PHYSICS AND CHEMISTRY OF SOLID STATE

V. 25, No. 4 (2024) pp. 880-884

Section: Physics

DOI: 10.15330/pcss.25.4.880-884

Vasyl Stefanyk Precarpathian National University

ФІЗИКА І ХІМІЯ ТВЕРДОГО ТІЛА Т. 25, № 4 (2024) С. 880-884

Фізико-математичні науки

PACS: 81.20.Ev; 87.14.E

ISSN 1729-4428 (Print) ISSN 2309-8589 (Online)

Fadhil K. Farhan, Mazin Mohammed Mawat, Jafer Fahdel Odah Effect of Bone Protein addition to the system: Calcium Titanate

AL-Karkh University of Science, College of Science, Baghdad, Iraq, mazin.alali@kus.edu.iq

Ceramic compound was prepared from mixing calcium carbonate in a mole ratio to nanoscale titanium dioxide powder using the method of manufacturing powders in addition to using mixing and grinding for long hours up to 72 hours in the presence of non-ionic water. After which the powder was extracted and then it was dried at a temperature of 100 °C for 4 hours, and then it was treated at 800 °C in a convection oven at 10 °C of heat for every 2 minutes. An X-ray diffraction test of the ceramic powder was performed before adding the synthesized bone protein that was prepared from cow bones. Scanning electron microscopy and energy dispersion spectrometry were also performed before and after adding the BMP. The results of the examination showed a big difference in the properties of the compound in terms of the atomic structure, as new compounds of bone components were introduced into the atomic structure, and the results were interpreted through the practical intensity of examining the body fluid-like solution (SBF).

Keyword: Bone calcium titanate, manufacturing powders, nano-scale titanium dioxide and synthesized bone protein.

Received 18 April 2024; Accepted 27 November 2024.

Introduction

The bone building process is a complex and highlyorganized physiological process, as it shares many cellular patterns, including how signals enter and exit the body [1]. Bone grafts (manufactured bio ceramic) serve as a framework for the development of the thrombus, its suctioning and its remodeling in order to support the process of bone building within areas of damage and bone defects resulting from accidents, and these grafts or the socalled orthopedic prostheses should be followed by the characteristic of biosimilar receptivity as well as the bone conduction in addition to its main work is bone formation [2, 3, 4]. The prosthetic materials should be non-toxic and compatible with the tissues of the body, and the properties of the bone dictate the organism as well as the cells of the body and have mechanical performance identical to the real bone [5,6]. Thanks to the scientific and technological development in the field of biological and genetic engineering, advanced materials engineering and bioceramics, which is considered one of the promising materials in bone compensation due to the convergence of its performance, biological and mechanical properties to

the bone of the organism, its abundance and ease of obtaining it [7]. As the bio ceramic materials are considered one of the most important and greatest materials used in the field of orthopedic fracture medicine and surgery and dental restoration.). Bone tissue is considered one of the tissues that have a high capacity for self-healing [8,9]. Bone defects have been successfully healed by using many biological and industrial materials such as auto grafts, allografts and other inorganic materials [10]. Calcium carbonate has forever been utilized in bone transplantation since it is described by its capacity to retain and bio-open, and it is a consequence of the parts of living creatures, for example, eggshells that are remembered for its organization at an extremely high rate. In this work, calcium carbonate and calcium dioxide, which is considered a bioactive ceramic material, binds with calcium oxide to be a highly effective compound that is very close to the bone of the living organism. It is a collection of development variables and cytokines that were first identified for their capacity to promote the development of bone and ligament yet are currently recognized as a collection of axial formative signals that control tissue shape all through the body. Dr. Marshall

Urist, an orthopedic surgeon, was the first to notice and conduct substantial study on the capacity of BMPs to trigger a cell reaction that outcomes in the creation of new bone tissue. Eurist practiced orthopedics and served as the director of the Orthopedic Research Lab at the College of California. His unique study involved the implantation of homogenous muscular bone de-calcium from а hydrochloric acid, extracted from grown-up bunnies, other research facility creatures, and individuals, into assorted muscle areas of bunnies, rodents, mice and pigs. Inside half a month after transplantation, new ligament and bone developments shaped close by the surfaces of the giver bone grid. The technique for auto enlistment, which is utilized to create new bone, is portrayed. Both the inducer cell and the prompted cells are gotten from cells that are filling in the host bed. The component in the bone matrix, which was later referred to as BMPs, is thought to be the cause of this phenomena [11]. Contingent upon the cell milieu and cooperation with other administrative factors, BMPs affect the separation, generation, development hindrance, and capture of development of different cells [12]. Numerous protein elements, including various development and differentiation factors, may be found in the bone matrix. As a result, it was unclear whether the reported BMP activity was due to the simultaneous actions of actual particular factors or was innate to another protein factor. The BMP signaling chain is triggered when BMPs attach to the mesenchymal cell's cell surface receptor. Specific proteins transmit signals to the cell's nucleus. The halfway cell turns into a chondrocyte or osteoblast because of the statement of the qualities that outcome in the development of the macromolecules expected to make ligament and bone. An intricate chain of cell occasions, including stromal cell penetration, ligament development, angiogenesis, bone development, and in the end rebuilding of new bone tissue with populaces by the hematopoietic bone marrow parts, were set off by the implantation of this protein part of the bone network [13]. The fusion action of BMPs and the complementing substrate supplying the osteoblast activity of the soluble molecular signal results in the induction of osteochondrosis. It has been demonstrated that BMPs induce direct osteoid cell development in addition to chondrocyte lineage cell differentiation.

