IDR International Dental Research

Original Article

Effects of surface treatment methods on the phase changes in zirconia

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Abstract

Aim: In this study, zirconia was subjected to various surface treatments, namely, Al_2O_3 sandblasting, CoJet, and coating; the effects of these methods were investigated with the use of SEM, EDX, and XRD. Coating of zirconia surface with silica using the ultrasonic spray method is performed for the first time in this study. The present results are particularly important for the conduct of future studies.

Methodology: Eighty-four zirconia samples were used for the XRD (n=28), SEM (n=28), and EDX (n=28) analyzes. Commercial semi-sintered discs (Kuraray-Noritake Katana HT10 14 mm, Japan) were used in this study. The size of the zirconia samples (i.e., 6 mm in diameter and 3 mm in height) was designed in a computer environment, factoring in the degree of shrinkage following sintering. The dimensions of the sintered samples were determined with a digital caliper. Four surface treatments were employed in this study.

Results: The surfaces of the zirconium samples subjected to different surface treatments were investigated. The SEM and EDX images of the treated zirconia were taken at 80×, 250×, 1000×, 10,000×, and 20,000× magnifications and then compared with those of the untreated sintered samples. The XRD analysis results showed that the intensity of the peaks of the zirconium oxide samples at the 2θ range of 20°-40° varied from the tetragonal phase to the monoclinic phase along the surface.

Conclusion: The sandblasting and CoJet surface treatments caused mechanical changes on the surface of the zirconia samples, and Al_2O_3 particles were detected on the surface, as shown by the SEM and EDX analysis results. The XRD analysis results for the blasting group differed the most from the results for the control group; by contrast, the results for the ultrasonic spray group were nearly the same to those for the control group.

Keywords: zirconia, surface treatments, phase changes

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Received: 10 February 2021
Accepted: 12 May 2021

Access Online

DOI:
10.5577/intdentres.2021.vol11.suppl1.17

How to cite this article: Özdemir G, Seyfioğlu Polat Z. Effects of surface treatment methods on the phase changes in zirconia. Int Dent Res 2021;11(Suppl.1):103-13. https://doi.org/10.5577/intdentres.2021.vol11.suppl1.17

Introduction

Esthetics has been becoming important in the field of dentistry, as in all other aspects of life. With the increasing esthetics-related concerns, the use of restorative materials whose color blends with that of the anterior and posterior regions of a tooth has been becoming widespread. Addressing esthetics-related concerns requires continuous developments in the field of dental materials. As an infrastructure material, zirconia demonstrates high mechanical performance, durability, tensile strength, and chemical and

dimensional stability, as well as reliability, compared with other full ceramic systems (1–4).

Zirconia consists of particles with small diameters, and its crystal structure has three main phases: cubic (C), tetragonal (T), and monoclinic (M). The M phase is seen in pure zirconium at room temperature. This structure is stable up to 1170°C; at higher temperatures, it transforms into the T phase; at temperatures above 2370°C, it further changes into the C phase. During a cooling process, a T–M phase change is observed at 1070°C, and an approximately 3%–4% increase in volume is observed during this change. This expansion causes stress, which in turn creates cracks in pure zirconium and causes its structure to disintegrate as it cools to room temperature (5–7).

Although the compressive forces resulting from transformation hardening contribute to the durability of a material, the increase in volume due to the control of this transformation will lead to severe fractures. Therefore, the T phase of zirconia should be maintained at room temperature (8). For this purpose, metal oxides, such as calcium, magnesium, aluminum, yttrium, and cerium, are added to zirconia to control transformation hardening, maintaining the T phase of zirconia at room temperature (8).

X-ray diffraction spectroscopy (XRD) is an analysis method using X-ray energy that is more powerful than ultraviolet light but weaker than gamma ray. Crystalline materials consist of repetitive three-dimensional structures called cell units (9). When X-ray beams with high energy and low wavelength are sent on a crystalline material, they are deflected in different directions by the electrons of the atoms or ions hit by the beams.

A diffractometer helps in understanding the geometry and size of a crystal structure by determining the angle at which diffraction is highest in a material (10). Diffraction refers to the harmonious fusion of waves, and it can be explained by Bragg's Law. Each atom array in the crystal structure reflects X-rays at various angles that fit Bragg's Law. When the distance between an X-ray wavelength and crystal atoms is known, the angles of the X-rays deflected by the crystal can be determined (11). A diffraction pattern, which is unique to each substance, is used to define substances and to determine the constituents of a mixture. With the use of a diffractometer, it is possible to assess a quality analysis, determine the components, and perform quantity analyses of a sample with unknown content (12).

The XRD analysis method is used to determine the crystalline structure of materials (e.g., ceramic and metals) that are extensively used in dentistry, as well as define the effect of physical and chemical properties on their crystal structure, crystal sizes, and contents of their components. Furthermore, it is used in the quantitative and qualitative assessments of chemical analyses related to material development, in the evaluation of stress formed in a material due to physical impact, and in the development of new materials (12–14).

