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Production and Study of Innovative Biomedical Materials Based on Bone Ash

MARINE SHAVLAKADZE[1], TEIMURAZ CHEISHVILI[2]
[1]University of Georgia, [2]Georgian Technical University
Mar.shavlakadze@ug.edu.ge

Abstract

The aim of the research was to obtain bioactive glass based on traditional glass-making technology using bone ash as the primary raw material, due to its complex content of P_2O_5 and CaO. It was established that by using rock crystal, chalk, soda, and bone ash as raw materials, it is possible to synthesize bioactive glass with a 45S5 composition at a temperature of 1340 ± 20°C. The resulting glasses tend to crystallize at 800–1000°C, forming a glass-crystalline material characterized by the emergence of $Na_2Ca_2(SiO_3)$ crystalline phases and a bioactive matrix glass enriched with P_2O_5.

The bioactive glass and glass-crystalline material obtained through this innovative methodology and raw material base demonstrated characteristic properties (compressive strength, chemical stability, density, porosity) in compliance with the requirements of ISO 13175-3:2015 standard. The economic efficiency of the resulting biomaterials, their simplified production technology, and their performance characteristics may be considered determining factors for the feasibility of their production and application.

KEYWORDS: bioactive glass, bone ash, synthesis, properties.

Introduction

Human social well-being and health are highly dependent on advances in medicine. At the same time, in several key areas of medicine, particularly in maxillofacial surgery and dentistry there is an increasing demand for biomaterials capable of replacing and regenerating bone tissue. Among such functionally significant materials, bio-ceramics and bioactive glass stand out for their reliability and effectiveness, and are already widely applied in practice [1, 2].

Currently, two types of crystalline ceramic biomaterials are widely used: tricalcium orthophosphate ($Ca_3(PO_4)_2$) and hydroxyapatite ($Ca_5(PO_4)_3(OH)$), for which standards have been established [3]. By nature, amorphous bioactive glass is characterized by a high affinity for bone tissue in living organisms. It promotes the activation of reactions and processes responsible for the regeneration of new tissue and the effective repair of bone defects [4].

The capability is determined by the composition of bioactive glass (basic compositions are obtained within the Na_2O–CaO–SiO_2–P_2O_5 system) and its slow solubility. These characteristics facilitate processes that, *in vivo*, lead to the formation of an active nanocrystalline hydroxyapatite layer on the glass surface. This ensures a strong bilateral connection of the bioglass or artificial implant not only with the bone tissue but also with soft tissues.

The technology for producing bioceramics and bioglass is similar at the initial stage and is simultaneously complex. Their similarity lies in the requirement to use the sol-gel method. For the selected raw materials, it is necessary to use water-soluble compounds (mostly artificially derived), such as calcium hydrophosphates, lime milk, hydrosilicates, orthophosphoric acid, nitrates of alkali oxides, etc., while strictly maintaining pH values and temperature within the reaction zone. In the case of bioceramics, the established technology yields two types of products: hydroxyapatite and tricalcium phosphate. In contrast, for bioglass, depending on the glass composition, a compound slurry is formed. The subsequent operations involve decantation and removal of excess liquid, drying of the materials, and finally, thermal processing: sintering for bioceramics and melting for bioglass.

Our research aimed to obtain a widely recognized and traditionally used bioglass (45S5 Bioglass) through an innovative approach. This consisted of producing the initial “dry batch” using raw materials typically used in glass technology (e.g., quartz sand/mountain crystal, calcium carbonate, carbonates of alkali oxides). Additionally, a complex phosphorus containing material cattle bone ash was purposefully incorporated. Using this innovative method to obtain bioglass would significantly simplify and reduce the cost of the technological process, thereby increasing the accessibility of this type of biomaterial.

Research Methods

The widely applied bioglass with 45S5 Bioglass composition in medical practice was obtained from a corresponding “dry batch” formulation and synthesized at high temperatures (1340 ± 20°C) in corundum crucibles, with a holding time of 2 hours at peak temperature. The resulting melt was processed either by casting into molds followed by annealing (620 ± 20°C) or by water quenching (fritting). Thermal treatment of the glass to induce crystallization (800–1000°C) was carried out in a chamber-type electrically heated muffle furnace.

The density of the obtained biomaterial was determined using the hydrostatic weighing method, while water absorption-based porosity was evaluated by measuring the relative (%) mass change between water-saturated and dry samples. Chemical stability was assessed based on the mass loss (%) of the powdered glass material after treatment in physiological solution (37 ± 1°C) for varying durations.

The types of crystalline phases formed within the thermally treated bioglass were identified via X-ray phase analysis using a DPOH-3M diffractometer.

