Effect of the Sandblasting Process on the Surface Properties of Dental Zirconia

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This paper presents the effect of the sandblasting and sintering processes on the surface properties of some commercial yttria stabilized zirconia (Y-TZP) for monolithic dental restorations in dentistry. The surface properties of dental zirconia can be improved further through various surface treatment methods, like surface abrasion, roughening, chemical treatment, tribochemical coating, or selective infiltration etching. But all these treatments are made on the sintered samples, not on the pre-sintered discs as are delivered by the dental suppliers. The hypothesis of this paper was that the mechanical effect on the surface of pre-sintered disc of Y-TZP assured by airborne-particle abrasion with alumina will be maintained after the sintering process. Additionally, we will follow the presence of alumina particles on the Y-TZP surface after airborne-particle abrasion process. Surface modifications of the experimental samples was performed by sandblasting for 15 s with Al_2O_3 particles (average particle size 83 μm) at pressure of 2 bars, using a SAB-Caloris equipment. Morphological and surface changes in the sandblasted, respectively sandblasted and sintered samples of Y-TZP are examined by using scanning electron microscopy, energy dispersive spectroscopy, and X-ray diffraction. It is found that the surface modifications made on the pre-sintered Y-TZP disc remain after sintering process, which not affects these mechanical modifications of the surface. Also, it was detected the presence of alumina particles on the Y-TZP samples after airborne-particle abrasion process and for this reason we recommend the use of zirconia particle for airborne-abrasion process.

Keywords: Zirconia, dentistry, sandblasting, surface, microscopy

Choosing an ideal aesthetic material for dental restorations is a major desideratum in dentistry and necessity for improvement of the materials has led to a significant change in available materials and usage techniques.

There are a large number of inert ceramic systems available for clinical use in dentistry [1-6], but the scientific researchers have been demonstrating big interest in Y-TZP ceramic (Yttria-stabilized Tetragonal Zirconia Polycrystal), mainly motivated by the aesthetics advantages [6-8].

Zirconia provides a unique combination of material properties allowing it to be tailored for some specific technologies. In the case of dental restoration, zirconia offers a combination of good mechanical properties with optical properties, chemical resistance and bio-compatibility. These properties enhanced long-term viability of the zirconia dental restoration than porcelain and other non-metallic alternatives. Although superior in terms of mechanical performance when compared to alternative materials, a major problem associated with zirconia is their poor adhesion to the variety of synthetic substances or natural tissue that appears in dental applications [9,10]. In the case of monolithic restoration in dentistry, zirconia will be daily exposed to various stimuli such as oral mastication forces, exposure to various substances and water at different pH, temperatures and microorganisms from oral cavity [4, 5 , 9-13].

Some studies have investigated the effect of zirconia surface characteristics such as topography, surface chemistry and energy on cellular response. It has been shown that the surface topography affects cell response, interaction with oral tissue and ultimate clinical success [12]. Usually, the dental applications made by zirconia will be machining with CAD/CAM systems (computer aided design/computer aided machining), sintered before clinical use and adjusted by clinicians with diamond grinding instruments in order to achieve a better adaptation to the patient needs [1,2,11,14,15].

There are some reports about the methods used for surface modification of zirconia, which could be included in different categories:

- *mechanical modification of the surface by sandblasting with* airborne-particle *or roughening* with diamond bur. This surface modification establishes adhesion through micro-mechanical retention with no chemical bonding benefits. Some papers describe that particle abrasion results in the creation of sharp crack tips and structural defects that make dental application made by zirconia to be susceptible to radial cracking during service [16-18].
- *chemical modification of the surface* by application of phosphate ester primers or chlorosilane treatment. Other chemical based modification technique is the selective infiltration etching that creates an inter-grain nanoporosity at the surface where other substances can infiltrate and facilitate higher bond strength [19-22]. Various fluorination processes could be used to modify the properties of other inert surfaces. Also, fluorination vapor technique could be used to obtain an oxyfluoride conversion layer on zirconia surfaces.
- tribochemical coating with silica allows for chemical bonds to a silane coupling agent and resin cement [20,21].

