

Characterization of the Specificity in Three Commercial Dental Ceramic Powders

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The aim of this study was to present and discuss the characteristics of three ceramic powders as well as the sintered material from the three commercial dental ceramics produced by Vita Zahnfabrik, Germany (VM13 and VMK Master; used for veneering metal frameworks, respectively VM9 used for veneering zirconia frameworks). An X-ray diffraction (XRD) analysis was performed, Raman spectra were recorded and the morphology of the samples was evidenced by using a high resolution scanning electron microscope, equipped with an EDXS detector. The results of this study corroborated with the presented literature data helps practitioners to better understand the interaction of these biomaterials with oral tissues, and, also helps researchers to modify the properties of ceramics for a better integration in the intraoral condition.

Keywords: dental ceramics, X-ray diffraction, Raman spectroscopy, scanning electron microscopy

The use of ceramics as dental materials experiences a continuous improvement of the commercial products and their properties in several prosthetic applications. Metallic frameworks, but also most of the high strength ceramics are layered with feldspar dental porcelain in order to get better match of aesthetics and functional needs in the mouth [1].

As restorative materials, dental ceramics have disadvantages mostly due to their inability to withstand functional forces that are present in the oral cavity. Hence, initially, they found limited application in the premolar and molar area, although further development in these materials has enabled their use as a posterior long-span fixed partial prosthetic restorations and structures over dental implants. All dental ceramics display low fracture toughness when compared with other dental materials, such as metals [2].

Metal ceramic systems combine both the exceptional esthetic properties of ceramics and the extraordinary mechanical properties of metals. Some metals used as restorative materials in dentistry may constitute a problem for some patients and these drawbacks, as well as the search for more esthetic materials by patients and dentists, have stimulated research and development of metal-free ceramic systems. While porcelain-based materials are still a major component of the market, there have been moves to replace metal ceramics systems with all ceramic systems [3].

There is a correlation between composition, structure and morphology of the available materials and their properties. This information may be used to predict properties and improve the design of new materials [4].

It is necessary to fully characterize the commercially available materials in order to understand their properties.

The aim of this study was to present and discuss the characteristics of the powders as well of the sintered material for three commercial dental ceramics produced by Vita Zahnfabrik, Germany: VM13 and VMK Master used for veneering metal frameworks and VM9 used for veneering zirconia frameworks.

Experimental part

Materials and methods

For each commercial product there were analyzed three precursor powders (two dentin powders and one enamel powder) and a tooth-like structure fired according to the manufacturer specifications.

An X-ray diffraction (XRD) analysis was performed with a Rigaku SmartLab diffractometer using Cu K α radiation in a 2θ range 20-120°. Raman spectra were recorded at room temperature using an inVia confocal Raman microscope (Renishaw) with a diode DPSS visible laser source (532 nm) and a Peltier cooled CCD detector. The single beam power of the laser was 25 mW and the 50x objective of the microscope was used.

The morphology of sintered pellets was evidenced using a high resolution scanning electron microscope (Hitachi SU8010). The microscope was equipped with an EDXS detector from Oxford Instruments. Images were taken using an accelerating voltage of 1 kV.

Results and discussions

X-ray diffraction results are shown below (figs.1-3).

In all precursor powders samples the phases detected were leucite and quartz.

All sintered samples showed alterations in phase composition and structure.

The Raman spectroscopy revealed the presence of the monoclinic structure (space group C 1 2/m 1) characteristic to sodium potassium alumino-silicate. (ICSD card 9583) (figs.4-6).

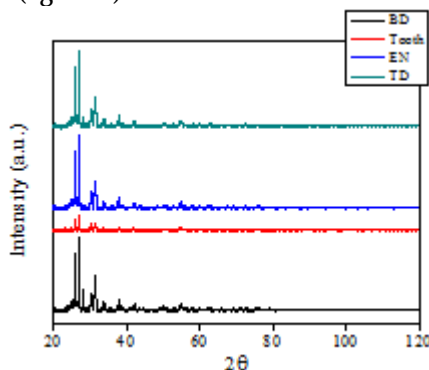


Fig.1. XRD patterns of MC_VM13 precursor powders and thermally processed tooth

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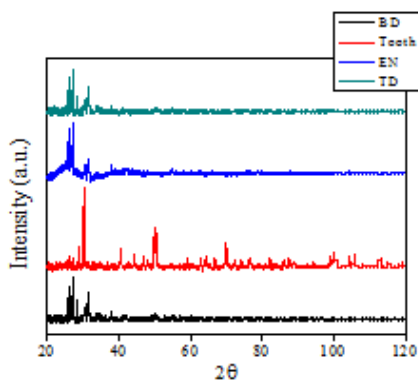


