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Review

Zirconia in dental medicine: a brief overview of its properties and processing techniques

[1]Ana Ispas, [2]Laura Iosif, [2]Cătălina Murariu-Măgureanu, [1]Antarinia Craciun, [1]Mariana Constantiniuc

[1]Department of Prosthodontics, Faculty of Dental Medicine, "Iuliu Hatieganu" University of Medicine and Pharmacy, Cluj-Napoca, Cluj, Romania; [2]Department of Prosthodontics, Faculty of Dental Medicine "Carol Davila" University of Medicine and Pharmacy, Bucharest, Romania.

Abstract. Over the last three decades, the medical scientific literature has shown increasing interest in zirconia, starting from the need to introduce promising alternatives to titanium which is used on a large scale for various human structure replacements, such as those in the field of orthopedics, prosthetic dentistry and oral implantology. As an inorganic non-metallic oxide material, zirconia successfully meets the biocompatibility criteria while also presenting other excellent properties, such as: interesting optical features, high flexural strength and fracture resistance, good chemical resistance, which, in turn, can be optimized by the addition of inorganic compounds, known as stabilizers. In the category of additives meant to increase the structural stability of zirconia, most of the attention in dentistry has been focused on rare-earth oxides, namely: yttrium (Y2O3), which contributes, significantly, to stabilizing the material that is applied in conditions of high corrosion and pressure, thus allowing for high bite forces to withstand a hostile oral environment. This important improvement in the strongest of dental ceramics may allow the fabrication of posterior fixed partial dentures. In addition, it has large economic consequences for dental manufacturers, clinical providers, while also being beneficial for patients. Thus, this article aims to review the zirconia generation that is currently attainable, together with the positive properties, weaknesses and processing techniques available in dentistry. We performed an electronic search in the literature for articles written in English using Pub Med. This review analyzed data provided by specialists in articles on the current generations, microstructure, properties and manufacturing process of zirconia.

Key Words: zirconia, prosthetic dentistry, stabilizers, processing techniques

Corresponding Author: L. Iosif, email: laura.iosif@umfcd.ro

Introduction

Zirconia (ZrO_2) is a bioceramic material, which was first accidentally extracted by the German chemist Martin Heinrich Klaproth in 1789, while analyzing a precious mineral from the class of silicates (Zircon) and named by him for the first time as "Zirkonerde" (zircon earth, or zirconia) (Iosif et al 2020). It was later isolated in pure form by the Swedish chemist Jöns Jakob Berzelius in 1824 (Wisniak, 2000).

The first research paper on the use of ZrO_2 as a biomaterial was published by Helmer and Driskel, in 1969 (Manicone et al 2007), the authors proposing a particular compound, namely: zirconia, for hip head prostheses, instead of titanium or alumina. The disadvantages of these metals, namely: the esthetic problems, the corrosion and fatigue-related aspects led to their substitution with ceramics and polymers in dentistry (Hanawa, 2020). Thus, zirconia was firstly introduced in dental medicine in the early 1990s and it was initially used as a core material to support more esthetic ceramic materials (Malkondu et al 2016). Currently, zirconia is widely used in prosthetic dentistry due to its excellent biocompatibility, low cytotoxicity, chemical stability, high mechanical strength, superior fatigue resistance, high fracture resistance and a Young's modulus similar to that of stainless-steel alloy. In order to avoid the formation of ceramic cracks during the heating/cooling process, different amounts of stabilizers, such as: yttria (Y_2O_3), magnesia (MgO), alumina (Al_2O_3), ceria (CeO_2), and calcium oxide (CaO) must be added to the zirconia (Pekka, 2015).

Due to its properties, as well as to the extended development of digital technological equipment, zirconia is actually used in the making of individual dental crowns, short fixed partial dentures (FPDs) and implants. Thus, the aim of this article is to update the current knowledge of the optical, physical, chemical, mechanical and biological properties, alongside the clinical performance of zirconia ceramics. This article is also meant to highlight the latest information related to the manufacturing process and use of zirconia, in the field of dentistry. We performed an electronic search in the literature for articles written in English using Pub Med. This review analyzed data provided by specialists in articles on the current generations, microstructure, properties and manufacturing process of zirconia.

Microstructure and types of zirconia used in dentistry

Zirconia (ZrO_2) is a heterogeneous, highly-resistant, polycrystalline ceramic. Zirconia is a well-known polymorph that can present three crystallographic structures according to the temperature: monoclinic (M), cubic (C) and tetragonal (T), as shown in Fig. 1.

