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CALCIUM-MAGNESIUM PHOSPHO-SILICATE BIOCERAMIC CEMENTS FOR DENTISTRY AND ORTHOPEDICS

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The novel Calcium Phosphate Silicate Cement (CPSC) combines the best biological and structural properties of Calcium Phosphate Cement (CPC) and of Calcium Silicate Cement (CSC). The resulting CPSC is relatively stronger than CPC and has extended and controlled degradability time range, as compared with CPC. The current FDA-approved applications of CPSC include dental root sealers and root repair materials. The materials are distributed worldwide by Vancouver BC Canada company Innovative BioCeramix Inc (IBC), and through the associated network of distributors including Brasseler USA Inc. and Henry Schein Inc.

One area of active CPSC research for orthopedics concerns its setting time, as the currently known CPSC variants set within ~1 hr after water contact. There is a desire to shorten this time to < 10 min for certain dental and orthopaedic applications. One approach includes admixture of biocompatible/biodegradable and fast-setting Mg compounds to CPSC, rendering it now Calcium-Magnesium Phosphate Silicate Cement (CMPSC).

This work reviews our research progress on these novel, faster-setting bio-cements, including both CPSC and the effects of Ca/Mg on the properties of CMPSC after hydration (setting) at 37°C for various length of time, and its final properties (variation of pH, compressive and 3-point bending strength, in vitro bioactivity). The phase transformations in CMPSC during setting show that calcium hydroxide, produced during the hydration of calcium silicates, reacts with the phosphate additives to form hydroxyapatite. The in-situ formation of a nanocomposite from the hydroxyapatite and calcium silicate hydrates appears responsible for the significant enhancement in CMPSC strength, bioactivity, and biocompatibility, as compared to pure CSC.

Keywords: CMPSC, Orthopedics, Setting time, Mechanical properties, Bio-cements

BIOMIMETIC TRENDS IN MODERN BIOCERAMICS: 3D PRINTING AND HYBRID COMPOSITION

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In nature, material grows drop by drop. The shape of bones, muscles and organs do not form in one step, like casting or forging. Natural soft and hard tissues grow layer by layer or segment by segment. The same is true for additive manufacturing. The finally geometry is formed by a multitude of single layer sintered together. There is no material waste, but the option to print porous structures with much freedom of design. For that reason, additive manufacturing is very close to natural growing processes of tissue. It really is a biomimetic process. In the presentation, suitable additive manufacturing technologies for bioceramic are introduced.

The presentation mention recent advances in 3D printed bioceramic, i.e., for bone tissue engineering scaffolds along with current challenges and future perspectives. Some of these inorganic scaffolds are biodegradable and have proven ideal for bone tissue engineering, sometimes even with site specific growth factor/drug delivery abilities. Other bioceramic parts should be implanted for longer time without strong degradation. The challenge here is the improvement of their low

strength. Here hybrid compositions by incorporation of reinforcements are potential options. Some aspects of improving fibre-matrix compatibility are introduced as well.

Keywords: Bioceramics, 3D-printing, Inorganic scaffolds, Mechanical properties,

CERAMIC IMPLANT MATERIALS: PREPARATION, STRUCTURE AND PROPERTIES

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There are more than 400 000 artificial hip joint operations made every year in the word and there are some 25 000 000 people who have either a partial or a total hip replacement. It has been estimated that the aged people population has increased tremendously in recent past and there will be seven times increase. Because the human body is at the same time both a very hostile and sensitive environment for foreign objects, the life span of a hip implant is limited. With time, the wear and risk of the implant loosening increases so that after 10 years 10%-20% of the implants have to be renewed. The materials used for artificial implants by default are Titanium ($TiAl_6V_4$, Ti alloy), CoCrMo and ceramics. The ultimate material with properties of native joints has not been found to date despite application of different biocompatible materials. Biomaterials used for implants should possess some important properties in order to provide the long-term usage in the body without rejection. Materials used as different biomaterials should be made with certain properties as excellent biocompatibility, superior corrosion resistance in the body environment, excellent combination of high strength and low modulus, high ductility and no toxicity. This new line research is focused on examination of ceramic based nanocomposites. The three different types of bioceramics: TiC/a:C thin films, hydroxyapatite based coating on TiC/a:C thin film and hydroxyapatite composite, are developed from the same based material. The sputtered TiC/a:C thin films are developed as an optimal solution for protective applications. The formation of TiC based surface coating has a passivation effect to titanium implant and Ti ions will be kept in the bulk implant introduced in the living organism. The nanosized hydroxyapatite coating on TiC thin films will help the quick and inflammation-free osiffication. The other main goal of this research is to develop a new type of coating onto medical implants, which simultaneously possesses antibacterial and biocompatible properties. Structure and other properties of bioimplants will be showed.