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- ultraviolet (UV) light treatment;
- coating and biofunctionalization;
- laser treatment [21].

Sandblasting is used to modify the surface properties and produce a rougher surface compared to a machined surface. Also known in literature as airborne particle abrasion, sandblasting results are sensitive to various parameters such as shape, size, and kinetic energy of the particles affect the roughness on the surface. During the sandblasting process, air pressure creates impulse to eject the particles and the kinetic energy gained by the particles depends on the density, volume, and velocity of the particles [22]. One advantage of sandblasting is that a quite homogenous anisotropic abrasion can be performed on hard materials such as zirconia. Alumina particles have been used in generally for sandblasting due to their low cost, shape and hardness. But failure of alumina particles can happen upon impact and this might decrease the surface roughness as a consequence. Literature shows that the mechanical processing after sintering may trigger a tetragonal (*t*) to monoclinic (*m*) phase transformation; in addition to superficial alterations [8,14], which compromises the predictability of longevity of the prosthetic rehabilitation [10,12,17].

In the case of Y-TZP, the material's susceptibility to *t*-*m* phase transformation will depend on density, grain size, stabilizer content, processing characteristics and presence of residual stress [8]. Also, the Y-TZP's response when submitted to stimuli will be material dependent. The use of Y-TZP for full-contour monolithic restorations bring some clear advantages but is still necessary more information regarding the effect of grinding, sandblasting on the surface properties and interaction with oral environment that needs to be explored.

Therefore, the following research aimed to evaluate the effect of the mechanical surface modification of pre-sintered disc of Y-TZP assured by airborne-particle abrasion with alumina on the surface properties before and after sintering of Y-TZP.

Experimental part

Materials and methods

The purpose of the experimental researches carried out under this paper was to determine the surface properties of samples of inert ceramic biomaterials, namely zirconia, which were sandblasted with alumina particles and sintered after sandblasting.

This experimental study was conducted on two types of dental zirconia samples which have been provided from two different companies (Sirona and Wieland) as non-sintered blocks. In order to modify the surface properties of the non-sintered zirconia samples, it was decided to sandblast them with alumina particles using the device CALORIS SAB-type. Subsequently, a series of five sandblasted samples of each material were subjected to sintering, using a thermal treatment furnace specific to inert bioceramics used in dentistry.

The yttria-stabilized zirconia (Y-TZP) samples were fabricated by Computer-Aided-Design/Computer-Aided-Manufacturing (CAD/CAM) technology. The stages of obtaining experimental samples from the non-sintered zirconium block using the CAD / CAM technique are presented in figure 1. The CAD software allows the operator to obtain a design of the appropriate dental prosthesis step by step. This software contains the same steps that the dental technician would follow to obtain a fixed partial denture (FPD) framework such as: placement of die spacer for a cement layer, sketch of any undercuts, selection of the margin and. After that, a CAM milling machine is used to define the outside contours of the framework for 15 to 50 min.

The samples of zirconia with parallelepiped shapes and measuring 1.2 ± 0.2 mm were subjected to sandblasting with 83 μm aluminum oxide particles at 2 air pressure at a direction perpendicular to the surface for 15 s at a distance 10 mm away between the nozzle head and surface of the zirconia samples. The design of the granules on the surface is carried out by compressed air. With this technique, sandblasting procedures were performed using the CALORIS device SAB-type. Therefore, sandblasting is an important method of surface treatment that could improve the bond strength of materials and their stability and plays a role in cleaning and removing deposits from the surface of the sample.

Subsequently, the experimental samples were sintered in a furnace Mihm Vogt HT at 1500°C for 2h to form homogeneous tetragonal ZrO_2 with an increased hardness.