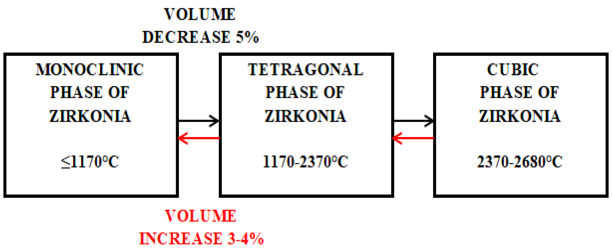

Fig.1. Temperature-dependent polymorphic phases of zirconia (Iosif et al 2020)

Pure zirconia is monoclinic at room temperature. This structure is stable up to 1170°C. When this temperature increases, zirconia gets transformed into the tetragonal form (T) and into the cubic (C) form at 2370°C, followed by a fluorite form at a temperature of 2370 °C (Iosif et al 2020). Transitions from one form to the other are known as martensitic transformations, thus subjecting the crystal structure to cracking. There are a multitude of strategies to avoid cracking in ZrO_2-based ceramics (Crystal et al 2020).

Many researchers have demonstrated that low quantities of calcia (CaO), magnesia (MgO), ceria (CeO_2) and yttria (Y_2O_3) in a solid solution of ZrO_2 can stabilize the tetragonal or cubic phase of ZrO_2 at ambient temperature, depending on the amount of oxide added (Hanawa, 2020). Thus, regarding the composition of the zirconia used in dental medicine, many types of zirconia-containing ceramic systems are currently available (Denry & Kenlly 2008; Guatam et al 2016; Hannink et al 2000), but only three types are used in dentistry to date. They are yttrium cation-doped tetragonal zirconia polycrystals (Y-TZP), magnesium cation-doped partially stabilized zirconia (Mg-PSZ) and zirconia-toughened alumina (ZTA). It has been reported that the nanocomposites of Al_2O_3–ZrO_2 have a high resistance to the crack propagation, which can improve the lifespan and reliability of the ceramic prostheses (Gautam et al 2016).

Magnesia partially stabilized zirconia (Mg-PSZ) has been studied for various biomedical applications (Garvie, 1984), mainly being used in high load bearing sites such as artificial knee and bone screws in orthopedic application and in the manufacturing of dental crowns (Hao & Lawrence, 2003). The structure of Mg-PSZ consists of tetragonal precipitates within a cubic stabilized zirconia matrix and is obtained by introducing Mg^{2+} cations into the c-ZrO_2 and/or t-ZrO_2 lattices (Soylemez, 2020). The amount of MgO in the composition of materials usually varies between 8 and 10 mol%. Moreover, the presence of porosity, associated with a large grain size (30–60µm) can induce wear (Denry & Kenlly 2008).

Regarding the yttria-tetragonal zirconia polycrystals (Y-TZPs), it must be mentioned that they have been introduced to dentistry for about a decade, in the purpose of manufacturing the crowns and the framework of FPDs (Guatam et al 2016; Hisbergues et al 2009).

Yttria (Y_2O_3) has proved to be the most effective material for providing a combination of high strength and toughness. Thus, Y-TZP receives unusual physical and mechanical properties such as: high flexural strength, fracture resistance, hardness, corrosion resistance, wear and tear in both acidic and basic environment, translucency, color stability, greater efficacy of diagnostic radiographs and high biocompatibility (Munoz-Saldana et al 2003). Firstly, tetragonal zirconia (TZP) stabilized with 3 mol% of yttria with about 0.25 wt% of alumina (3Y-HA) was used as a core ceramic instead of metal framework. It was covered with porcelain because 3Y-HA provided an insufficient translucency. The studies have shown that the veneering porcelain does not withstand the load in the posterior area of the dental arch (Zhang & Lawn 2018).

Furthermore in 2011, a high translucent 3Y-TZP has been promoted as an alternative to titanium implants and abutments, owing to its more natural coloration, greater wear and corrosion resistance, improved biocompatibility, soft tissue integration, lower affinity to bacterial plaque and low development of peri-implantitis. Clinical research has indicated, however, that early fracture rates of zirconia implants tend to be higher than the titanium ones, (Pieralli et al 2017) so improvement in mechanical integrity becomes an overriding concern.

