	0.070
all	Air-polished	No treatment	1.827	0.632	0.041*
		Paper-grinded	0.747	0.298	0.126
		Stone-grinded	0.179	0.364	1.000
		High-polished	1.169	0.364	0.015*
all	High-polished	No treatment	0.657	0.632	1.000
		Paper-grinded	−0.421	0.298	1.000
		Stone-grinded	−0.990	0.364	0.070
		Air-polished	−1.169	0.364	0.015*

* statistically significant

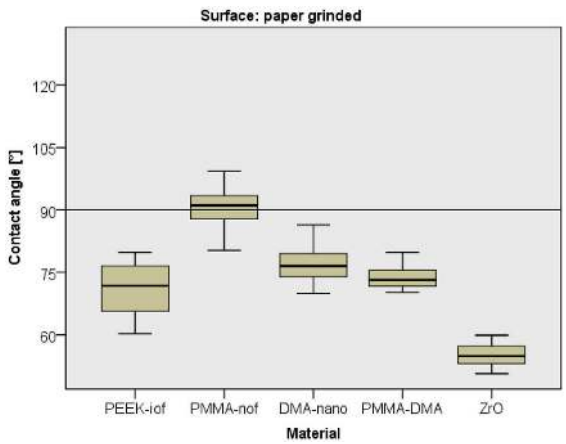

Fig. 25 Contact angles of the five tested materials with paper-grinded surface.

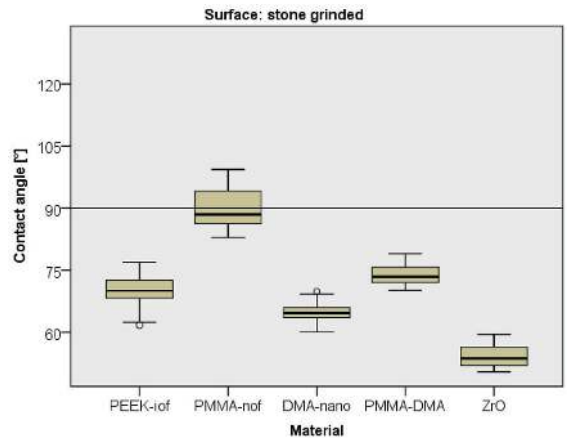

Fig. 26 Contact angles of the five tested materials (medians and 25/75th percentiles) with stone-grinded surface.

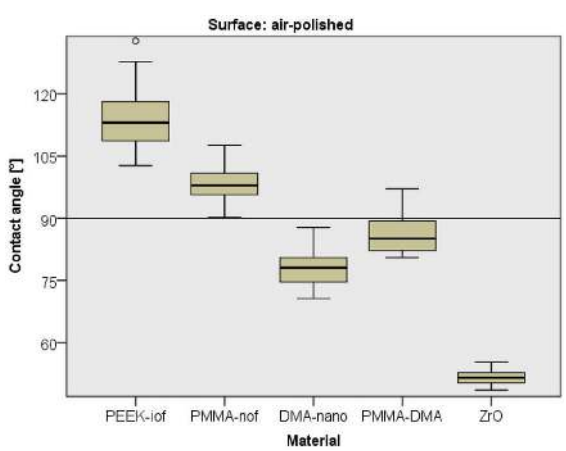

Fig. 27 Contact angles of the five tested materials (medians and 25/75th percentiles) with air-polished surface.

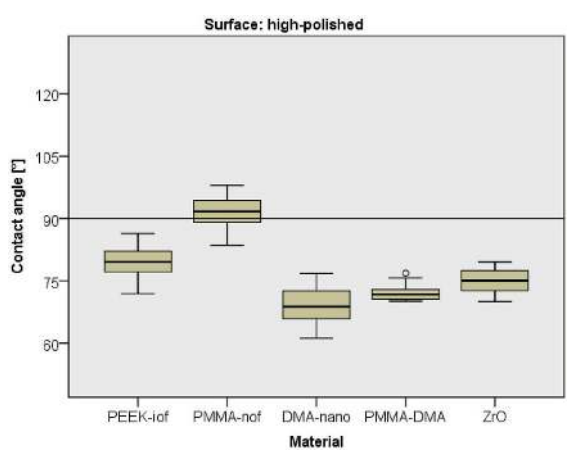

Fig. 28 Contact angles of the five tested materials (medians and 25/75th percentiles) with high polished surface.