Table 1
EXPERIMENTAL SAMPLES USED IN THIS STUDY

Sample encoding	Sample type
Z1	Y-TZP Zirconia taken from non-sintered block (from Sirona company)
Z1-s	Y-TZP Sandblasted Zirconia (from Sirona company)
Z1-ss	Y-TZP Sandblasted and Sintered Zirconia (from Sirona company)
Z2	Y-TZP Zirconia taken from non-sintered block (from Wieland company)
Z2-s	Y-TZP Sandblasted Zirconia (from Wieland company)
Z2-ss	Y-TZP Sandblasted and Sintered Zirconia (from Wieland company)

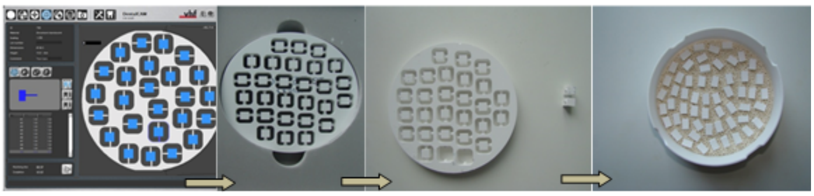

Fig.1 Stages of obtaining experimental samples from the non-sintered zirconium block using the CAD / CAM technique

Characterization techniques

In order to identify the amount of transformation which was induced by sandblasting and heat treatment, we measured the peak intensity ratio in the X-ray diffraction (XRD) patterns of samples. The determinations were performed by a diffractometer X'Pert PRO MPD-Panalytical using CuKα=1.5406 Å radiation at 40 Kv and 120 mA.

The surface properties of the experimental samples were investigated comparatively by scanning electron microscopy (SEM) analysis in order to evaluate the effect of subsequent sandblasting and sintering process on dental zirconia samples. Scanning electron microscopy analysis was realized on both types of zirconia using a Scanning Electron Microscope (SEM) Quanta Inspect F50 (FEI) coupled with energy dispersive X-ray spectrometer. For investigation the surface morphology, the microscope it was operated at a voltage of 30 kV and a pressure of 0.7 torr, after the samples were coated on the surface with gold. Energy dispersion X-ray spectroscopy (EDX) was used for the surface elemental composition evaluation.

Results and discussions

Determination of X-ray diffraction (XRD)

The structural characteristics of non-sintered and sintered zirconia samples in order to evaluate the changes of crystalline structure are shown by XRD diffractograms in the following figures (fig. 2 and 3). The sintering process converted the ZrO_2 from the monoclinic to tetragonal system according to the ICDD file: 01-075-9648.

Following X-ray diffraction analysis, we can state that for both types of sintered or non-sintered zirconia Z1 and Z2, the basic compositional element is ZrO_2, which demonstrates a high stability of this inert bioceramics, regardless of the operations to which it can be subject.

Also, the X-ray diffraction analysis helps us to observe the transformations of the crystalline systems according to the diffraction peaks and the interplanar distances of these samples, following the sintering operation. Thus, the figures 3 show the XRD patterns of both zirconia types after sandblasting by aluminium oxide particles and sintering process. In comparison to the XRD patterns after the sintering process, the diffraction peaks due to monoclinic ZrO_2 increased with sandblasting process while the diffraction peaks for tetragonal ZrO_2 decreased and shifted to a lower degree.

Determination of Electronic Scanning Microscopy (SEM) coupled with Energy Dispersion X-ray Spectroscopy (EDS)

The images obtained from SEM coupled with EDS analysis are shown in the following figures (figs. 4,5 and 6).

Figure 4 and 5 shows the SEM images of the samples Z1, respectively Z2, at higher magnification. Following the investigations, some surface changes can be observed following the sintering and sandblasting techniques applied to both samples of zirconia (Z1, Z2). After sintering, we can observe a gradient reduction at nanometric level with scanning electron microscopy.

Also, the micrographs reveal that the samples have high densification and composed of small grains (on the average of 68 nm). After the sintering process, it can be seen a decrease in the size of ceramic grains.