Fig. 2. XRD patterns of MC_VMKMaster precursor powders and thermally processed tooth

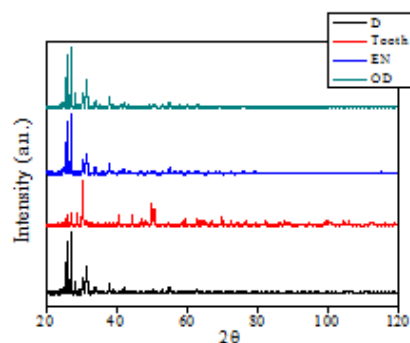


Fig. 3. XRD patterns of Zr_VM9 precursor powders and thermally processed tooth

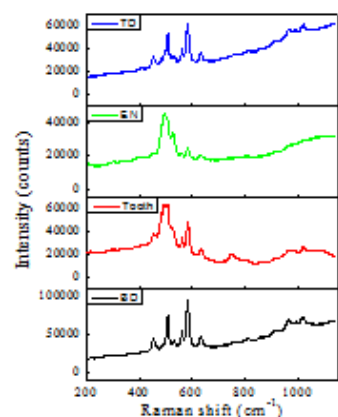


Fig. 4. Raman spectra of MC_VM13 precursor powders and thermally processed tooth

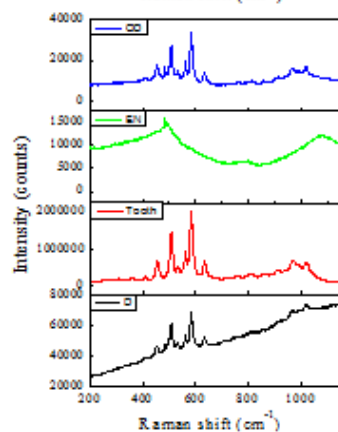


Fig. 5. Raman spectra of MC_VMKMaster precursor powders and thermally processed tooth