Table 5 Contact angle values (°), mean ±standard deviations

Material	Surface treatment	Mean	SD
PEEK-IOF	Paper-grinded	70.8	5.85
	Stone-grinded	70.2	3.35
	Air-polished	114.0	6.46
	High-polished	79.4	3.57
PMMA-noF	Paper-grinded	90.7	4.29
	Stone-grinded	90.0	4.90
	Air-polished	98.6	3.91
	High-polished	91.5	3.46
DMA-nano	Paper-grinded	76.9	4.01
	Stone-grinded	65.0	2.16
	Air-polished	77.9	4.10
	High-polished	69.1	4.13

Table 5 continued

Material	Surface treatment	Mean	SD
PMMA-DMA	Paper-grinded	73.8	2.65
	Stone-grinded	73.9	2.47
	Air-polished	86.3	4.96
	High-polished	71.9	1.55
ZrO	Paper-grinded	55.0	2.70
	Stone-grinded	54.2	2.45
	Air-polished	51.6	1.61
	High-polished	75.0	2.63
ZrO reference		94.2	1.18

Table 6 Summary of ANOVA mean contact angles

Group (surface treatment)		Sum of squares	df*	Mean square	*F***	Significance
1	Between groups	39,321.631	4	9,830.408	590.370	<0.001***
	Within groups	4,912.121	295	16.651		
	Total	44,233.752	299			
2	Between groups	41,393.544	4	10,348.386	991.979	<0.001***
	Within groups	3,077.460	295	10.432		
	Total	44,471.004	299			
3	Between groups	131,648.269	4	32,912.067	1,625.944	<0.001***
	Within groups	5,971.336	295	20.242		
	Total	137,619.605	299			
4	Between groups	21,155.056	5	4,231.011	423.270	<0.001***
	Within groups	3,038.784	304	9.996		
	Total	24,193.840	309			

* df: degrees of freedom
** *F*: Variance ratio value
*** statistically significant

Table 7 Summary of ANOVA mean contact angles

Group (material)		Sum of squares	df*	Mean square	*F***	Significance
PEEK-IOF	Between groups	77,210.926	3	25,736.975	1,027.300	<0.001***
	Within groups	5,912.516	236	25.053		
	Total	83,123.442	239			
PMMA-noF	Between groups	2,888.147	3	962.716	55.186	<0.001***
	Within groups	4,117.014	236	17.445		
	Total	7,005.161	239			
DMA-nano	Between groups	7,042.344	3	2,347.448	171.293	<0.001***
	Within groups	3,234.215	236	13.704		
	Total	10,276.560	239			
PMMA-DMA	Between groups	7,946.808	3	2,648.936	263.667	<0.001***
	Within groups	2,370.979	236	10.047		
	Total	10,317.787	239			
ZrO	Between groups	20,960.043	3	6,986.681	1,219.340	<0.001***
	Within groups	1,352.253	236	5.730		
	Total	22,312.296	239			

* df: degrees of freedom
** *F*: Variance ratio value
*** statistically significant

DISCUSSION

Dental restorative materials are regarded as artificial predilection sites for the adherence and accumulation of oral microorganisms[53]. Various studies have been carried out with a focus on bacterial adherence on materials used in conservative dentistry[1,32,52], but few studies have been conducted on resin restorative materials used in prosthodontics[25,40]. Thus, this study was carried out to assess chair-side surface treatment methods of prosthodontically used resin restorative materials by relating possible differences to surface roughness, hydrophobicity and type of matrix.

In the present study, differences in contact angle values and surface roughness values after specific surface treatment did not follow a clear pattern, suggesting that the chemistry of the material itself with respect to differences in matrix composition and filler fraction plays a decisive role. These results are supported by previous studies showing that surface roughness as well as composition of the resin composites (filler size and matrix monomer) influence biofilm formation[3,24,45,47].

Filled resins (composites) and particularly DMA (organic composites) display several features. The fillers are functionalized differently, either silanzied or without functionalization as in thermoplasts, partly also mixed with additives[19]. Those additives are supposed to prevent the agglomeration in the resin during the manufacturing and subsequent processing. This implicates that the mere presence of ceramic or anorganic fillers (displayed by the filler fraction) on the surface is not unequivocally and does therefore not necessarily lead to a conclusive prognosis of the materials hydrophilic or hydrophobic properties. The reason for this is that abrasive surface or compacted surface treatment methods also expose the components differently, respectively only lead to changes in surface structure. Therefore the investigations of different surface treatment methods under lab-technical and clinical conditions are useful. PEEK-IOF is a filled thermoplast and hence likewise inhomogenous in regard to the properties[59]. Pure cold-curing resins, being low molecular, not filled and cross-linked, display a rather consistent contact angle, but do react with significant changes of surface roughness. PMMA is known to be hardly susceptible to plaque which confirms these theories. PMMA-DMA composites, being low filled (<10% nanoscale), higher molecular and more net-worked with DMA, displays a significantly better surface stability and provides relatively constant contact angles (<90 / >75). DMA-nano, having a 50% filler fraction (0.05 μm) provides the most constant results with respect to the contact angles, despite different surface roughness. PEEK as a thermoplast with a filler fraction of <30% and a 0.5 μm medium particle size responds noticeably inhomogenous to the surface treatment with respect to the contact angles, however not as pronounced as PMMA responds to the surface roughness. Changes in contact angles could therefore either result from the polymer (aromatic structure) and/or result from the inhomogenous surface which is caused by the fillers.

Moreover, it has been demonstrated that the physicochemical characteristics of the specific substratum affect the quality of the bacterial adhesion to the surface of the material, but the influence of surface roughness and hydrophobicity is considered dominant in this regard[24]. In contrast, other studies found no relationship between surface roughness values and bacterial adhesion. Those studies attribute firm bacterial adhesion to filler particles of the composite resin surfaces but also to electrical interactions between bacteria and the material surfaces[46,50].