The main conclusion after our SEM analysis is that the surface modifications made by sandblasting process on the non-sintered zirconium blocks keep their profile and morphology after the sintering process.

Due to the strong difference on the hardness of the zirconia samples, we could recommend the use of surface modification for dental zirconia using mechanical methods like sandblasting before the sintering process because these surface changes are kept after sintering.

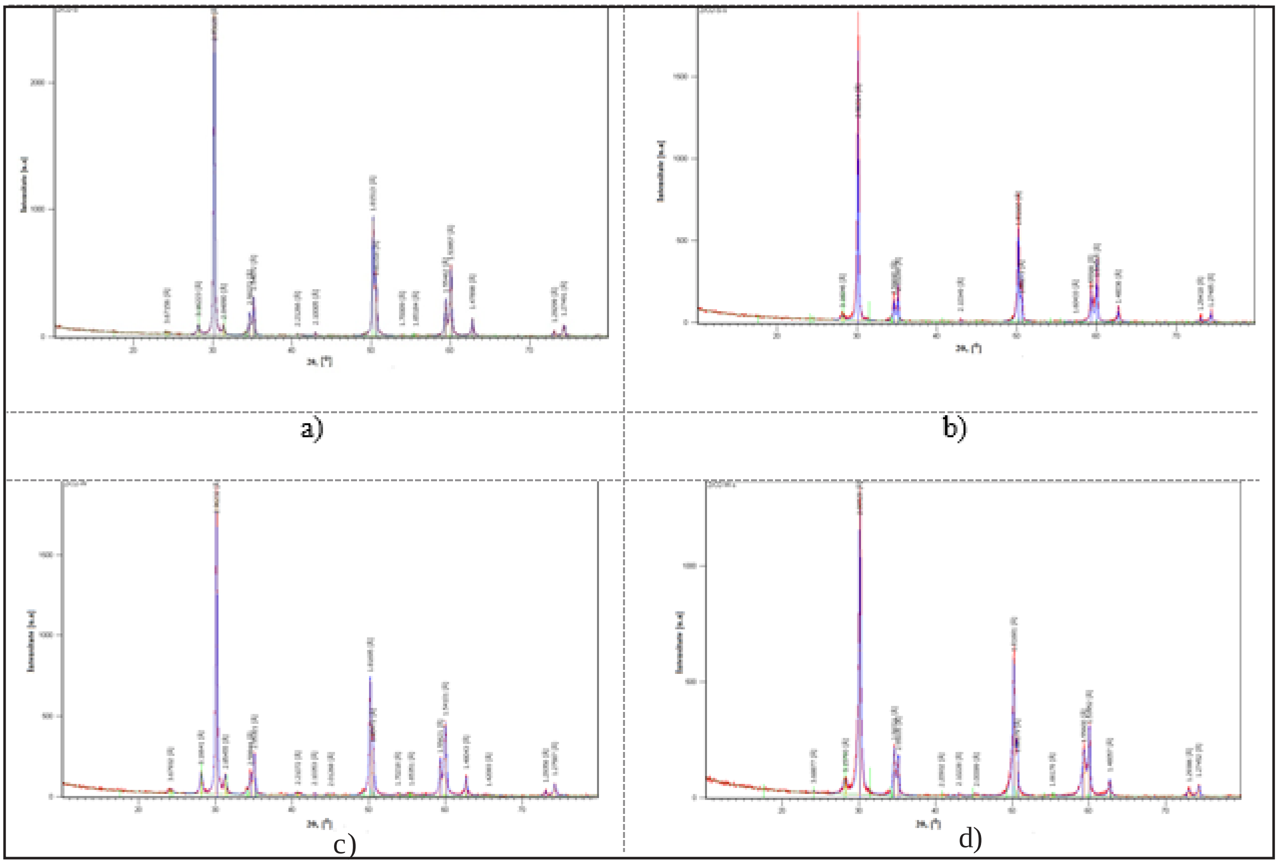

Fig 2. XRD spectra showed the interplanar spacing's of the diffraction peaks for experimental samples Z1 and Z2, before and after sandblasting process: a) Z1; b) Z1-s; c) Z2; d) Z2-s.