With the XRD method, the existing phases in the structure of a material can be determined. In such determinations, no damage occurs in a material. A few samples are also sufficient for analysis. Moreover, the XRD method is faster than chemical analysis methods. Diffraction analysis can characterize the compound(s) arising from different atomic configurations as well as the atoms in the structure of a substance. However, in cases where a phase change needs to be measured, a sensitive analysis cannot be achieved with this technique if the amount of the phase-changing particles is considerably small and superficial (9, 14, 15).

This study aimed to subject zirconia to various surface treatment methods, namely, Al_2O_3 sandblasting, CoJet, and coating, and the effects of these methods were investigated using SEM, EDX, and XRD. Coating of zirconia with silica via the ultrasonic spray method is performed for the first time in this study. The results obtained in this study would serve as the foundation for future studies.

Materials and Methods

In this study, the tests were performed in vitro at the Faculty of Dentistry, Department of Prosthodontics, Dicle University Science and Technology Application and Research Center (DÜBTAM) and the private Orhan Aydogan Dental Studio, Multi-Purpose Prosthesis Laboratory.

A total of 84 zirconia samples were analyzed, of which 28 were analyzed using X-ray diffraction (XRD), 28 using scanning electron microscopy (SEM), and 28 using energy dispersive X-ray spectroscopy (EDX) (Fig. 1).

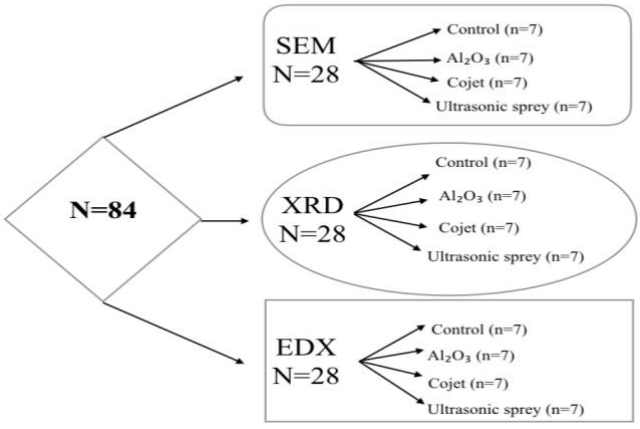

Figure 1. The shemathic view of the experimental groups.

Preparing Zirconium Discs

Semi-sintered discs, Katana Zirconia HT10 14 mm, were obtained from Kuraray Noritake, Japan. The zirconia sample size (diameter: 6 mm; height: 3 mm) required for the experiments was obtained via

numerical simulations by factoring in the calculated amount of shrinkage upon sintering. The semi-sintered discs were placed in a CAD/CAM unit (Yena Makine San. ve Tic. Ltd., Istanbul, Turkey) and were scraped according to the predefined program data. Next, the cylindrical samples were separated from the bonding regions to prepare them for sintering. Finally, the sample sizes were checked using a digital caliper (Alpha Tools, Mannheim, Germany).

Surface Treatment of Zirconium Discs

Once the zirconium discs were sintered, different surface treatments were conducted. The treated samples are divided into four different groups:

1. Control group,
2. Al_2O_3 group,
3. CoJet group,
4. Ultrasonic spray group,

1. Control group

The control group contain 21 zirconia samples that did not receive any surface treatment.

2. Al_2O_3 group

In this group, 21 zirconia samples were sandblasted for 15 s from a distance of 10 mm and at a pressure of 2.8 mm with 110 µm Al_2O_3 sand (Renfert 110, Strahlmittel, Germany) using a sandblasting machine (Renfert Basic eco, Strahlmittel, Germany). An acrylic base containing zirconium disc housings and a plastic top made it possible to sandblast the samples from a distance of 10 mm. All samples in this group were sandblasted individually.

3. CoJet group

In the CoJet group containing 21 samples, silica with a particle size of 30 µm (3M ESPE, Seefeld, Germany) was sandblasted perpendicularly to the sample surfaces for 15 s at a pressure of 2.8 bar and from a distance of 10 mm as recommended by the manufacturer. Subsequently, the recommended silane (Espe-Sil, Seefeld, Germany) was applied to the surfaces, and the samples were placed in lightproof environment.

4. Ultrasonic spray group

A solution of 3% (3-aminopropyl) trimethoxysilane was applied to zirconium disc surfaces (21 samples) using an ultrasonic spray device (Sono-Tek, New York, USA) at 110°C and with a flow rate of 1–5 ml/min. The solution was steadily pulsated at a frequency of 125 kHz, and the distance between the nozzle and the pad was set to 10 cm, followed with the magnification of the interlayer film. The silica-coated zirconia samples were then heat-treated at 450°C for 1 h.

SEM (Quanta FEG 250, USA) was used to evaluate the surfaces of the all the aforementioned samples. The samples were coated with a thin gold film using a gold coating device for 300 s (Mini Plasma Sputtering Coater with Vacuum Pump & Gold Target, Korea) to make the surface conductive and to prevent load accumulation prior to imaging. The samples were fixed on an aluminum block using an adhesive tape before SEM was conducted. Images were taken at magnifications of ×80, ×1000, ×10000, and ×20000.

