

Surface roughness evaluation of different novel CAD/CAM dental materials

Stefan VULOVIĆ ^{*}, Ivica STANČIĆ, Aleksandra POPOVAC ^{*}, Aleksandra MILIĆ-LEMIĆ

University of Belgrade, School of Dental Medicine, Belgrade, Serbia

*Corresponding author: stefan.vulovic@stomf.bg.ac.rs

Keywords

surface roughness
profilometer
SEM
AFM
composite
zirconia
CoCr

Abstract

The aim of the study was to evaluate and compare surface roughness among different CAD/CAM dental materials when all materials are finished and polished according to the manufacturer's instructions. The 45 specimens were split into three groups: RBC (resin-based composite), ZR (zirconia) and CoCr₄ (CoCr₄ alloy). The surface roughness was evaluated using a mechanical profilometer, atomic force microscopy (AFM) and scanning electron microscopy (SEM) analyses. The data were analysed using the Kruskal-Wallis test with a Dunn's post hoc analysis and all data were presented as mean ± SD. Surface roughness analysis on a microscale (µm) revealed significantly higher *Ra* (µm) mean values in the ZR group compared to RBC and ($p < 0.05$) and CoCr₄ group ($p < 0.05$), between which no statistically significant difference was observed ($p > 0.05$). Surface roughness analysis on a nanoscale (nm) has shown the opposite results, with the lowest values in the ZR group and statistically significant differences among all material groups ($p < 0.05$). AFM 3D and SEM 2D images revealed heterogeneous surface morphology for all groups.

History

Received: 07-06-2022
Revised: 24-06-2022
Accepted: 24-06-2022

1. Introduction

Surface roughness can be considered the most important surface characteristic of any dental restoration. Numerous researches based on commercially available dental materials confirmed that this surface parameter plays a crucial role when it comes to the question of the other material characteristics in the oral environment, such as microbial adhesion on its surface, corrosion behaviour and biocompatibility [1-5].

However, the most studied and described was the correlation between surface roughness and microbial adhesion. As it is known from the literature, surface irregularities provide desirable growth conditions for microorganisms by giving them shelter and protection against shear forces in the oral cavity [6]. Furthermore, in surface defects, such as pits and grooves, microorganisms establish a strong attachment to the material's surface,

which represents the initial, most important phase in microbial colonisation on the surface of dental restoration [7]. As a general rule, 0.2 µm represents the threshold surface roughness value, below which no impact of the surface roughness on microbial colonisation could be expected [8]. It is easy to conclude that creating a smooth and glossy surface through an adequate finishing and polishing procedure is very important when fabricating dental restorations [9-11].

The introduction of computer-aided design and computer-aided manufacturing (CAD/CAM) systems in dental medicine significantly enhanced the mechanical and esthetic properties of dental materials, due to a standardised manufacturing process, good reproducibility and reduced production cost [12,13].

Over the past few years, numerous novel CAD/CAM dental materials appeared on the market. One of the most popular are resin-based composites, zirconia and cobalt-chromium alloy. The mechanical and esthetic properties of composite materials have been improved over the years, still

considering these materials as one of the most desirable solution in dental medicine [14]. However, the major drawback, the polymerisation shrinkage [15] and the resultant internal stresses that might lead to microcracks formation and bacterial colonisation [16], created a need for modulation of composites properties [17] by the introduction of micro- and nanotechnology in dental materials field [18]. Zirconia has shown increased popularity in the past decade, mainly due to excellent mechanical properties, high resistance to wear [19,20], resistance to corrosion, good biocompatibility and excellent esthetic and mechanical properties [21-23]. However, several authors [24,25] have expressed concern about the long-term presence of zirconia in the oral environment, pointing out some of the disadvantages such as the wear of opposing surfaces and almost impossible reparable process after breakage (ceramic chipping), which encouraged dentists to look for another options [26]. The development of cobalt-chromium alloy in the 80's has given the opportunity for replacing high-priced gold restorations [27]. This base-metal alloy was many times described as the extremely recommended solution in modern dentistry, due to its high strength, heat resistance, resistance to wear and duration over time [28].

Despite previously described advantages, neither of the CAD/CAM dental materials reveals complete smooth surface morphology, without any irregularities. Therefore, the aim of the study was to evaluate and compare surface roughness among different CAD/CAM dental materials, when all materials are finished and polished according to the manufacturer's instructions.

The following null hypotheses were: 1. No statistically significant difference would be found between surface roughness on a microscale among different CAD/CAM dental materials. 2. No statistically significant difference would be found between surface roughness on a nanoscale among different CAD/CAM dental materials.

2. Materials and methods

2.1 Specimen preparation

Three different CAD/CAM dental materials, with the description given in Table 1, have been included in the research. A total number of 45 cylinder-shaped specimens, with 10 mm diameter and 2 mm height, were split into three groups: RBC (resin-based composite), ZR (zirconia) and CoCr₄ (CoCr₄ alloy).