TZP stabilized with 3 mol% of yttria with less than 0.05 wt% of alumina (3Y) was introduced to dentistry, in 2011. It is indicated for manufacturing the monolithic zirconia crowns, which are applied in the posterior area of dental arches. Some studies have demonstrated that the fracture resistance of monolithic zirconia crowns has an obvious superiority for monolithic crowns, even if they are glass-infiltrated (Kontonasaki et al 2020) in comparison to veneered bilayer. The structure of 3Y-TZP ceramics for the dental applications consists of small grains (0.2–0.5µm in diameter), depending on the sintering temperature.

The mechanical properties of 3Y-TZP depend on the grain size and the sintering temperature. For example, given a grain size greater than 1 µm, 3Y-TZP is less stable and more susceptible to spontaneous T » M transformation, while the grain size smaller than 0.2 µm offers reduced fracture resistance (Denry & Kenlly 2008; Preis et al 2012; Kaizer et al 2019).

Furthermore, in view of this aspect, recent research has proved that high temperature and long sintering periods determine the larger grain sizes and will subsequently diminish the mechanical properties because of the large pore sizes (Kontonasaki et al 2019).

The next stage in the development of monolithic zirconia has been to increase the yttria percentage ($6 < Y_2O_3 <10$ wt %) and translucency that is superior to that of 3Y-TZP. This is stabilized zirconia (PSZ), which is stabilized with about 4 mol% of yttria (4Y) and partially stabilized zirconia (PSZ) stabilized with about 5 mol% of yttria (5Y). All of these materials have been developed for anterior crowns (Zhang & Lawn 2018). Thus, in this respect, 5Y-PSZ is recommended due to the natural esthetic appearance, reduced thickness of the preparation, short production time and low cost.

Zirconia, which has a higher translucency value, showed a higher fatigue strength than the other ceramic materials (feldspathic ceramic, polymer-infiltrated ceramic network, lithium disilicate glass-ceramic and zirconia-reinforced lithium silicate glass-ceramic) after being loaded in a cyclic manner (Sulaiman et al 2016).

Furthermore, translucent PSZ stabilized with 6 mol% of yttria (6Y) was developed in 2017. From 2016 to 2018, not only shade-lamination, but also a mixed composition-lamination type has been provided consisting of TZP and PSZ (Ban, 2020).

Zirconia properties

1. Physical properties

In the ceramic classification, zirconia (ZrO_2) is characterized by favorable mechanical properties (toughness: 9–10 MPa√m, flexural strength: 900–1200 MPa, Young's modulus: 210 GPa) (Malkondru et al 2016).

Previous studies have reported that Al2O3 in zirconia can also enhance the mechanical properties of zirconia.

In their studies, Kontonasaki et al (2019) showed that mechanical properties, such as the flexural strength and the fracture resistance, were significantly reduced when Y_2O_3 increased.

Regarding the studies carried out *in vitro*, they demonstrated that monolithic zirconia presented higher resistance to fracture than the bilayered ones, after mechanical cycling and aging (Kontonasaki et al 2019; Ban, 2020).

The finishing procedure of the occlusal surface did not influence the mechanical resistance and neither did the cementation procedure, particularly onto implants; on the contrary, according to a few studies (Zarone et al 2019; Mitrov et al 2016; Øilo et al 2016), the fracture resistance has been shown to be affected by the type of preparation and low temperature degradation, opposite to the finishing procedure of the surface and the type of cementation technique.

However, mention must be made that the type of preparation and material are vital for increasing the longevity of monolithic zirconia restorations (Kontonasaki et al 2019; Kontonasaki et al 2020).

2. Chemical properties

Among the chemical properties of zirconia we count: dissolution resistance, chemical etching and discoloration. Low temperature degradation is usually highlighted as being a type of degradation occurring because of the phase transformation, in the presence of moisture (Zhang & Lawn 2018; Ban 2020).

This process can appear when the surface is exposed to humid environment and moderate temperatures. In this respect, the zirconia crown is exposed directly to the oral environment and it cannot survive in the oral cavity for a long time (Pekkan et al 2020).

Furthermore, the process depends on the microstructure, composition and stress state. The ageing phenomenon - as it is defined in the scientific literature - consists in a slow tetragonal to monoclinic transformation of the grains at the surface in contact with water molecules (Camposilvan et al 2018).

However, some studies have concluded that the zirconia degradation by water is caused by oxygen defects (Ban 2020; Chevalier et al 2009).