After the surface treatment, EDX (EDAX, Inc., Mahwah, New Jersey, USA) was employed to determine the types and amounts of elements present on the sample surfaces. In addition, XRD was performed. To analyze the crystal structure of the samples using the Bruker D8 X-ray diffractometer, Germany, with a monochromatic CuKα (1λ=1.54 Å) beam at 40 kV and 40 mA, and from a distance of 250 mm from the sample. The cylindrical samples were placed in the sample holder of the diffractometer to collect data in the scanning angle (2θ) range of 20°–40°.

The values obtained from XRD were recorded, and the density and 2θ values in the regions where density increased were noted for each sample. To calculate the extent of monoclinic and tetragonal phase change (XM) on the surface of the samples, the Garvie and Nicholson equation was used (16).

$$X_M = \frac{I_{M(111^-)} + I_{M(111)}}{I_{M(111^-)} + I_{M(111)} + I_{T(111)}}$$

I: The highest value of the phase density

T (111): The plane showing the (111) crystal geometry of the tetragonal phase

M (111−): The plane showing the (111−) crystal geometry of the monoclinic phase

M (111): The plane showing the (111) crystal geometry of the monoclinic phase (16).

Results

SEM Results of the Surface-Treated Zirconia Structure

In the ×80 magnification images of the control group, the surface was not homogeneous. The rough surface of the sintered zirconia is clear in the ×1000 magnification images. This roughness of the untreated zirconia surface is further clearly evident in the ×10000 and ×20000 magnification images when compared with the treated surfaces (Fig. 2).

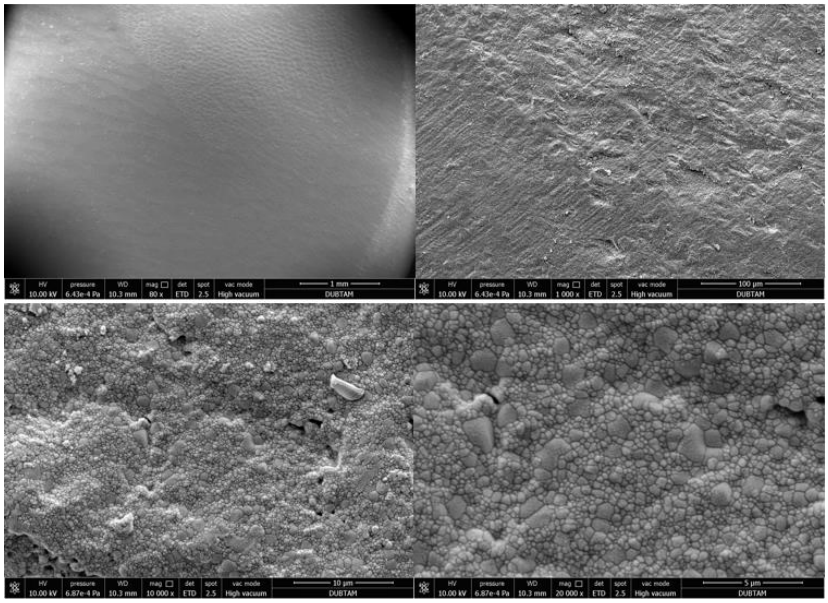

Figure 2. The SEM images of the control group.

In the ×80 magnification images of the samples from the Al_2O_3 group, very small holes are seen in some parts of the surface, which are clear in the ×1000 magnification images. The ×10000 and ×20000 magnification images clarify that the sandblasting completely changed the zircon surface. Compared to the control group, the formation of cracks on the zirconia surface is evident (Fig. 3).

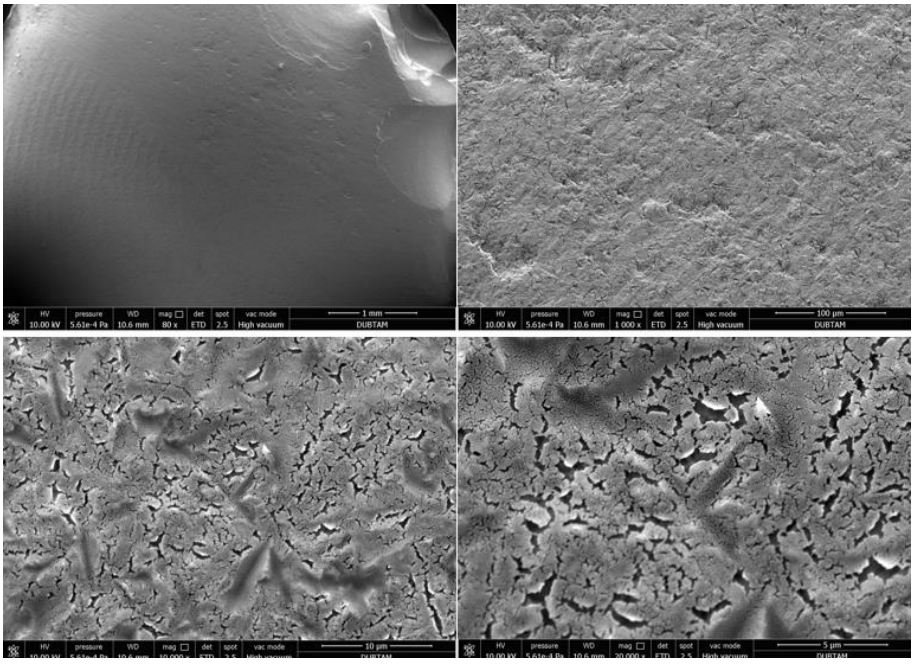

Figure 3. The SEM images of the sandblasted group.