Table 1. Materials used in the study

Material	Composition
Resin-based composite	27 wt. % inorganic fillers in a polymer matrix
Zirconia	88 – 95.5 wt. % ZrO ₂ 4.5 – 7 wt. % Y ₂ O ₃ < 5 wt. % HfO ₂ < 1 wt. % Al ₂ O ₃ < 1.5 wt. % other oxides
CoCr ₄ alloy	63 wt. % Co 29 wt. % Cr 6 wt. % Mo < 2 wt. % Fe, Mn, Si, Nb

The diamond blade (15LC, Buehler) placed in a cutting machine (Isomet 4000, Linear Precision Saw, Buehler) was used for cutting the RBC disc (Structur CAD, VOCO), with a diameter of 98.4 mm and thickness of 20 mm and zirconia disc (Ivotion IPS e.max ZirCAD Prime, Ivoclar Vivadent), with a diameter of 98.5 mm and thickness of 20 mm. For cutting CoCr₄ alloy disc (Colado CAD CoCr₄, Ivoclar Vivadent), with a diameter of 98.5 mm and thickness of 12 mm, the laser cutting machine (Water Jet 5X 3080, Knuth Machine Tools) was used. The cutting process for all materials was performed at slow speed and under constant water cooling.

Cut RBC and CoCr₄ specimens were rubber-finished and polished, following the manufacturer's instructions. After the sintering process in the furnace (Standard program for IPS e.max ZirCAD Prime, Programat S1/S1 1600, Ivoclar Vivadent) cut ZR specimens received a thin glaze layer (IPS Ivocolor Glaze Paste FLUO, Ivoclar Vivadent) to both sides of the specimen, followed by crystallisation in a furnace (Stains and Glaze firing program, Programat P142, Ivoclar Vivadent).

A total of 45 specimens, 15 specimens per group (n = 15) were obtained. Their dimensions were checked on a digital calliper (Lukas Tools Digital Caliper 300mm, Vogel).

2.2 Mechanical profilometer analysis

The surface roughness on a microscale (µm) was analysed on all 45 specimens, 15 per group (n = 15) using a mechanical profilometer (Surface Roughness Tester TR200, Beijing TIME High Technology) under the following test conditions: total length to 1.25 mm, cut off value set to 0.25 mm, resolution to 0.02 µm, with Gaussian filter. Three measurements per specimen were executed,

analysing the mean value of all peaks and valleys in the given profile (Ra). For each specimen average value was calculated, and the mean value for the group was obtained.

2.3 Atomic force microscopy (AFM) analysis

Atomic force microscope (Veeco Bruker DI Dimension Hybrid XYZ Scanning Probe Microscope Head/AFM, Veeco Instruments) was used for presenting the surface morphology in three-dimensional space (3D) and analysing surface roughness on a nanoscale (nm). One randomly selected field of one specimen per each group ($n = 1$) was observed under ambient conditions in air and using the tapping mode, which is the opposite of contact mode [29].

The images of a covered field of $10 \times 10 \mu\text{m}$ were recorded at the resolution of 512×512 pixels with the 1 Hz scanning rate and analysed using systemic software (Nanoscope analysis 1.40, Bruker).

Surface roughness measurements on a nanoscale were performed on three randomly selected spots. For each specimen average value was calculated, and the mean value for the group was obtained.

2.4 Scanning electron microscopy (SEM) analysis

In order to present the surface morphology of each material in two-dimensional space (2D), scanning electron microscope (JEOL JSM-6610LV, Jeol) has been used. The scanning process was performed on one randomly selected field of one specimen per group ($n = 1$).

Before the SEM procedure, the specimens were sputter-coated with a 20 nm layer of gold for 2 min in order to prevent electrical charge build-up within a specimen and provide more quality images without forming the artifacts. The prepared specimens were then examined under the following parameters: voltage 20 kV, tilt angles 10° to 45° and $1000\times$ magnification.

2.5 Statistical analysis

The data of mechanical profilometer Ra (μm) and AFM Ra (nm) measurements were firstly tested for normal distribution using the Kolmogorov-Smirnov test with a $\alpha = 0.05$ level of statistical significance and statistical power of 80 % (SPSS v22.0, SPSS). Data for both parameters were not normally distributed which led to comparison among groups using the Kruskal-Wallis test with a Dunn's post hoc analysis. Descriptive statistics

were used to describe all statistical units, and all data were presented as mean \pm SD.

3. Results and discussion

3.1 Mechanical profilometer analysis results

The results from the mechanical profilometer analysis are presented in Table 2.