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Keywords: Biomaterials, TiC/a:C, Titanium implant, Thin film, Nanosized hydroxyapatite

BIOCERAMIC BONE CEMENTS

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Bone fracture and damage result in more than 2 million surgical procedures every year, and this number is predicted to increase. The population is aging and a large number of people receive implants to repair, replace or augment diseased or damaged bones. Major injuries can be treated in three ways: (i) by replacing lost tissues with prosthetic devices based on artificial implant materials, (ii) by replacing with grafts (auto-, allo- or xenografts) or (iii) by regenerative approaches, where bioactive scaffolds with cells are used as hybrid materials (constructs) to generate tissues *de novo*.

Ceramic scaffolds on the basis of calcium phosphates (CaPs) have been widely applied as orthopedic and dental implants. Calcium phosphates meet a number of requirements for bone substitutes. Due to similarity in chemical and mineral composition to natural bone, CaPs exhibits high biocompatibility and bioactivity. Clinical investigations indicate also that hydroxyapatite (HA) and α-tricalcium phosphate (α-TCP) are osteoconductive materials [1]. The major disadvantage of current bioceramic bone substitutes is that they exist in the form requiring the surgical intervention to fit the surgical site around the implant or to carve the graft to the desired shape. The need for minimal invasive surgery has induced the development of self-setting bioceramic bone cements, which may be applied as injectable and/or mouldable bone substitutes [2,

3]. Calcium phosphate bone cements (CPCs) are produced by a chemical reaction between two phases: solid, consisting of calcium orthophosphates, and liquid. When cement components are mixed together they form a shapeable paste which can conform to osseous defects and progressively sets and hardens *in situ* into a solid mass.

Currently, there is a constant search for the materials that would exhibit a whole set of properties necessary to be simultaneously completely biocompatible and biofunctional. Despite numerous CPCs formulations, there are only two possible final products for the CPCs setting reaction: brushite (dicalcium phosphate dihydrate, DCPD) and apatite (HA or CDHA) [4]. In our research group apatite bone cements on the basis of α-TCP as well as composites on the basis of calcium sulphate and modified hydroxyapatite have been investigated. Furthermore, various inorganic and organic additives, such as chitosan, sodium alginate, methylcellulose, have been added to improve material characteristics. A number of studies regarding setting time (Gilmore Needles), rheological behaviour (Physica MCR 3010 rheometer), phase composition (XRD, XRF, FT-IR, Raman), microstructure (SEM, AFM, MIP) and biocompatibility (*in vitro* tests in SBF, cell cultures) have been conducted. The effect of composition on the setting process as well as mechanical and biological properties of final materials have been tested. It was found that calcium phosphate cements are a family of promising bone grafting materials and may constitute an alternative for traditional sintered bioceramics due to their biocompatibility, good adaptation to shape and size of bone defect and excellent handling.

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Keywords: α-TCP, Biological properties, Bone cements, Mechanical properties, Setting

MICROPATTERNING OF CALCIUM PHOSPHATE BIOCERAMICS, BY FEMTOSECOND PULSED LASER, FOR BONE TISSUE ENGINEERING APPLICATION

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The bioactivity of synthetic bone implants is highly impacted by their surface topography, especially by the presence of micro-patterns likely to generate cells growth guidance. In this study, laser machining technology was employed in order to obtain controlled regular micro-patterns on dense calcium phosphate surfaces, without any contamination. In literature, this kind of laser patterning is usually performed on metallic alloys, but there are few works dealing with calcium phosphate ceramics because of unwanted phase transformations induced by the thermal impact of such a process. In our work, a femtosecond pulsed laser was used in order to limit this thermal impact. Substrates with perfectly controlled micropatterning and without any secondary phase were obtained by optimization of the process parameters (laser power, scanning speed, pulse frequency). The microstructural characteristics were investigated by microscopy (optical, confocal, scanning electron) and the phase analyses were verified by XRD. This work allowed us to highlight the effects of the process parameters on the patterning. The high benefits of the laser treatment on wettability was shown by contact angle assays. Relationships between surface topography and wetability mechanisms were established thanks to a wide variety of micropatterned designs, allowed by the precision and the a ccurate control of the laser process. This technique seems to provide an interesting alternative to conventional surface treatments of calcium phosphates. In vitro experiments are currently being performed to demonstrate the influence of the micro-patterns on the cell behavior.