The main steps of zirconia degradation are:

- Chemical adsorption of H_2O on the ZrO_2 surface,
- Reaction of H_2O with O_2 on the ZrO_2 surface to form a hydroxyl ion (OH),
- Insertions of OH into the inner part by grain boundary diffusion,
- Adding of oxygen vacancies within the grains by OH ions, followed by the formation of proton defects.
- Occurrence of the transformation from the tetragonal form to monoclinic form when the oxygen vacancy concentration is diminished to the extent that the tetragonal form is no longer stable.

Thus, Chevalier et al (2009) have highlighted the importance of precise processing techniques in avoiding accelerated ageing.

3. Optical properties

Zirconia presents optical and esthetic properties superior to other restorative materials. One of the optical properties of zirconia is translucency, which is defined as the amount of light transmitted through a material. A translucent material permits light to pass through it, the light is absorbed and eventually scattered and the rest is reflected on its surface. According to the literature, the translucency of zirconia is related to its additives, grain size, microstructure, thickness and sintering parameters (Arena et al 2019). The scattering is influenced by the following factors: the chemical nature, the crystalline content, the porosity and voids and the quantity and size of the crystals compared to the incident light wavelength (Pekkan et al 2020).

Some studies have reported that crystalline ceramics, which are characterized by an intense scattering effect, have an opaque appearance and are less translucent than glass ceramics (Zhang & Lawn 2018).

In this respect, in order to increase the translucency of zirconia, the pressing procedures at the time of molding should be altered (Chun-Chuan et al 2020). Residual pores should be reduced because they can lead to optical scattering on the surface of zirconia and to a diminishing of the translucency. Residual pores are defined as gaps between the grains, which occur at the time of the molding process (Harada et al 2016).

Casolco et al (2008) have shown that the translucency of zirconia can be improved by significantly limiting the final size of crystals to 55 nm (Casolco et al 2008). This occurrence is owed to the fact that crystals of a smaller size than the wavelength of visible light (400–700 nm) should not significantly hinder the passage of light (Kim et al 2013).

As to the impact of the grain size upon the optical properties, researchers have introduced two concepts. Firstly, they have suggested that the larger grains, which are manufactured through sintering at higher temperature, can lead to the elimination of porosity and the increase in density and translucency.

The second concept regarding the large grain size, is associated with the decreasing translucency and the enhancing light scattering (Kontonasaki et al 2019).

The ceramics with the largest grain size presented the highest translucency, according to the following studies: Ebeid et al (2014), Kim et al (2013), Tuncel et al (2016), Ban (2008), Carraba et al (2017), and Kontonasaki et al (2019).

The study of Zhang & Lawn (2018) concluded that a grain size <100 nm is necessary to produce acceptable transmittance in 3Y-TZP ceramics. They showed that in order to obtain translucency similar to dental porcelains, the mean grain size of 3Y-TZP must be about 82 nm (for 1.3 mm thickness), 77 nm (for 1.5 mm), and 70 nm (for 2 mm).

One of the studies conducted by Zhang & Lawn (2018) has evaluated the influence of the yttrium oxide added to improve the optical properties of zirconia. However, the mechanical properties got reduced, even though the fracture resistance was shown to be higher than the functional masticatory loads (3000 N).

Another recent study (Shiraishi & Watanabe, 2016) has revealed that the alumina content can reduce the scattering light transmission when it is added to zirconia. As to alumina, a source of opacity is added to zirconia in order to prevent the formation of impurities, such as residual pores (Shiraishi & Watanabe, 2016).

4. Mechanical properties

Zirconia (ZrO_2) is characterized by beneficial mechanical properties, such as high flexural strength, fracture resistance, hardness, corrosion resistance, wear and tear in both acidic and basic environment (Iosif et al 2020).

Some studies have shown that the mechanical properties of zirconia ceramics depend not only on the manufacturing process, but they are also influenced by the microstructure (Amat et al 2018). According to the literature, it is well known that a microstructure with fine grain size, an excellent microstructural homogeneity and a fully dense structure offer improved mechanical properties (Denry & Kelly 2008).

In the study of Silva et al (2010), the mechanical properties, microstructure, densification and crystalline phase of the yttria-stabilized zirconia blocks were assessed for dental applications. In this respect, they sintered blocks at 1400 _C (GS _ 250 nm) and microstructured blocks (GS _ 1 _m) at 1600 _C; thus, an increase in flexural strength values (1020 MPa for nano and 855 MPa for micro) was found; also Weibull Modulus (13.1 for nano and 9.8 for micro), and fracture toughness values (11.2 MPa_ m1/2 for nano and 9.0 MPa_m1/2) were reported.