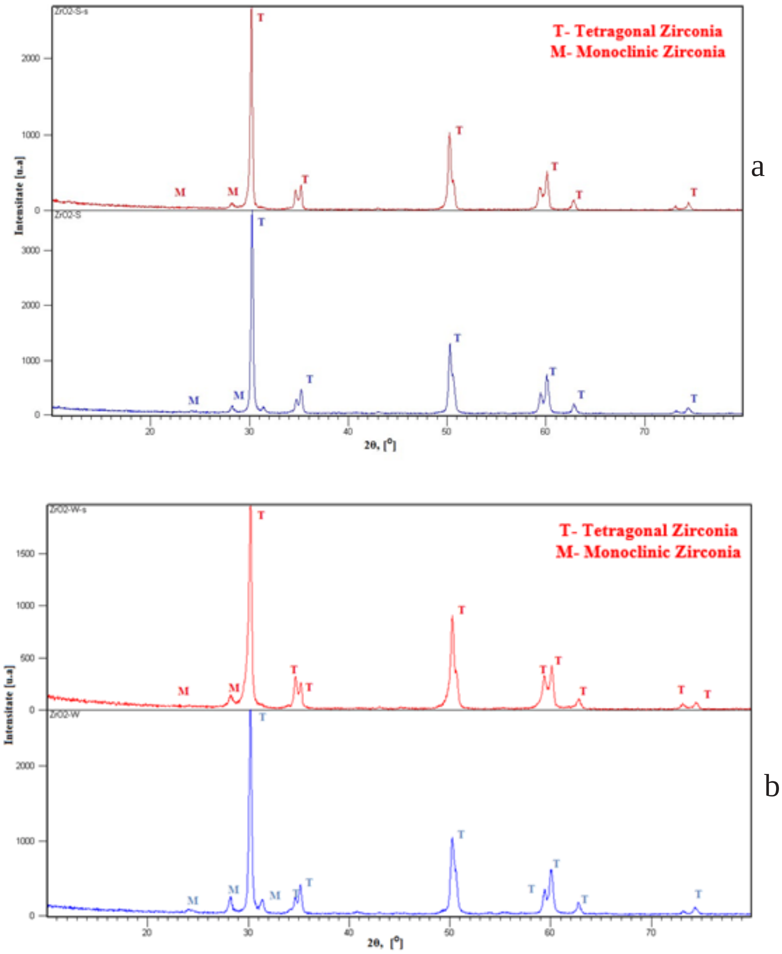

Fig. 3. XRD patterns shown a comparison between the two types of zirconia (Z1, Z2) depending on their crystalline systems after the sandblasting and sintering process: a) Z1-s and Z1-ss ; b) Z2-s and Z2-ss

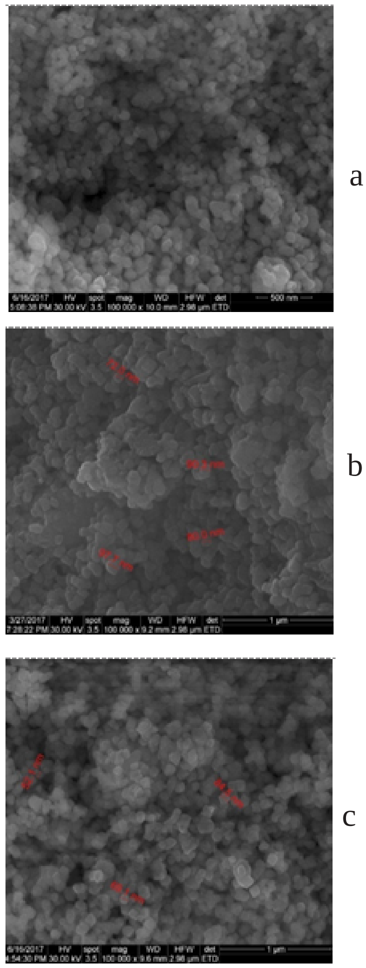

Fig. 4. SEM images for the surface analysis of the Z1 samples (100.00x): a) Z1; b) Z1-s; c) Z1-ss