Keywords: Bioceramics, Calcium phosphate, Laser machining, Micropatterning, Wettability

A COMPARATIVE STUDY OF CHEMICALLY BONDED CALCIUM PHOSPHATE BASED BIOCERAMICS

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The Calcium phosphate bone cements (CPCs) belong to the group of biocompatible chemically bonded materials with unique properties for bone regeneration applications. CPCs are produced using one or more calcium phosphates which upon mixing with liquid phase form pastes that are able to set and hardened *in situ* (Fig. 1). As CPCs solid components hydroxyapatite (HA, $Ca_{10}(PO_4)_6(OH)_2$) and α-tricalcium phosphate (α-TCP, α-$Ca_3(PO_4)_2$) are of particular interest.

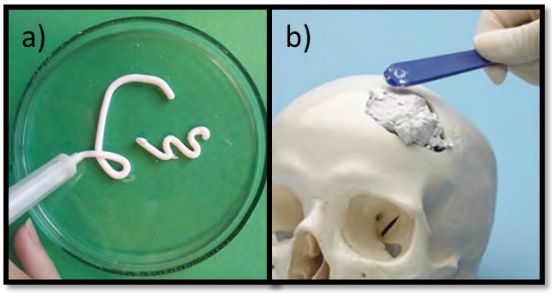

Fig.1. Chemically bonded bioceramics a) injectable, b) non-injectable.

In our study chemically bonded bioceramics on the basis of α-TCP, calcium sulphate, hydroxyapatite as well as chitosan and methylcellulose has been developed. The influence of CPCs initial composition on setting times (Gilmore Needles), phase composition (X-Ray Diffraction), microstructure (Scanning Electron Microscopy) and porosity (Mercury Intrusion Porosimetry) of the final cement bodies was investigated. Furthermore, cytocompatibility of the obtained biomaterials was tested using human bone-derived cells (hBDC).

It has been found that the composition of bone cements influenced both physicochemical and biological properties of biomaterials. The most promising results have been obtained in the case of CPCs consisting of α-tricalcium phosphate. The α-TCP- based bioceramics exhibited appropriate physicochemical properties, excellent handling and good biocompatibility.

Keywords: α-TCP, Bioceramics, Bone cements, Calcium sulphate, Hydroxyapatite

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SYNTHESIS METHOD AFFECTS THE STRUCTURE OF BIOACTIVE GLASSES – FTIR, RAMAN, NMR AND XPS INVESTIGATION

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Bioglasses are able to bond to bone through formation of carbonated hydroxyapatite in body fluids. Bioactivity is influenced by the glass composition and structure but also by the synthesis route: melt-quenching versus sol-gel. The recognition of the bioglass structure can allow one to determine the relationship between chemical composition, molecular structure and bioglass properties, *i.e.*, dissolution rate, bioactivity and biological response. Therefore, the present work aims to prepare bioglasses with the same composition by using the two methods and study its effect on glass structure.

The two most extensively studied bioglass systems: SiO_2-CaO-Na_2O-P_2O_5 (45S5 glass) and alkali-free SiO_2-CaO-P_2O_5 (A2 glass) were obtained using the low-temperature sol-gel technique and the traditional melt-quenching method. The A2 and 45S5 gel-derived glasses were prepared in different media, namely, alcohol-based and water-based, respectively. The gel-derived and melt-derived glasses were milled and sieved to obtain bioglass powders with a particle size below 45 μm. The glasses were investigated in terms of their structure using X-ray diffraction, FTIR, Raman, ^{29}Si and ^{31}P MAS-NMR, and XPS spectroscopy.