The ×80 magnification images of the samples from the CoJet group are similar to those of the samples from the Al_2O_3 group. However, for the CoJet group samples, small bumps instead of holes are observed in the ×1000 magnification images. In the ×10000 and ×20000 magnification images, the bumps are more obvious, and the observations are different from those of the control and Al_2O_3 groups. The bumpy areas of the surface were the remnants of silica from the silica-coated Al_2O_3 sand, which is used in the CoJet system. The EDX results confirm these findings (Fig. 4).

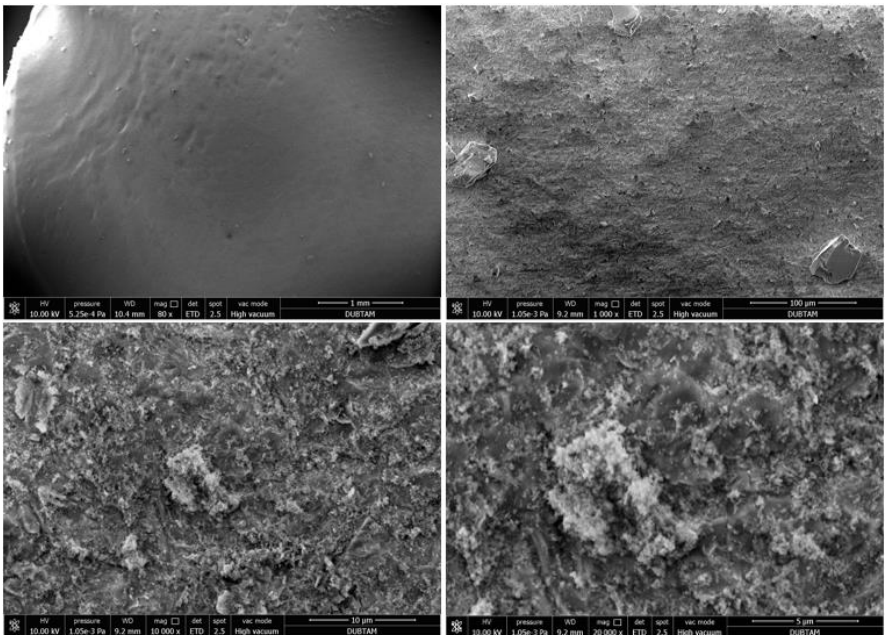

Figure 4. The SEM images of the Cojet group.

No surface changes similar to the ones observed in the aforementioned three groups are noted in the ×80 and ×10000 magnification images of samples from the ultrasonic spray group. In the ×10000 magnification and other images, the cracks on the surface are almost negligible (Fig. 5). However, in the ×20000 magnification images, a cracked region on the surface was selected specifically to make the coated silica layer more visible (Fig. 5). The visible layer was silica, which is confirmed by the EDX results.

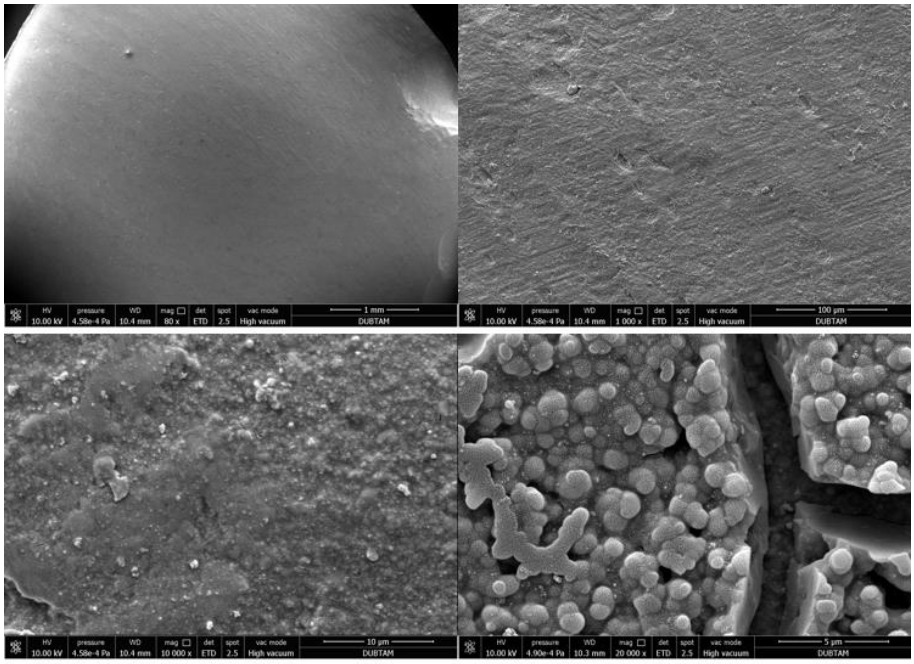

Figure 5. The SEM images of the ultrasonic spray group.