Results

The selected object of study was glass with the composition of *45S5 Bioglass*, characterized by the following chemical composition (wt.%): 45 SiO_2, 24.5 CaO, 24.5 Na_2O, and 6.0 P_2O_5 [5]. To satisfy the required amount of silicon dioxide, mountain crystal was used (containing 99.97% SiO_2). The Na_2O content was supplied using sodium carbonate (58.4% Na_2O and 41.6% volatile components). The CaO content from chalk was 55.8% (the remaining 44.2% represented by LOI). As for phosphorus anhydride, its full amount was provided by bone ash, which, based on chemical analysis, contained the following oxides (wt.%): 40.3 P_2O_5, 53.2 CaO, 2.8 MgO, 0.4 SiO_2, and 0.4 (Al_2O_3 + Fe_2O_3), while the volatile components were 0.3%.

The bone ash was obtained from deproteinized cattle bone using laboratory crushing and grinding. Based on its composition, bone ash represents a material of complex formulation containing the two main oxides present in bioglass P_2O_5 and CaO. Importantly, its composition also includes oxides containing essential elements (Mg and Fe), namely 2.8% MgO and 0.2% Fe_2O_3, which effectively "enhance" the properties of the glass synthesized using the innovative raw material base. In addition to the chemical analysis, the mineral composition of the bone ash was investigated using X-ray analysis. It was determined that, in its initial state, the bone ash predominantly contains hydroxyapatite.

A batch mix was prepared and used to synthesize the bioglass using the selected four raw material ingredients. Trial samples were produced from the melt and their characteristic properties were tested; these are presented in Table 1.

It is known that forced crystallization of glass may alter its properties, as crystalline phases may form from specific constituent oxides within the resulting glass–ceramic material. This may lead to changes in the composition and, consequently, in the properties of the glassy matrix. Based on this principle, glass synthesized from the batch containing bone ash and the other raw materials was subjected to forced crystallization at 850°C (identified through the study of the glass crystallization process), corresponding to the crystallization peak.

According to X-ray analysis, bioglass ceramic material was obtained, in which $Na_2Ca_2(SiO_3)_3$ crystalline phases were identified. The characteristic properties of the crystallized glass are also presented in Table 1.

Table 1. Characteristic properties of bioglass (BG) and bioglass ceramic material (BGT) obtained using the new raw material base

NO.	TEST PROPERTY	UNIT	VALUES OF THE CHARACTERISTIC PROPERTIES OF BIOGLASS (BG) AND BIOGLASS–CERAMIC MATERIAL (BGT)	
			BG	BGT
1	Compressive strength	MPa	510	470
2	Chemical stability	%	1.6	1.7
3	Density	kg/m³	2660	2680
4	Water absorption	%	0.05	0.06

Discussion

The study of the biomaterial properties obtained using the new raw material base both amorphous (initial bioglass) and amorphous–crystalline (forced crystallization) revealed that their characteristic/test properties comply with the requirements regulated by the ISO 13175-3:2015 standard for biomaterials. However, although both materials were produced from melts of identical composition, the properties of the bioglass and the subsequently crystallized glass–ceramic material showed slight differences.

This variation may be attributed to the internal structural rearrangement, namely the transition from the disordered structure of the amorphous glass to a more ordered configuration in the glass–ceramic material, containing crystalline inclusions. This structural transformation could have induced internal rearrangements, leading to changes in material properties. Specifically, crystallization resulted in a slight increase in density but a decrease in compressive strength. Additionally, due to expected surface microcracking, minor increases in chemical reactivity and water absorption (open porosity) were observed.

A noteworthy and significant factor is the formation of $Na_2Ca_2(SiO_3)_3$ crystalline phases in the glass–ceramic material. As a result of crystallization involving Na_2O, CaO, and SiO_2, the residual matrix glass becomes enriched with P_2O_5, which is known to enhance bioactivity. Consequently, the matrix of the crystallized material can be considered to exhibit superior bioactive properties.

Conclusion

The innovative production of bioglass demonstrated that it is possible to obtain 45S5-type biomaterial widely used in medical practice through high-temperature synthesis, when P_2O_5 is introduced into the glass composition using bone ash as a raw ingredient. At the same time, the remaining batch components were selected from traditional and readily available raw materials commonly used in glass manufacturing (mountain crystal, soda, and chalk). This ensures the economic efficiency of the proposed bioglass production technology.

The bioglass obtained using this new approach fully complying with conventional glass production methodology and its crystallized modification both meet the requirements of the ISO 13175-3:2015 standard for conventional biomaterials in terms of their properties.

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