Melt-quenched materials demonstrate the amorphous glass structure, while gel-derived ones show the beginning of crystallisation of the following crystalline phases: $Na_2Ca_2Si_3O_9$ (combeite) and $Na_2Ca_4(PO_4)_2SiO_4$ (silicorhenanite) – 45S5 glass, and Ca_2SiO_4 and $Ca_5(PO_4)_3OH$ (hydroxyapatite) – A2 glass. Both 45S5_melt and 45S5_gel glasses mainly contain Q^2 Si units, while A2 glasses vary in terms of silicon entities (Q^n) as follows: A2_melt – Q^2 (major) and Q^3 (minor), A2_gel – Q^3 (minor) and Q^4 (major) units. This indicates that the polymerisation of the glass structure increases in the following order: 45S5_melt ≈ 45S5_gel < A2_melt A2_gel. For both 45S5 glasses, Q^2 Si units are associated with both Ca^{2+} and Na^+ modifiers. NMR and Raman spectroscopy indicate the presence of both Q^0 (major) and Q^1 P (minor) entities and the absence of Si-O-P bonds in resulting glasses. This confirms that phosphorus acts as a glass network modifier rather than a network former. The relative ratio of Q^0/Q^1 species depends on the synthesis method and also the chemical composition of glass. These results clearly show that the synthesis route affect the structure of bioactive glasses of both ternary and quaternary systems. Further investigations are needed to confirm the real influence of the synthesis route on the glass dissolution and bioactivity.

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Keywords: Bioglasses, MAS-NMR spectroscopy, Raman spectroscopy, Sol-gel, Melt-quenching

CuO DOPED GEL-DERIVED BIOGLASSES WITH POTENTIAL OSTEOGENIC, ANGIOGENIC AND ANTIBACTERIAL PROPERTIES

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Bioactive glasses are recognized to belong to the most important biomaterials due to their high biocompatibility, bone-bonding ability and also positive biological effects after implantation. However, for better regeneration of large bone defects, bioactive glasses should possess not only osteoconductivity but also the ability to stimulate both osteogenesis (for promoting new bone formation) and angiogenesis (for inducing vascularization). In addition, antibacterial activity to prevent postoperative bacterial infection is also desired. For these reasons, bioactive glasses are doped with different inorganic ions with known therapeutic action. In particular, Cu^{+2} ions can be considered as good candidates for the development of doped bioactive glasses due to their osteogenic, angiogenic and antibacterial characteristics.

The effect of substitution of CuO for CaO on structure and bioactive properties was investigated in two sol-gel glasses with different chemical compositions: A2Cu – (54-*x*)CaO-*x*CuO-6P_2O_5-40SiO_2 and S2Cu – (16-*x*)CaO-*x*CuO-4P_2O_5-80SiO_2 (*x* = 0, 1, 3 and 5 by mol.%) stabilized at 700 °C. The structure of resulting glasses was studied using FTIR spectroscopy and X-ray diffractometry. Furthermore, bioactive properties of materials was assessed using SEM/EDX and FTIR methods after 7-day immersion in simulated body fluid (SBF).

The results show that the effect of copper substitution on the structure and crystallization ability of glass strongly depends on SiO_2/CaO molar ratio in the glass composition. XRD and FTIR studies indicate that a gradual increase of CuO content in both A2 and S2 glasses results in the enhancement of crystallization process. The presence of 3-5 mol.% of copper ions in calcium-rich glass (A2 group) leads to expansion and disruption of the silicon-oxygen network. Whereas, the addition of Cu to the silica-rich glass (S2 group) does not significantly change the polymerisation of glass network. The *in vitro* bioactivity test showed that with the increasing CuO content in both group of materials (A2 and S2) the apatite-forming ability remained at the same high level as for the unmodified A2 and S2 glasses.

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Keywords: Bioglasses, Bioactive properties, FTIR spectroscopy, CuO, Structure, XRD

STRUCTURAL INVESTIGATION ON THE GEL-DERIVED BIOGLASSES FROM THE SiO_2-CAO AND THE SiO_2-CaO-P_2O_5 SYSTEMS

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Gel derived glasses from the binary SiO_2-CaO and ternary SiO_2-CaO-P_2O_5 systems were obtained. Materials differed in the CaO/SiO_2 molar ratio which varied between 0.2 to 1.5. Starting materials in the sol-gel synthesis process were TEOS, TEP and calcium nitrate tetrahydrate. The materials were stabilized at 600 °C (binary glasses) and 700 °C (ternary glasses). For all of the materials, the network connectivity parameter and optical basicity were calculated.