EDX Results of the Surface-Treated Zirconia Structure

EDX was employed to determine the amount of change in the elements before and after surface treatment. The EDX data and graphs of all the groups were prepared.

Atomic O (63.82%), Y (2.56%), and Zr (46.75%) were the main elements detected on the surface of the samples from the control group. The graphical results are shown in Figure 6.

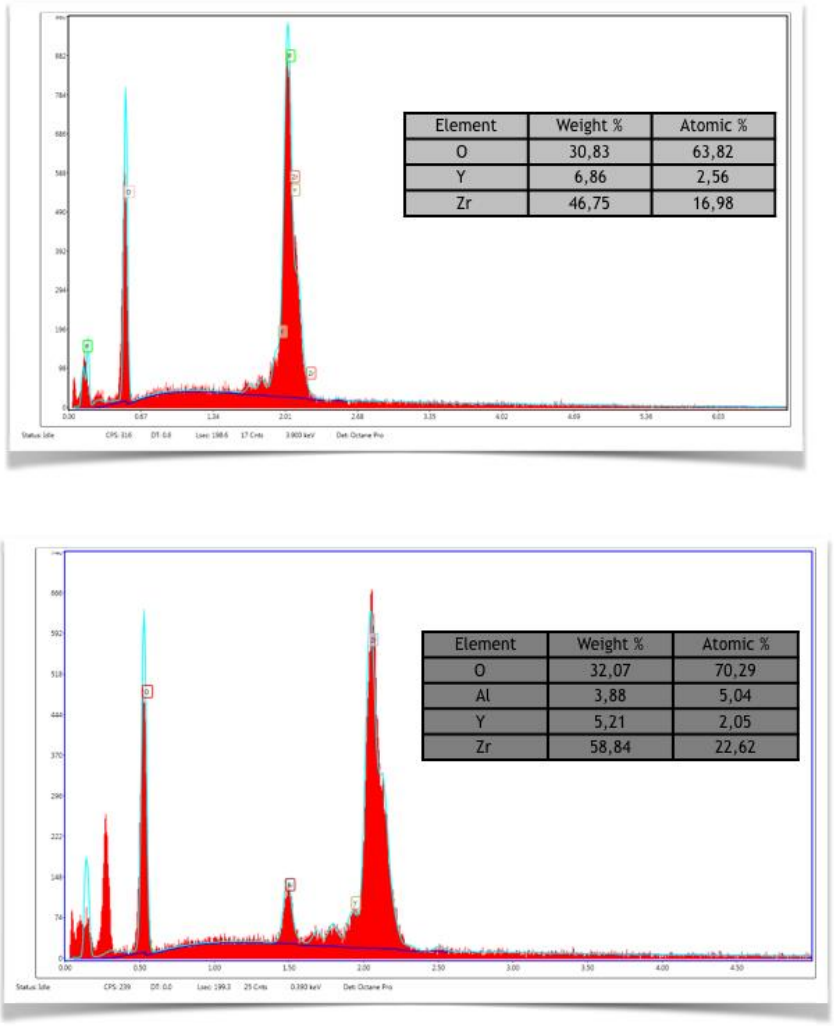

Figure 6. The EDX analysis of the control group and sandblasted group.

For the samples of the Al_2O3 group, considering that Al_2O_3 sand is used for sandblasting, 5.04% atomic Al is noted. The EDX results show that after the surface treatment, some of the Al particles are embedded on the zirconia surface.

Both atomic Al (6.06%) and atomic Si (8.8%) are detected on the zirconia surface in the CoJet group because silica-coated Al_2O_3 was used for surface treatment (Fig. 7).

Atomic Si (20.05%) is detected on the surface of the samples from the ultrasonic spray group. The amount of silica noted on the surface of the samples in this group was 2.5 times higher than that noted in the CoJet group. In addition, no atomic Al was found on the surface because Al_2O_3 sand was not used in the silica coating of the surface in this group.