Table 2. Results of mechanical profilometer and AFM surface roughness measurements*

Specimen group	Profilometer Ra , μm (mean \pm SD)	AFM Ra , nm (mean \pm SD)
RBC	0.31 ± 0.06^b	70.35 ± 9.38^b
ZR	0.72 ± 0.12^a	21.60 ± 5.86^c
CoCr ₄	0.34 ± 0.05^b	89.89 ± 6.86^a

*different superscript letters indicate statistically significant difference inside the respective column $p < 0.05$.

ZR specimens have shown statistically significant higher Ra (μm) mean values, compared to RBC and CoCr₄ group ($p < 0.05$). Between RBC and CoCr₄ groups, no statistically significant difference was observed ($p > 0.05$), with slightly higher Ra (μm) mean values in the CoCr₄ group, compared to the RBC group. Figure 1 presents the diagram curves of representative specimens from each group, supporting the numeric results. Compared to RBC and CoCr₄, the ZR specimen (Fig. 1a') revealed the most irregular curve, rich with high peaks and deep valleys. The RBC (Fig. 1a) and CoCr₄ specimen (Fig. 1a'') displayed a more regular curve, with sharp transitions between short peaks and shallow valleys.

3.2 Atomic force microscopy (AFM) analysis results

AFM scanned areas of representative specimens of each group are displayed in Figure 1. RBC specimen (Fig. 1b) revealed similar-sized sharp peaks and surrounding valleys all over the scanned field, with an absence of grinding grooves. CoCr₄ specimen surface (Fig. 1b'') could be described with mixed surface texture, rich with a few different-shaped granular parts and a relatively flat surrounding surface. However, on the left and the right side of imaged surface can be seen a large grinding groove. The smoothest morphology revealed the ZR specimen (Fig. 1b'), which surface was almost completely homogenous, with a couple of pointy different-sized formations.

Surface roughness measured with an AFM device on a nanoscale (nm) (Table 2) revealed statistically significant differences between all groups of specimens ($p < 0.05$). Contrary to profilometer measurements, ZR specimen revealed the lowest Ra (nm) mean value, compared to RBC ($p < 0.05$) and CoCr₄ ($p < 0.05$). The CoCr₄ specimen displayed a higher Ra (nm) mean value, compared to the RBC group ($p < 0.05$), similar to the profilometer measurements on a larger scale (μm).

3.3 Scanning electron microscopy (SEM) analysis results

2D SEM micrographs (Fig. 1) supported the previously described 3D AFM images, revealing clear differences in surface morphology among different groups. ZR specimen (Fig. 1c') demonstrated relatively regular surface morphology, with a few round islands, previously described on AFM image as elevated peaks. Surface morphology defects, such as grooves, scratches and cracks, caused by the finishing and polishing procedure were clearly present on the CoCr₄ micrograph (Fig. 1c''), which specimen revealed the

least homogenous surface morphology, compared to other groups. RBC 2D micrograph (Fig. 1c) on the same magnification did not reveal peaks and valleys, as the AFM 3D image did, pointing out the small dimensions of these formations.

3.4 Discussion

Mechanical profilometer measurements revealed that ZR specimens were significantly rougher compared to rubber-finished and polished RBC and CoCr₄ groups. Therefore, the first null hypothesis is rejected. The fact that during the glazing procedure technician manually apply the glaze layer with the brush onto the material's surface could be the best explanation for these results [30]. In addition, the glazed surface is rich with bubbles, which leads to the impossibility to create a surface as smooth as when finishing the material's surface using burs and rubbers [22]. Profilometer analysis results have shown that RBC specimens have the smoothest surface morphology, which is in accordance with numerous previous investigations, where this material is considered as the one with a relatively smooth surface [4].