XRD patterns were measured in order to investigate the phase composition of obtained powders. The beginning of crystalization was observed as the CaO content increased. There have been FTIR analysis performed in order to characterize the structure of materials. FTIR spectra showed changes in the materials structure depending on the CaO/SiO_2 ratio. It has been proven that with the increase of CaO/SiO_2 ratio the number of bridging oxygens significantly decreased.

The local structure of silicon and phosphorus (the ternary glass system) in the obtained materials were examined with magic angle spinning nuclear resonance (MAS-NMR). The ^{29}Si MAS-NMR spectra revealed that silicon was present in Q^2, Q^3 and Q^4 structural units. Moreover, ^{31}P MAS-NMR indicated that phosphorus existed mainly as a monophosphate complex. It has been also shown that the presence of phosphorus in the structure inducted the process of silica network repolimeryzation.

Bioactive properties of the powders were tested through their immersion in the simulated body fluid solution (SBF) for 7 days. Changes in the powders structure after the incubation were characterized with FTIR spectroscopy and XRD. Considerable changes in the structure have been indicated, confirming the appearance of apatite after immersion in the SBF.

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Keywords: Bioglasses, Bioactive properties, Gel derived glasses, MAS-NMR spectroscopy, Structure

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EFFECT OF THE BIOACTIVE GLASS CHEMICAL COMPOSITION ON THE BIOACTIVITY AND DEGRADATION RATE OF POLYMER-CERAMICS COMPOSITES

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Bioglasses are ceramics materials that are known for the ability of creation of a strong bond with bone tissue. They can also significantly improve biological performance of materials. Incorporation of bioglass particles into a polymer matrix enables to obtain composite materials with new deserved properties. The aim of this study was to describe the influence of chemical composition of gel bioglasses on *in vitro* bioactivity and degradation of polymer-ceramics composite materials.

There were several gel-derived bioglasses from the SiO_2-CaO and SiO_2-CaO-P_2O_5 systems produced in the form of powders, differing in the molar ratio of CaO/SiO_2. The bioglass powders were incorporated into the PLGA polymer matrix in order to obtain 2D composite films. *In vitro* bioactivity, described as the ability of forming apatite layer at the materials surface in contact with incubating solution, was examined in three different immersion solutions: simulated body fluid (SBF), phosphate-buffered saline (PBS), and cell culture medium. Changes in the ions concentrations in the immersion solutions were quantified with ICP. The study of degradation was performed in the PBS solution for 4 months.

SEM and EDX analyses confirmed the presence of carbonate apatite on surfaces of all of the materials. Moreover, the SEM analysis indicated differences in morphologies of the apatite depending on the type of incubating solution and the chemical composition of the materials. The HCA layer formation was confirmed by FTIR spectroscopy. Changes in the ions concentration in the immersion solutions depended also on the glass compositions. The degradation rate increased significantly after the incorporation of the bioglass particles into the polymer matrix.

Our study confirmed that the chemical composition of bioglasses affects the process of bioactivity and the degradation rate of composite polymer-ceramics films. We proved that it is possible to modulate the chemical properties of that kind of composites depending on requirements of an application.

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Keywords: Gel bioglasses, Bioactive properties, Composite film, Degradation, PLGA

WPŁYW JONÓW ANTYBAKTERYJNYCH NA WŁAŚCIWOŚCI TERMICZNE SZKIEŁ KRZEMIANOWO-FOSFORANOWYCH

INFLUENCE OF ANTIBACTERIAL IONS ON THERMAL PROPERTIES OF SILICON-PHOSPHATE GLASSES

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Celem badań było otrzymanie szkieł krzemianowo-fosforanowych z układu $NaCaPO_4$-SiO_2, zawierających różne jony antybakteryjne, i porównanie ich właściwości termicznych. Szkła z badanego układu należą do jednych z bardziej atrakcyjnych biomateriałów ze względu na ich zdolność do tworzenia wiązania chemicznego z żywą tkanką poprzez wytwarzanie na ich powierzchni warstwy hydroksyapatytu w kontakcie z płynami fizjologicznymi. Podczas implantacji istnieje ryzyko kolonizacji bakterii na powierzchni implantu, która może doprowadzić do infekcji w organizmie, a w rezultacie do zniszczenia lub odrzucenia implantu przez organizm. Zatem istnieje potrzeba opracowania bioaktywnych materiałów pozwalających zapobiegać wyżej wspomnianym infekcjom bakteryjnym. Bioaktywne szkła i materiały szkło-krystaliczne, zawierające dodatek odpowiednich jonów antybakteryjnych, mogą być drogą do rozwiązania tych problemów.