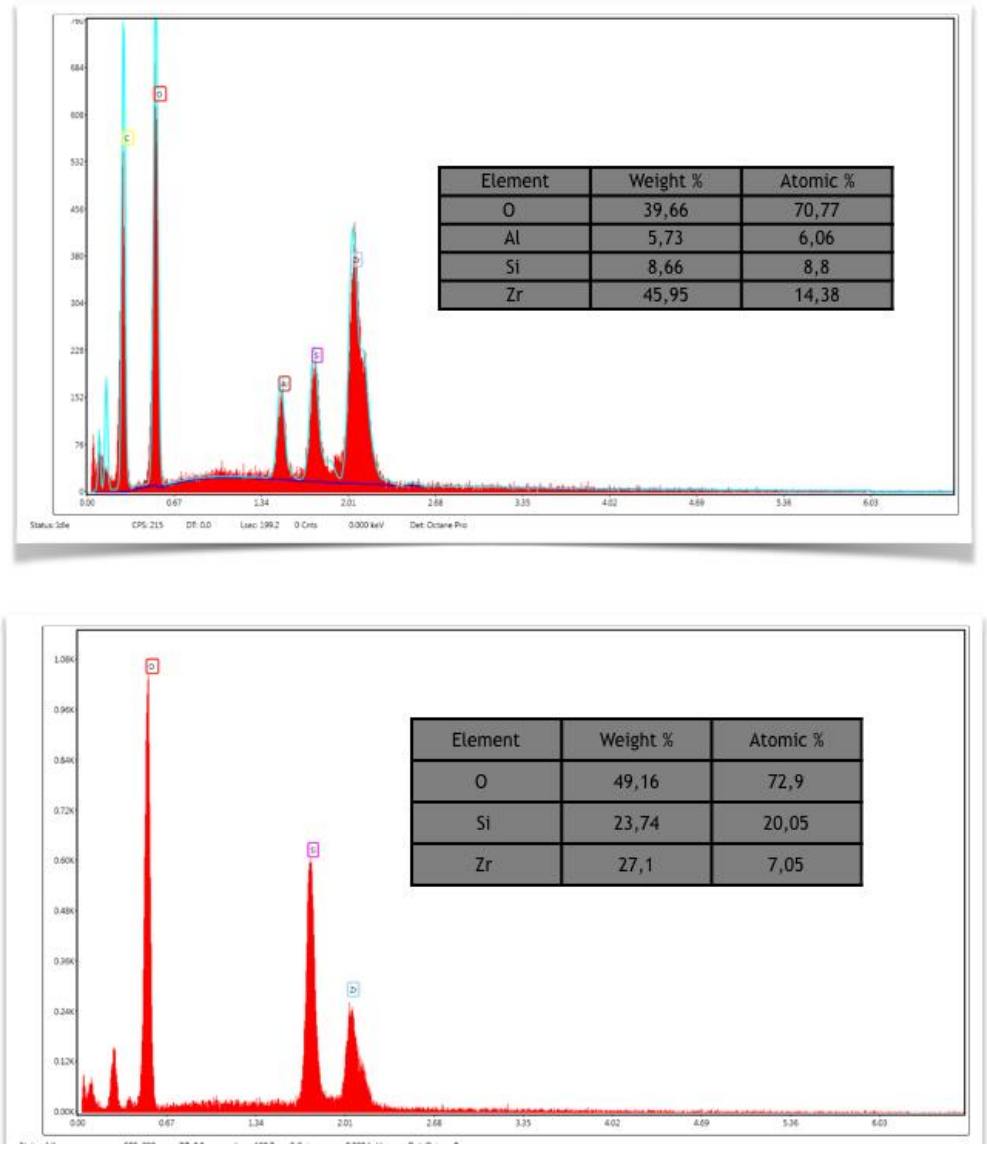

Figure 7. The EDX analysis of the Cojet group and ultrasonic spray group.

XRD Results of the Surface-Treated Zirconia Structure

In the XRD results, the intensity of the peaks of the zirconia samples for 2θ in the range of 20°-40° was noted. The XRD graphs for the samples from all groups are shown in Figure 8. The intensities vary from the tetragonal phase to the monoclinic phase. The extent of the monoclinic and tetragonal phase change (XM) of the treated and untreated zirconia surfaces are compared. Moreover, the XM value is obtained of Garvie and Nicholson equation (1972).

The XRD graphs of the samples from the ultrasonic spray group are the closest to those of the samples from the control group, while the XRD graphs of the Al_2O_3 and CoJet groups are significantly different.