Other studies (Zhang & Lawn 2018; Kontonasaki et al 2019; Maminskas et al 2020) have concluded that the addition of Al_2O_3 brings about an increase in the mechanical properties of zirconia.

5. Wear

The wear of zirconia prostheses and antagonistic enamel has been the subject of numerous studies. Their conclusion was that the wear rates of enamel were minimized when set against a highly polished zirconia surface (Zhang & Lawn 2018).

The wear rate of enamel when in contact with polished zirconia was comparable to that of a resin-based composite but much lower than that of a pressed lithium disilicate glass-ceramic (Sripetchdanond & Leevailoj 2014). Abrasiveness increased from polished, then glazed and stained, adjusted and glazed and lastly porcelain veneered (Lawson et al 2014). Monolithic zirconia crowns are responsible for more enamel wear than the natural teeth but less than porcelain-veneered prostheses. Despite the high hardness of zirconia, antagonistic abrasion of opposing teeth enamel can be kept to a minimum by producing highly smoothed surfaces (Zhang & Lawn, 2018).

6. Biocompatibility of zirconia

The biocompatibility of a material is defined firstly by its compatibility with different tissues and/or organs and implies the absence of any inflammatory, allergic, immune, toxic, mutagenic, or carcinogenic reactions (bioinertia); and secondly by generating the most useful and beneficial cellular or tissue response in a specific situation while enhancing therapeutic performances (bioactivity) (Iosif et al 2020).

In this regard, zirconia provides good biocompatibility; it acts as a chemically inert material and does not present any side effect or general tissue reactions. Moreover, the zirconia material prevents the accumulation of plaque, by creating a favorable surface for gingival tissues (Guatam, 2016).

With regard to the biocompatibility of zirconia, this was evaluated multiple times by using *in vitro* and *in vivo* tests (Manicone et al 2007; Stanford et al 2006; Lohmann et al 2002; Osman & Swain, 2015; Maminskas et al 2020) and by using studies performed on animals such as rabbits, rats, mice, dogs and monkeys (Piconi, 1999).

In their studies, Akagawa et al (1998) and Kohal et al (2004) reported that the zirconia material does not have any side effects on the studied histological tissues. Zirconia powders were also subjected to different cell lines and some authors have shown that ZrO_2 had no cytotoxic effects when fibroblasts were cocultured with it, or with extracts using different methods (Dion et al 1994; Hisbergues et al 2009).

Regarding the comparison with titanium - as a gold standard for dental implants - Degidi et al. (2006) made the initial revolutionary discovery.

In their study they demonstrated that vascular endothelial growth factor expressions and the inflammatory infiltrate occur at a lower level around implants of ZrO_2 than throughout the titanium implant.

Rimondini et al (2002) compared oral bacterial colonization on the surfaces of disks fabricated from machined Ti and Y-TZP. Y-TZP was shown to accumulate fewer bacteria than Ti and it was concluded to be a suitable material for the manufacturing of abutments. In this respect, authors like Zembic et al (2009) conducted a randomized controlled clinical trial comparing zirconia and titanium abutments supported by implants. After three years, both materials showed survival rates of 100%. The biological outcomes of zirconia showed a significant reduction in bacterial adhesion when compared to titanium.

More recently, Guatam et al (2016) have demonstrated that the Y-TZP material presents fewer bacteria accumulations around its surface than titanium does, which can be explained by their different protein adsorption properties. Thus, according to the latest literature, (Osman &Swain, 2015; Maminskas et al 2020) it is well-known nowadays that ZrO_2 is an appropriate material for manufacturing implant abutments with low bacterial colonization potential.

Manufacturing procedures

The development of new manufacturing techniques using computer-aided design/manufacturing (CAD/CAM) technologies, has led to the introduction of metal-free restorations, such as all-ceramic systems (Manicone et al 2007).

In this respect, zirconia is processed by CAD-CAM milling, this being done according to two different production techniques, namely: soft machining of pre-sintered zirconia and respectively hard machining of fully sintered zirconia. These manufacturing procedures can be performed in dental offices or laboratories.

1. Soft machining

Since its development in 2001, direct ceramic machining of pre-sintered 3Y-TZP has become increasingly attractive in dental medicine and is now provided by a growing number of producers (Filser et al 2003; Denry &Kelly 2008).