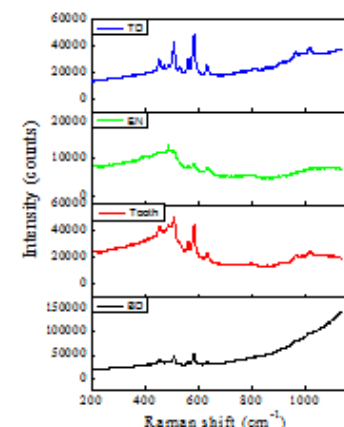


Fig. 6. Raman spectra of Zr_VM9 precursor powders and thermally processed tooth

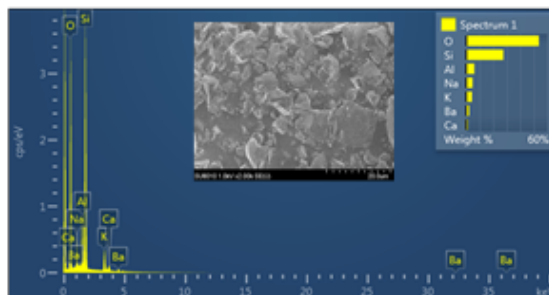


Fig. 7. MC_VM13_Base Dentin

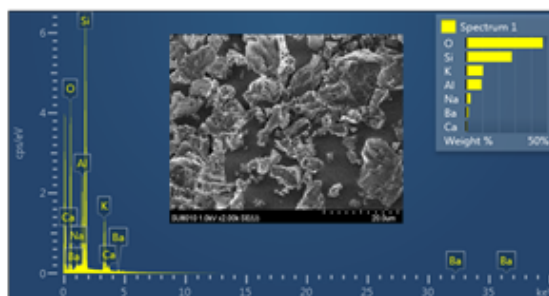


Fig. 8. MC_VM13_Transparent Dentin

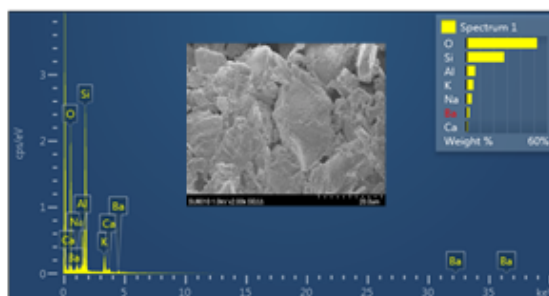


Fig. 9. MC_VM13_Enamel

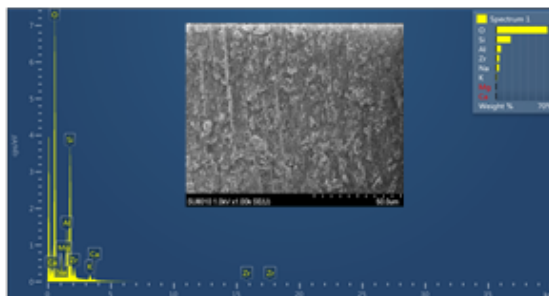


Fig. 10. MC_VM13-Tooth

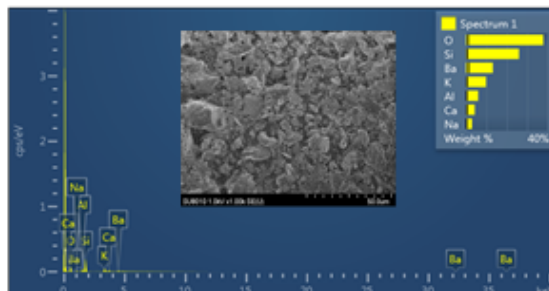


Fig. 11. MC_VMK Master_Opaque Dentin

Even the EDXS analysis evidenced slightly differences in composition, it offered a chemical composition on the prismatic crystals corresponding to potassium feldspar. The scanning electron microscopy revealed the appearance of crystals with various sizes grouped in clusters non-uniformly distributed throughout the matrix with a characteristic dendritic appearance.

The precursor powders and the sintered samples morphology and composition determined by the EDXS analysis (figs. 7-18).