I. Experimental Synthesis of CaTiO₃ and BMPs

In this research, bio-ceramic powder was prepared from calcium carbonate compounds and prepared from table eggshells, where the eggshells were taken, cleaned and boiled with hot water, and then were burned in an oven at a temperature of 500°C in order to crush them and get rid of carbohydrates. Then the powder is kiln at a temperature of 1000°C to obtain calcium carbonate. After preparing calcium carbonate, it is mixed and mixed with titanium dioxide in a mole to mole ratio (1:1), and then effective mechanical mixing is used using an electric grinding device in the presence of balls for the purpose of obtaining a homogeneous mixture of the two powdered particles and then the two compounds are reacted at a temperature of 1000°C according to the following equation. Then a bio ceramic of calcium titanate powder will be gotten.

$$TiO_2 + CaCo_3 \qquad 1000 \ ^0C \qquad CaTiO_3 + CO_2$$

After the compound or bone material has been arranged in the subsequent stage, the bone protein will be ready from the remaining parts of cow bone in the wake of washing, cleaning and sanitizing it well, and afterward treating it at a temperature of 500°C, we will get a semistrong material that is effectively squashed and crushed and afterward got back to the broiler by treating it at a temperature of 1250°C, we get a white powder that will in general marginally yellow and this is called blended bone protein. It will be added by 5% to the bio-ceramic powder, and the mixing and blending is done, and then the components are sintered together for the purpose of conducting the structural, biological and mechanical tests.

II. Results and Discussion

Bio-ceramics are advanced ceramics which compactable with the human body tissues in addition to the similarity of natural characterizations between these materials and the normal bones of human. So, bioceramics materials could be used in bones and teeth replacements. Figure (1) shows scanning electron microscopy of CaTiO₃ bio- ceramic powder reinforced by BMPs.

Crystalline CaTiO₃ particles have lower particle sizes and contain a significant quantity of aggregated particles as compared to CaTiO₃ sample, according to the SEM pictures of the reagent CaTiO₃ powders. The particles in figures 2-a and b, which have an average size of 1 m, have a rounded appearance, which is consistent with CaTiO₃ samples made from bio waste. Contrarily, the synthesized CaTiO₃, as seen in figure (2-b), is mostly made up of largely separated particles with average diameters of around 10 um. Additionally, it is simple to distinguish between particles of different sizes and shapes since most particles have extremely uniform shapes. This observation can be made sense of in the way that follows. First, take into account the non-uniform size of the CaCl₂₂H₂O particles in the feeds since they were mechanically quickly grounded because of their high moisture absorption capacity. Second, linear thermal expansion of crystalline CaTiO₃ was visible in this experimental setup. CaTiO₃ may therefore increase quickly. Contrarily, a mixture of the two varieties of crystalline CaTiO₃ was found, and this crystal's smooth surface is a characteristic of crystals.

After being submerged in SBF for four weeks, the $CaTiO_3$ with hydroxyapatite (HA) layers are shown in a cross-sectional SEM picture in Figures 1c and 1d. This research had the objective of tracking how apatite formed on their surfaces. The materials depict a sample with calcium titanates (CaTiO₃) on the surface that developed after being submerged in SBF for seven days.

Figure (2) and table demonstrate the element distribution of O, Ca, and Ti as determined by Energy Dispersive XRD (EDX), using a spot-scan approach (1).

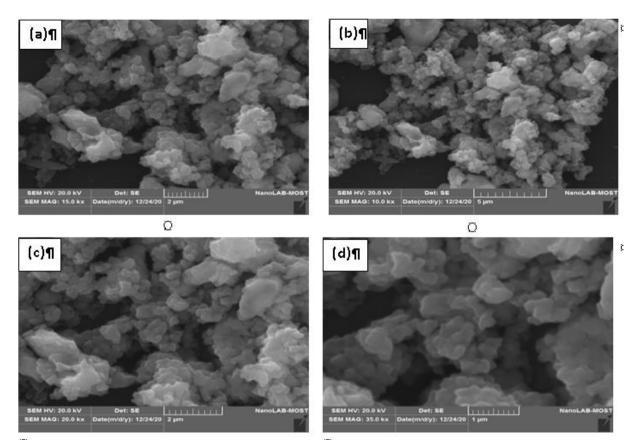
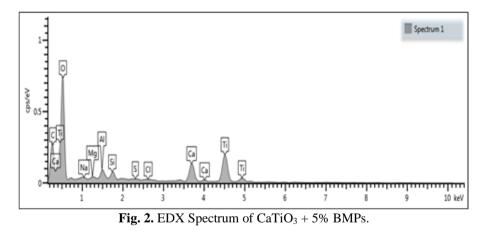


Fig. 1. SEM of CaTiO₃ bio- ceramic powder reinforced by BMPs.