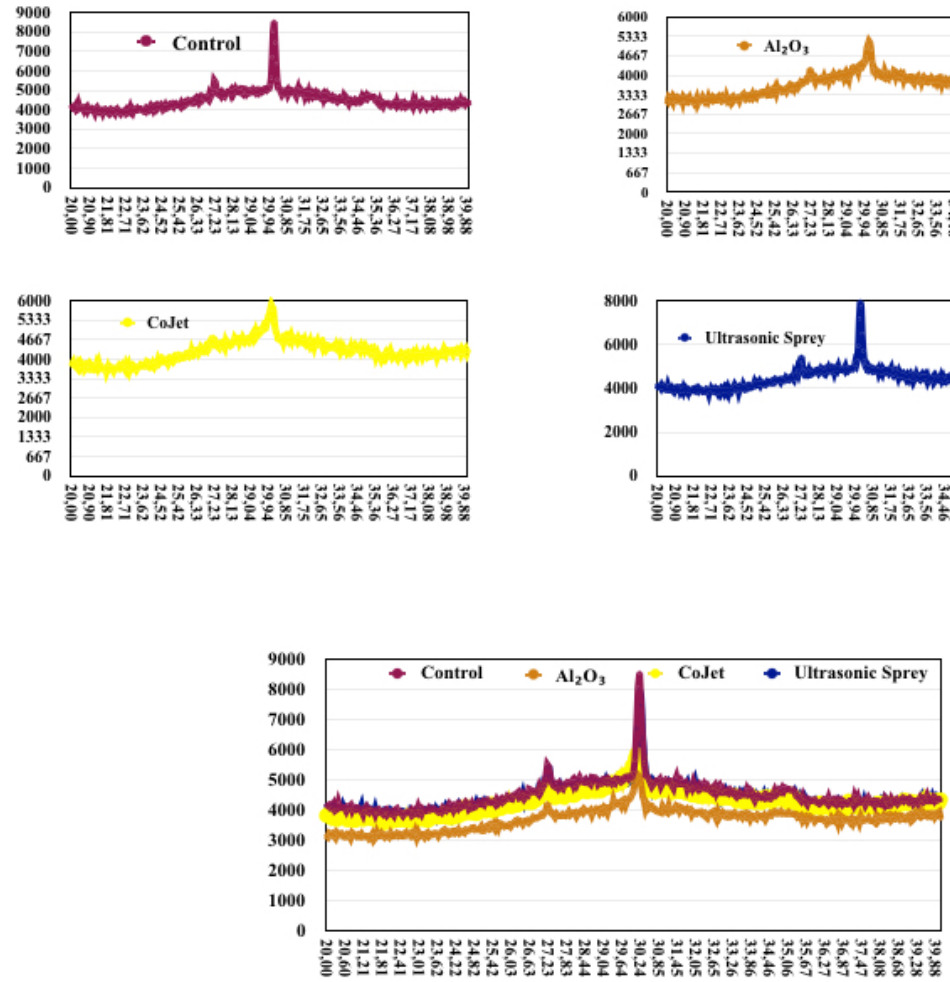

Figure 8. The XRD results of the analysis of control, sandblasted, comet and ultrasonic spray groups.

Discussion

High translucent semi-sintered zirconia block (Katana Zirconia) was used in our study to meet the increasing demand of aesthetics and to create similar translucency with the natural teeth.

Fully sintered zirconia ceramics do not show shrinkage, but in CAD/CAM systems, scraping processes increase the cost and cause time loss due to the hardness of the material and the shortening of usage life of drills. Abrasion of the semi-sintered zirconia is easy and fast compared to the complete sintering. In the studies, it was stated that the forces applied by CAD / CAM drills during the scraping of semi-sintered zirconia will be less sensitive and the scraping process will be shorter. To achieve faster and more precise scraping, semi-sintered zirconia blocks were used in our study in parallel to the suggestions from the previous research (17).

Sandblasting is one of the most frequently used methods to increase the mechanical connection of the ceramic surface with the zirconia infrastructure (18). Some of the researchers investigating the effect of the sandblasting process on the zirconia surface stated that it increased the resistance of the zirconia while others claimed the opposite (6).

Zhang et al. and Aboushelib et al. concluded that the sandblasting process reduced the interface failure rate (19, 20). Yılmaz et al, investigated the fracture strength and phase change of zirconia material with the various surface treatments. They stated that sandblasting caused severe fractures in the applied surface treatments and thus reduced fracture resistance (21).

Sandblasting process on the surface of zirconia leads to the transformation from monoclinic phase to the tetragonal phase. It has been emphasized that not only in the sand size but also the sandblasting device factors such as pressure, sandblasting density, and duration should be determined in the sandblasting process (22).

In the study of Guazzato et al., it is stated that the blasting process greatly increased the flexural strength. The reason suggested for this was that it caused a loss of resistance due to the formation of a layer of pressure on the surface by the conversion of the tetragonal phase into the monoclinic phase which is larger in volume (23).

Papanagiotou et al. applied sand blasting and polishing with 50 µm and 270 µm Al_3O_2 to the zirconia

ceramic surfaces and reported an increase in the durability of the material as a result of surface treatments and the highest bending strength value was in the sandblasted samples with 50 µm Al_2O_3 particles (24).

In some earlier studies, sandblasting with Al_2O_3 having a particle size of 110 µm at 4 bar pressure was applied to the ceramic surface in dry and wet environments before and after abrasion. The results of these studies indicated that the blasting process applied both before and after the abrasion process increased the flexural strength of the material (25, 26). In another study, the effect of different particle sizes on Al_2O_3 particles in dry and wet environments was investigated and the effect of sandblasting on the bending strength was found to be highest with 110 µm Al_2O_3 particles made under water cooling (27).

In a recent study, two different zirconia substructures were subjected to sandblasting with 70 µm Al_2O_3 particles and 125 µm silica carbon powder and the bending strength, the surface roughness, and the relative monoclinical phase changes of the samples were examined. The application time of the sandblasting process is standardized as 10 and 90 seconds. The study concluded that sandblasting with Al_2O_3 further increased the bending strength. As a result, when an external force is applied to the material, as in the sandblasting or abrasion process, a portion of the tetragonal (t) particles are converted into monoclinic (m) particles that are larger in the volume (t → m), (26).