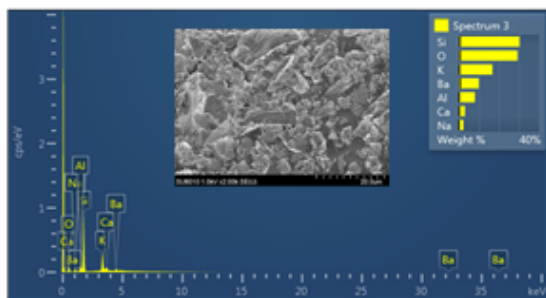


Fig. 12. MC_VMK Master_Dentin

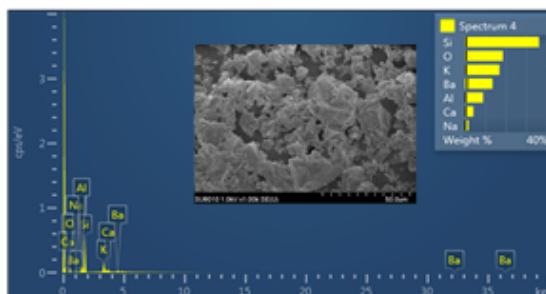


Fig. 13. MC_VMK Master_Enamel

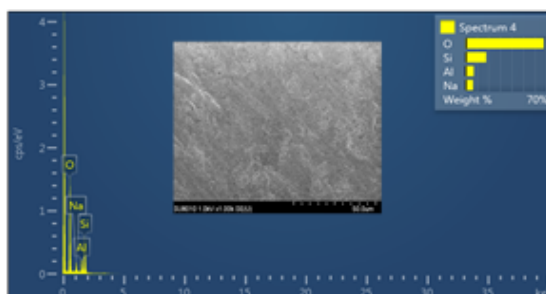


Fig. 14. MC_VMK Master_Tooth

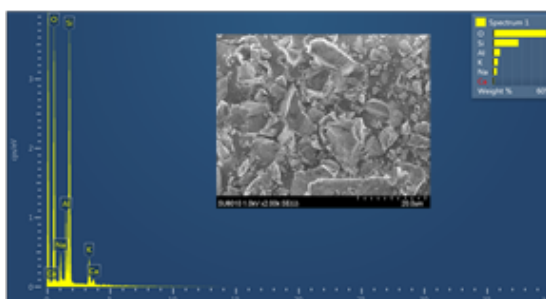


Fig. 15. Zr_VM9 Base Dentin

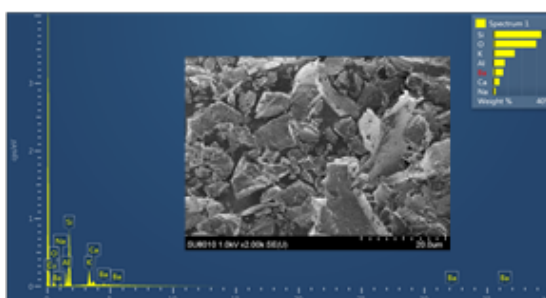


Fig. 16. Zr_VM9 Transparent Dentin

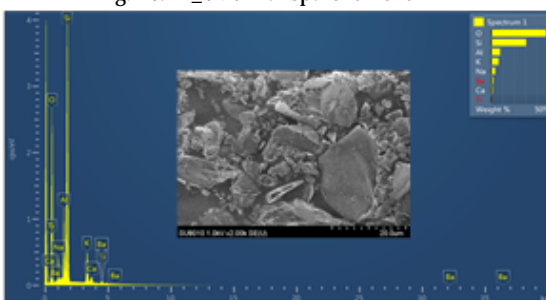


Fig. 17. Zr_VM9 Enamel

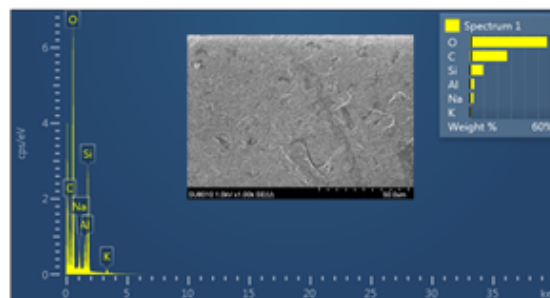


Fig. 18. Zr_VM9 Tooth

Regarding to the materials used in our study, according to the producer information, VITA VMK Master is a natural feldspar veneering ceramic for conventional bonding alloys (approx. $13.8 - 15.2 \mu\text{m/mK}$). VITA VM 13 is a highly-esthetic, fine-structure feldspar ceramic that is perfectly adapted to the CTE value of conventional bonding alloys (approx. $13.8 - 15.2 \mu\text{m/mK}$), while VITA VM 9 is a ceramic that is perfectly adapted to the CTE value of zirconia frameworks (approx. 10.5, e.g. such as VITA YZ). The main components of these ceramics are pure-grade potash and albite feldspar materials that offer brilliant shade effects as well as optimum physical properties, such as extreme flexural strength values [5].

Ivoclar Vivadent also succeeded in developing different types of ceramics by applying the principles of controlled nucleation and crystallization. The main challenge in the process was to imitate the optical, mechanical and chemical properties of natural teeth. In order to meet these demands, different nucleation and crystallization mechanisms must be developed together in order to achieve crystal-phases that are chemically different and exhibit different properties. Leucite, for example, imparts the glass-ceramic with a high coefficient of thermal expansion, CTE, while a fluorapatite phase leads to beneficial optical properties. In order to combine these characteristics in one material, twofold nucleation and crystallization are necessary, which were achieved in the $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-K}_2\text{O-Na}_2\text{O-CaO-P}_2\text{O}_5\text{-F}$ system [6]. It is also possible to prepare a very thin coating on the surface of the glass-ceramic powder to improve the slurry processing technology. The resulting leucite- fluorapatite glass-ceramic demonstrates unique properties. It exhibits a high CTE of about $12 \cdot 10^{-6} \text{ K}^{-1}$ ($100\text{-}500^\circ\text{C}$) as well as translucent properties. This type of glass-ceramic (IPS d.SIGN®, Ivoclar Vivadent AG) is suitable for the veneering of metal or pressed ceramic frameworks [7].