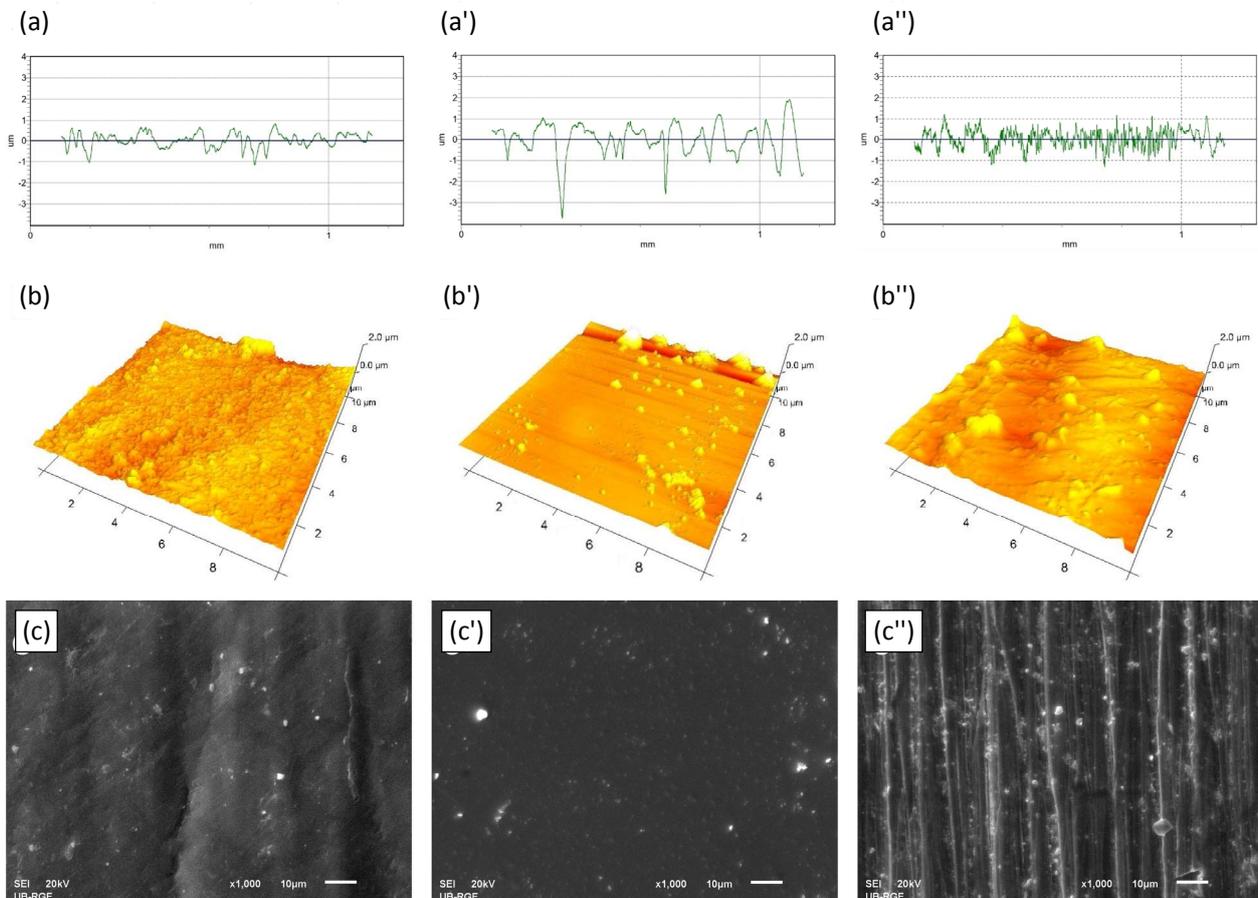


Figure 1. Profilometer analysis – diagram curves from representative specimens of each group: (a) RBC, (a') ZR and (a'') CoCr₄; AFM 3D images from representative specimens of each group: (b) RBC, (b') ZR and (b'') CoCr₄; SEM 2D micrographs from representative specimens of each group: (c) RBC, (c') ZR and (c'') CoCr₄

AFM and SEM analyses revealed the opposite results. RBC and CoCr₄ images corroborate the profilometer analysis results, describing CoCr₄ with a more heterogeneous surface compared to RBC. In addition, the CoCr₄ specimen presented the most evident grinding grooves on the SEM micrograph, which could be related to the fact that CoCr₄ specimens were finished in one direction, contrary to the RBC specimen, where circular movements were used, following the manufacturer's instructions. However, AFM images described ZR with the most homogeneous surface texture, compared to other groups. The reason for disagreement between mechanical profilometer and AFM analysis could be the difference in the scanning field. The covered area on the 3D AFM image was only 10 × 10 μm, which was significantly smaller than the total evaluation length of 1.25 mm using a profilometer. Therefore it is possible that the AFM scanned a flatter part of the surface, giving less reliable data than the profilometer did. As a consequence, AFM surface roughness measurement on a nanoscale (nm) has shown significantly lower Ra mean values in the ZR group, compared to RBC and CoCr₄, between which a statistically significant difference was also observed. Therefore, the second null hypothesis is also rejected.

It is easy to conclude that the used device and the dimensions of the scanned area play a significant role in determining surface roughness. Nevertheless, the surface roughness results from the present study using both devices revealed that mean values for all material groups were above 0.2 μm, which was previously reported as the threshold value, below which the role of surface roughness in plaque adherence on the material surface is eliminated. However, future researches are needed to correlate surface roughness and microbial adhesion on the surfaces of investigated materials.

4. Conclusion

Within the limitations of this current study, it was concluded that there are significant differences in surface roughness on a microscale and nanoscale among different CAD/CAM dental materials.

Measured on a microscale using a mechanical profilometer, zirconia revealed a significantly rougher surface compared to rubber-finished and polished resin-based composite and CoCr₄ alloy.

Measured on a nanoscale using an atomic force microscope, zirconia presented smoother surface topography, compared to resin-based composite and CoCr₄ alloy.

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