Noting that some materials are more plaque-prone than others, Nassar *et al.* clearly emphasize that the degree of hydrophobicity alone is not a useful discriminant for dental plaque build-up. Their findings indicate that plaque adherence to prosthetic materials depends more directly on the value of the surface free energy of the material than on its relative wettability by water solutions[25]. Within the limitations of the present study, contact angle measurements could therefore only serve as an indication of surface free energy changes. According to Glantz, acrylic resin has a low specific surface energy[58]. However, more plaque was found to adhere in clinical studies. This could be explained by the acrylic resins' highly porous nature, causing liquid absorption and enabling adhesive forces to rise[39]. In consequence, a greater amount of plaque adheres to this surface than could possibly result from the low values of surface free energy alone[39]. Moreover, smooth low-energy surfaces have shown to attract less plaque than smooth intermediate-energy surfaces[30]. In addition, several studies have been conducted concerning the effect of surface hydrophobicity on bacterial adhesion, supplying evidence that hydrophobic bacteria adhere much more readily to hydrophobic supports, while hydrophilic bacteria display less adhesion to hydrophobic supports[32,40,42]. As hydrophobicity can be used as a discriminant to predict bacterial adhesion[41,43], the results of the present study have shown that selected chair-side surface treatments do have a significant ($p<0.001$) impact on substratum hydrophobicity values. Changes in hydrophobic properties can thus be explained by the physicochemical composition of the material.

Furthermore, Bollen *et al.* clearly state that preferential bacterial retention occurs on rough surfaces since bacteria on such surfaces are better protected against shear forces[20]. These results are confirmed by other investigators who state that increasing surface roughness not only increases plaque retention[23,24,28,36,44] but also leads to plaque adhering much more rapidly and in larger quantities[20,25]. Comparing all influencing factors, several studies concluded that surface roughness plays the most important role in initial bacterial adhesion and the influence of surface roughness exceeds the influence of surface free energy[23,29,30,34,37]. Carlen *et al.* state that polishing procedures lead to an unfavorable surface with lower protein resistance that is thus more prone to accumulate biofilms. They point out that polishing leads to an increased polar and notably basic contribution to the total surface free energy for the

composite resin, whereas unpolished composite resin is weakly basic with a small polar (acid-base) contribution to the total surface free energy[22].

Other investigators state that polished composite surfaces supported larger amounts of plaque due to the loss of superficial filler particles during the polishing process[4,25,50,52]. These observations contradict those of other authors who found that surface polishing of composite materials rendered the materials more plaque resistant and that the procedure was important to reduce bacterial accumulation[8,37,45]. The use of air-polishing devices in oral hygiene and periodontal therapy has proven to be a very effective means in rapidly removing stains and dental plaque[10]. However, numerous studies have shown that air-polishing instrumentation can potentially remove considerable amounts of resinous restorative material[13,14], due to the low wear resistance of the resinous matrix material[16,18,19]. In line with these findings, the present study found significant increases ($p<0.001$) in surface roughness values (Ra) after APD surface treatment for all tested materials (except ZrO). An accumulation of agglomerated bicarbonate particles of the jet stream has been found within the filler-matrix interface after air-polishing instrumentation[15]. It could therefore be assumed that embedded bicarbonate particles may also affect the material surface texture to the extent that the physicochemical surface properties of the material are being masked.

In the present study, PMMA-noF exhibited by far the greatest increase in surface roughness values (Ra) after APD treatment. Its relatively low wear-resistance and agglomeration of bicarbonate particles could account for these results. Furthermore, other studies observed staining and pitting on porcelain surfaces[15,16]. The results of the present study confirm earlier work indicating that the use of an air-polishing device on resinous restorative materials leads to a significant increase in surface roughness[12,14,15]. Treated surfaces may subsequently become more plaque-retentive, and the use of an APD around the tested restorations should therefore be avoided.

As adhering bacteria play a crucial role in the development of infectious diseases, a low susceptibility of restorative materials to microorganism adherence is of major interest[2].

According to our results, the hypothesis that the use of different surface treatment methods does have a significant impact on the properties of the materials that were tested could not be rejected. For clinical use different dental resin materials for customized veneering and coating have shown to be indispensable.

The findings of the present investigation indicate that supplementary studies on adsorption patterns and the distribution of adhering bacterial strains by means of fluorescence imaging are necessary to permit drawing conclusions about substratum surface properties and bacterial adsorption.

CONCLUSION

Within the limitations of this study, significant differences were found between the chair-side surface treatment procedures applied on dental restoratives used in prosthetic dentistry. However, no general correlation could be found between changes in surface hydrophobicity and surface roughness values. Air-polishing treatment and polishing procedures with Zirpolish paste resulted in higher surface roughness for all materials except zirconium dioxide. This treatment may facilitate microbial retention and infection. Polyether ether ketone (PEEK-IOF) displayed the greatest change (increase) in contact angle values after air-polishing treatment. However, this effect can be prevented by veneering PEEK-IOF with DMA-nano components. Nevertheless, the use of an air-polishing device around these materials should be considered carefully.

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