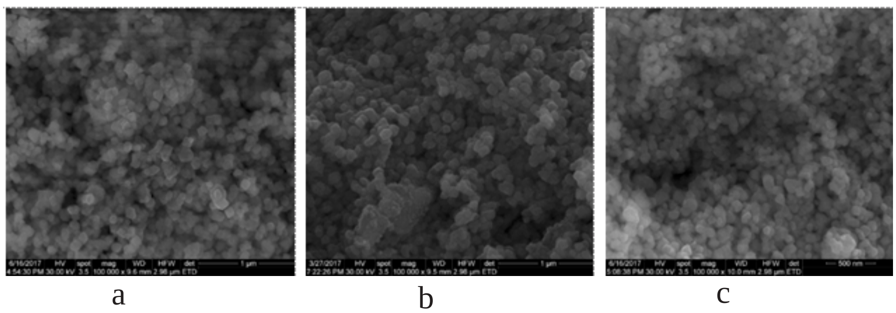

Fig. 5. SEM images for the surface analysis of the Z2 samples (100.00x): a) Z2; b) Z2-s; c) Z2-ss

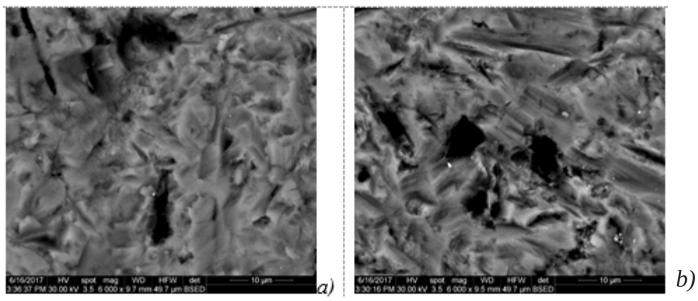

Fig. 6. SEM images for all zirconia samples after sandblasting and sintering process, revealing the presence of alumina particles at the surface: a) Z1-ss; b) Z2-ss.

Also, using the SEM analysis we could observe the continuous presence of alumina particles by lighter colored specific portions (fig. 6).

Figure 7 shows the EDX spectra of the all zirconia experimental samples, in different stages of the surface treatment, recorded on selected punctual area, in order to obtain information about the elemental composition.

The EDX spectra of the investigated samples, recorded on 50 x 40 mm areas revealed that the main element from the elemental composition of the experimental samples were zirconium and identify the presence of yttrium. According the results, aluminium was identified at the surface of all zirconia samples after sandblasting process with alumina particle. Because is still present after the sintering process, we recommend to use other particle like zirconia for sandblasting process of dental zirconia.

In another paper were studied the effects of alumina sandblasting on the orthodontic bracket surce [23].

Conclusions

According to the results of the current study, after sintering process the experimental zirconia samples reduced their volume and also the crystalline monoclinic system was converted to a tetragonal crystalline system.