One common crystalline filler is the mineral leucite, used in relatively low concentrations in porcelains for metal-ceramic systems and is used in higher concentrations as a strengthening filler in numerous all-ceramic systems. In general, higher fraction of polycrystalline components increase the strength and the toughness of a ceramic. The development of substructure ceramics for fixed restorations represents a transition towards fully polycrystalline materials [8]. Leucite ceramics used in dentistry is prepared from highly viscous alkaline aluminosilicates, from which leucite crystallized as a main crystalline phase. The technology of separated preparation of leucite crystals and glassy matrix seems to be more suitable (compared to high temperature process) for the fabrication of leucite ceramics having controlled microstructure, realized by homogenous dispersion of leucite particles in a glassy matrix. Final microcomposite structure is obtained by sintering of these two phases. This technology ensures reproducible control of leucite ceramics' microstructure, which is required to improve its fracture toughness and persistence. The fundamental step

for an extensive use of this technology consists in the reproducible synthesis of leucite [9].

Most of the ceramics for the metal-ceramic restorations are composed of leucite crystals dispersed in a glassy matrix. Leucite has a high coefficient of thermal expansion (CTE) and raises the overall thermal expansion of the bulk porcelain leading to thermal compatibility with metal frameworks. The amount of leucite and the amount and composition of glass determine decisively the CTE of the final product [10]. Also other studies revealed the fact that potassium feldspar can be produced from leucite in excess of SiO_2 which exists both in the amorphous phase and in the form of quartz. Although sanidine, the stable phase of potassium feldspar at room temperature can be precipitated under certain heat treatments and especially in low fusing dental ceramics [11], and leucite has been observed to convert to the sanidine polymorph of feldspar during heat treatments within the normal firing range of dental porcelain [12]. For the feldspar ceramics an increase of leucite content may be observed after sintering. This increase in crystalline content was attributed to the crystal growth of leucite nuclei that are present in the starting powders. In leucite-based porcelain, most of the crystalline phase is introduced in the frit and additional uncontrolled crystallization might occur during firing procedures employed in dental laboratories [13].

A 2011 study made on a CAD-CAM dental ceramics showed for the feldspar ceramic sample two crystallization patterns, with peaks identified as *a* and *b*, with *a* being asodium potassium aluminum silicate peak ($\text{Al}_8\text{K}_2\text{Na}_6\text{O}_{34}\text{Si}_9$) and *b* being a potassium sodium aluminum silicate peak ($\text{AlK}_0.29\text{Na}_{0.71}\text{O}_8\text{Si}_3$) [14]. Other findings reported in the literature showed microstructure based on an aluminum- potassium- and sodium-based silicate with grains of about $4\mu\text{m}$ for the feldspar dental ceramics [15].

The evolution of dental ceramics over the past 30 years has been most interesting from a biomedical engineering point of view. Introduction of zirconia ceramics has opened a wide range of all-ceramic applications unthinkable 30 years ago. The consensus, however, is on caution in selecting highest product quality and strict respect of manufacturers' recommendations, with special attention on sintering temperature [16].

In present, alumina and zirconium-based ceramics are the latest materials used for fixed partial dentures core and ceramic crowns [17].

The requirements for an acceptable dental material are many, but one of the most important is the biocompatibility. Furthermore, it should contain no toxic, leachable, or diffusible substances that can be absorbed into the circulatory system, causing systemic responses, including teratogenic or carcinogenic effects [18].

Conclusions

Characterization of widely used commercial dental ceramics helps practitioners to better understand the interaction of these biomaterials with oral tissues, but also helps the researchers to modify their properties for a better integration in the intraoral condition.

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Manuscript received: 17.11.2016