The wax pattern is scanned and the prosthetic restoration is designed by computer software (CAD). Subsequently, a pre-sintered

ceramic blank is milled by computer-aided machining and then the zirconia restoration is sintered at high temperature.
Today, soft machining stands for the most used manufacturing technique. As previously stated, the method is based on the milling of pre-sintered zirconia blanks fabricated by cold-isostatic pressing a mixture of zirconia powder, stabilizing oxides and binding agents (the latter being eliminated during the pre-sintering technique). With this procedure, zirconia acts as a highly homogenous material that is easier to mill, thus reducing production times, machinery wear and surface flaws. Moreover, soft machining generates negligible internal porosities (about 20–30 nm) (Miyazaki et al 2013, Camposilvan et al 2018). This process requires an oversizing of 25%-30% in the framework to be milled. Following this procedure, a shrinkage in the framework volume occurs. Although the milling procedure is an easy one, soft machining requires a precise matching between CAD oversizing and the shrinking material in order for size inexactness not to occur
(Denry & Kelly, 2008. Kontonasaki et al 2019).

2. Hard machining
The procedure requires milling of fully sintered zirconia blanks mainly produced with hot isostatic pressing (HIP) at 1400°-1500°C (Chen et al 2016; Denry & Kelly 2008).
This method cancels the problem of post-milling shrinkage, since neither oversizing nor sintering are necessary. However, hard machining requires longer milling periods and more complex manufacturing, concerning higher financial efforts due to accelerated wear of production machinery and growing risks of attrition flaws. Furthermore, right after hard machining, zirconia frameworks may be exposed to the risk of a certain amount of monoclinic transformation phase due to mechanical stress, working burs friction and overheating subsequent to the machining of the hard material (Zarone et al 2019).Thus, literature data still argues in regard to which method is the most favorable; the choice mainly depends on the operator's preference and knowledge, as exposed in the previous considerations (Kontonasaki et al 2019; Camposilvan et al 2018, Miyazaki et al 2013, Denry & Kelly, 2008; Sundh et al 2005; Kosmac, 1999).

Conclusions

This article reports the development of current and forthcoming high-performing dental zirconia ceramics with esthetic qualities. Due to the excellent mechanical properties of zirconia and superior biocompatibility, zirconia-based restorations are a suitable prosthodontic alternative to metal-based restorations. The future of zirconia or any other restorative ceramic is based on fundamental improvements in the scientific research on materials; this is to be followed by the development made by entrepreneurial dental manufacturers and the implementation by skilled clinical practitioners. The challenge consists in enhancing the esthetic value of prosthetic restorations, while maintaining the high intrinsic mechanical properties, such as high strength and fracture resistance. Therefore, further research is required in the fields of ageing, veneering, framework design, surface alteration and esthetic requirements in order to further evade possible compromises and accelerate expected clinical results.
In order to satisfy the increased demand of the patients we have to choose a material which provides a good biocompatibility, highly esthetic areas and good functionality. The monolithic zirconia can be used for high load bearing area while the layered zirconia can be used mainly for high esthetic area.
However, when planning a treatment, the practitioner has to take the following factors into account: the esthetic issue, the occlusion, the restoration area, the status of the tooth and the parafunctional habits of the patient.

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Authors

•Ana Ispas, Department of Prosthodontics, Faculty of Dental Medicine, "Iuliu Hatieganu" University of Medicine and Pharmacy, 32 Clinicilor Street, 400006, Cluj-Napoca, Cluj, Romania; email: ispas.ana@umfcluj.ro

•Laura Iosif, Department of Prosthodontics, Faculty of Dental Medicine "Carol Davila" University of Medicine and Pharmacy, 17-23 Calea Plevnei Street, Sector 5, Bucharest, Romania; email: laura.iosif@umfcd.ro.

•Cătalina Murariu-Magureanu, Department of Prosthodontics, Faculty of Dental Medicine "Carol Davila" University of Medicine and Pharmacy, 17-23 Calea Plevnei Street, Sector 5, Bucharest, Romania; email: dr_catalina_magureanu@petralaboratory. ro

•Mariana Constantiniuc, Department of Prosthodontics, Faculty of Dental Medicine, "Iuliu Hatieganu" University of Medicine and Pharmacy, 32 Clinicilor Street, 400006, Cluj-Napoca, Cluj,Romania; email: mconstantiniuc@umfcluj.ro

•Antarinia Craciun, Department of Prosthodontics, Faculty of Dental Medicine, "Iuliu Hatieganu" University of Medicine and Pharmacy, 32 Clinicilor Street, 400006, Cluj-Napoca, Cluj,Romania; email: mconstantiniuc@umfcluj.ro

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