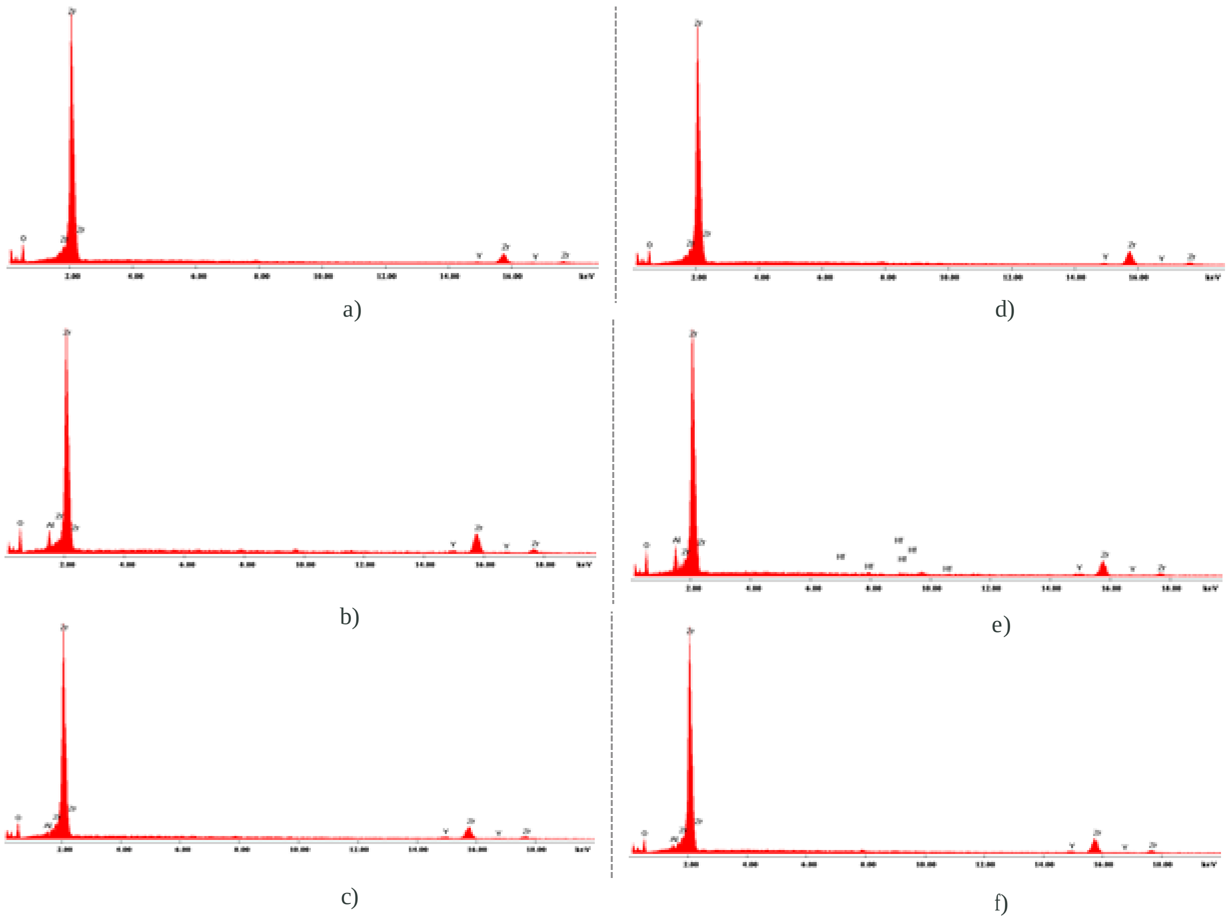

Fig. 7. EDX spectra of the all zirconia experimental samples, in different stages of the surface treatment:
a) Z1; b) Z1-s; c) Z1-ss; d) Z2; e) Z2-s; f) Z2-ss

Scanning electron microscopy confirmed the results of XRD analysis, such that after the sintering process it can be seen a decrease in the size of ceramic grains.

The surface modifications made by sandblasting process on the non-sintered zirconium blocks keep their profile and morphology after the sintering process. Due to the strong difference on the hardness of the zirconia samples, we could recommend the use of surface modification for dental zirconia using mechanical methods like sandblasting before the sintering process because these surface changes are kept after sintering.

Also, by scanning electron microscopy (SEM) and EDX spectrometry, we can observe the presence of small alumina particles at the surface of all zirconia samples after sandblasting process. Because is still present after the sintering process, we recommend to use other particle like zirconia for sandblasting process of dental zirconia.

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