Tribochemical silica coating method gives a specific heat while spraying silica coated particles on the application surface. The heated particles are cooled when they hit the surface, forming a stable and durable glass phase layer on the surface of the ceramic. They then form a chemical connection with the applied silane (28, 29). The CoJet system is a tribochemical silica coating method that can be used in the clinic and consists of a coating-abrasive sand and silane agent. Silica modified with 30 µm aluminum oxide sand particles are sprayed with a pressure of 2.8 bar at a distance of 10 mm for 15 seconds. This application not only prepares the surface for silane application but also provides micromechanical retention (30).

In the studies on tribochemical silica coating, silicon dioxide is transformed tribochemically and formed a connection with high density ceramics such as metal alloys and zirconia. In the study investigating the activity of the zirconia on the surface of silica-coated Al_2O_3 particles formed by the silica layer, aluminum and oxygen bonds with cements were detected with EDX analysis (31, 32).

In our study, CoJet system was used which was a tribochemical coating method that applied silica-coated Al_2O_3 particles of 30 µm size to the surface at a pressure of 2.8 bar for 15 seconds from 10 mm distance to allow silica particles to be embedded in the zirconia ceramic surface. EDX analysis was performed to the control group as well as to surface treated samples in order to determine the effectiveness of the procedures. When the surface treatments were compared to the control group, Al was detected in the sandblasting group, Si (atomic 8.8%) and Al on the CoJet group, and Si (atomic 20.05%) was detected on the surface in the ultrasonic spray group. The amount of Si in the ultrasonic spray group was approximately 2.5 times higher than the CoJet group. We believe that the increase of Si increases the chemical connection between zirconia and ceramics.

In the literature review, there is no report of the use of ultrasonic spray for covering the zirconia surface with tribochemical silica in the zirconia and ceramic intermediate layer. In this study, the ultrasonic spray device was used to coat the zirconia surface with silica. The silica coating of the surface was conducted with a 3-axis robotic nozzle that had a high movement sensitivity at 110 °C, with a flow rate of 1-5 ml/min and the vibrator was steadily pulsed at a frequency of 125 kHz and the spacing between nozzle and base was kept at 10 cm. The film was then magnified with complete silica coating by spraying (18).

In contrast to the CoJet system, Al_2O_3 sand is not used while applying silica and thus the negative effect of sand on the zircon surface is prevented. In SEM and EDX analysis, both the silica particles and Al_2O_3 particles were observed on the surface in the CoJet group and the whole surface was covered with silica in this system. In addition, the effect of Al_2O_3 sand on the surface was not seen in the SEM images of the ultrasonic spray group. In the EDX analysis, the silica layer was found to be on the entire surface and was present about 2.5 times more than the CoJet group.

The researchers examined the phase change of the surface application on the zirconia surface used two different Y-PRP zirconia. As control group, they prepared 100% tetrogonal zirconia from both materials. The results of the study indicated that the application of sandblasting alone or after abrasion increased the relative amount of monoclinic phase (33, 34).

Kosmac et al. emphasized that each application method causes phase change on zirconia and ceramics and multiple procedures are applied to the same sample, the monoclinic phase value of the last applied procedure is valid in determining the monoclinic phase value (35).

In the present study, XRD analysis performed to compare surface treatment applications with the control group revealed that the ultrasonic spray method gave almost the same values as the control group while the sandblasting group gave the most deviated results.

Conclusions

In this study, SEM, EDX, and XRD were used to analyze zirconia samples from the control, Al_2O_3, CoJet, and ultrasonic spray groups.

The following conclusions are drawn from this study:

- The SEM and EDX results show that the Al_2O_3 and CoJet surface treatment led to mechanical changes of the zirconia surface, with Al_2O_3 particles being detected on the

surface. It is possible that the external matter will negatively affect the connection of zirconia and porcelain. However, further studies are required to obtain a clear understanding.

- The XRD results show that the Al_2O_3 and CoJet groups yielded the most different results from the control group; however, the results of the ultrasonic spray group are almost similar to those of the control group. It is possible that ultrasonic spray treatment does not cause any phase change on the zirconia surface. However, further research is required to support this hypothesis.

Acknowledgments: This study was presented as a full-text oral presentation at the 1st International Dental Research and Health Sciences Congress held between 20-22 May 2021.

Peer-review: Externally peer-reviewed.

Author Contributions: Conception – G.Ö.; Design – Z.S.P., G.Ö.; Supervision – Z.S.P.; Materials – G.Ö., Z.S.P.; Data Collection and/or Processing – G.Ö., Z.S.P.; Analysis and/or Interpretation – G.Ö.; Literature Review – Z.S.P., G.Ö.; Writer – Z.S.P.; G.Ö.; Critical Review –Z.S.P.

Conflict of Interest: No conflict of interest was declared by the authors.

Financial Disclosure: This research has been supported by Dicle University, Scientific Research Project Coordination Unit. Project Number: DİŞ.16.001

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