W niniejszej pracy porównano wpływ wybranych jonów bakteriobójczych (miedzi, cynku i ceru) na budowę strukturalną i właściwości termiczne likwacyjnych szkieł krzemianowo-fosforanowych.

Słowa kluczowe: jony antybakteryjne, hydroksyapatyt, struktura, szkło krzemianowo-fosforanowe, właściwości termiczne

CHARAKTERYSTYKA SZKIEŁ POCHODZENIA ŻELOWEGO Z UKŁADU BINARNEGO CaO-SiO_2 ZAWIERAJĄCYCH JONY MIEDZI

CHARACTERIZATION OF SOL-GEL DERIVED GLASSES FROM BINARY SYSTEM CaO-SiO_2 CONTAINING COPPER IONS

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Współczesne badania wykazują, że nie tylko szkła zawierające jony fosforu wykazują właściwości bioaktywne, ale także szkła z prostych układów binarnych (np. CaO-SiO_2 oraz Na_2O-SiO_2), a nawet czysta krzemionka, otrzymywane za pomocą syntezy zol-żel, mogą również wykazywać te własności. Materiały te zyskują wyższą bioaktywność ze względu na nanoporowatość i wynikające z tego rozwinięcie powierzchni. Jest to korzystne z punktu widzenia przerastania porowatego materiału tkankami - wiązanie biologiczne tkanki z implantem. Ponadto w strukturze takich szkieł obecna jest znaczna ilość grup OH^-, a także jonów H^+, odgrywających rolę modyfikatorów więźby szkła. Zwiększona liczba zerwanych mostków krzemo-tlenowych w szkłach otrzymywanych metodą zol-żel powoduje szybsze ich rozpuszczanie się, co znacząco przyspiesza powstawanie biomimetycznego HCA, a w konsekwencji zwiększa ich bioaktywność.

Celem badań była ocena własności szkieł z układu CaO-SiO_2 domieszkowanych jonami miedzi, otrzymanych za pomocą syntezy zol-żel. W tym celu wykonano badania spektroskopowe, termiczne oraz testy bioaktywności otrzymanych materiałów.

Słowa kluczowe: CaO-SiO_2, szkło bioaktywne, jon miedzi, właściwości termiczne, zol-żel

PROCESSING AND PROPERTIES OF C-TZP NANOCERAMICS FOR BIOMEDICAL APPLICATIONS

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Despite the excellent biocompatibility and great mechanical properties of 3Y-TZP ceramics, susceptibility to low temperature degradation poses a risk to medical applications. The degradation process occurs in a humid environment and involves the tetragonal to monoclinic transformation, leading to microcracking and destruction of the material. Many efforts are carried out to manufacture tetragonal zirconia ceramics being resistant to aging.

The present work reports a simple and efficient method for preparation of calcia-zirconia nanopowder of a controlled chemical composition which is sinterable at low temperatures to fully-dense, fully-tetragonal Ca-TZP ceramics with nano-sized grains and improved aging resistance. A physical mixture of zirconia gel and calcium hydroxide was the precursor of zirconia nanopowder partially stabilized with 4 mol.% calcium oxide. The mixture was calcined at 500 °C for 1 h in air. The 4Ca-TZP sintered bodies with a 100% of the tetragonal polymorph, a relative density above 99%, and an average grain size of 112.4 nm were manufactured by pressureless sintering for 2 h at 1200 °C in air. The sintered material showed a bending strength and fracture toughness of about 730 MPa and 14,5-20 $MPa \cdot m^{0,5}$, respectively. After a five-hour accelerated aging test in water vapour at 134 °C, changes in the phase composition and flexural strength were not observed in the studied material. The 4Ca-TZP nanoceramics showed improved resistance to the low-temperature degradation and may be used as a suitable replacement for the Y-TZP materials in biomedical applications.

Keywords: Ca-TZP, Low temperature degradation, Mechanical properties, Nanoceramics, Phase composition