Crystallized character.

The sample included Ti, O, and Ca that were evenly distributed, according to the CaTiO₃ EDX spectra. The material's morphology, however, was not homogeneous. The Ca/Ti ratio is 0.43:1 and the elemental composition shows that no other contaminants than carbon were found. Additionally, the CaTiO₃ powder was found to be free of contaminants since the EDX examination revealed that the nanostructures are made up of Ca, Ti, and O atoms and that only distinct peaks of these three elements were present.

The XRD patterns of the before and after $CaTiO_3$ immersion are depicted in Figures 3-a and 3-b, respectively.

The Perovskite compound $CaTiO_3$ could be classified as orthorhombic unit cells, and the crisp, well-defined peaks in the pattern of figure (3-a) demonstrate that the sample had crystallized. The translucent stages that

EDX Test of CaliO ₃ + 5% BMPs Elements Analysis		
Element	Wt%	Wt% Sigma
C	9.32	1.16
0	38.03	1.82
Na	0.22	0.31
Mg	0.28	0.27
Al	1-14	0.35
Si	1.54	0.34
S	0.04	0.33
Cl	0.07	0.36
Ca	11.22	0.99
Ti	37.87	1.82
Total	100.00	

 Table 1.

 EDX Test of CoTiO
 5% PMPs Elements Analysis

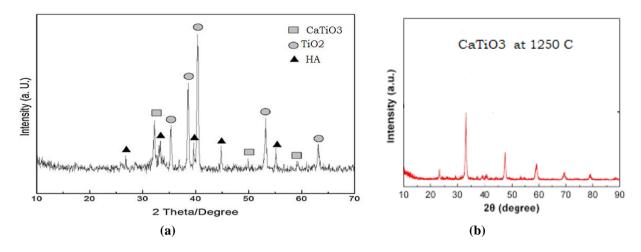


Fig. 3. XRD of CaTiO₃ (a) before SBF immerse and (b) after SBF immerse.

created were likewise perceived by their diffraction designs as anatase TiO₂ (ICDD 86-1157), rutile TiO₂ (ICDD 86-0148), and CaTiO₃ (ICDD 82-0228), which are presented in Table (1). This outcome made sense since excess Ti oxides would form due to Ca deficiency in the CaTiO₃ molecules. Because the powders were sintered at 1250°C and rutile remained stable at such a high temperature, it is noteworthy to notice that rutile rather than the anatase TiO2 phase appeared in the Ca deficient CaTiO₃ powders [14]. The CaTiO₃ diffraction patterns match the results that have been reported in the literature [15]. Brooding in SBF arrangement containing calcium and phosphorus particles and apatite precipitation on the surfaces of analyzed materials are two essential measures that uncover a material's possible bioactivety. Figure (3-b) demonstrates that after 4 weeks of immersion in SBF, the sample exhibits orthorhombic calcium titanate (JCPDS 22-0153) reflections superimposed on the reflections of the polycrystalline titanium substrate (JCPDS 44-1294). There was a trace amount of sodium titanate [16]. CaCO₃ reflections were not seen.

Conclusions

Ceramic system: calcium titanate was successfully prepared using mixing method from calcium carbonate and titanium dioxide. XRD pattern of the final system shows a high purity and homogeneity crystalline structure due to the thermal treatment. The results of SEM and EDX before and after adding BM protein show a different structure according to adding a new reinforced material to the ceramic system. The final system represents a sample containing calcium titanates (CaTiO₃) by all accounts, which was shaped following seven days of test drenching in SBF.

Farhan Fadhil K. – Assistant Professor, Ph.D; *Mawat Mazin Mohammed* – Lecturer, Ph.D; *Odah Jafer Fahdel* – Professor, Ph.D., Dean of the College of Science at AL-Karkh University of Science.

- D.H. Kempen, L.B. Creemary, Growth factor interactions in bone regeneration tissue, Eng. Bart. B., 16(6), 551 (2010); <u>https://doi.org/10.1089/ten.teb.2010.0176</u>.
- [2] Haiping Lu, Yinghong Zhou, Yaping Ma, Lan Xiao, Wenjun Ji, Yi Zhang and Xin Wang, Current Application of Beta-Tricalcium Phosphate in Bone Repair and Its Mechanism to Regulate Osteogenesis, Front. Mater., 8, 698915 (2021); https://doi.org/10.3389/fmats.2021.698915.
- [3] C. Romagnoli, F. D'Asta and M. L Brandi, Drug delivery using composite scaffolds in the context of bone tissue engineering, Clin. Cases. Miner. Bone Metab. 10(3), 155(2013).
- [4] L. Dupoirieux, D. Pourquier, M. Neves and L. Teot, *Resorption kinetics of eggshell: an in vivo study*, J. Craniofac. Surg. 12(1), 53 (2001); <u>https://doi.org/10.1097/00001665-200101000-00009.</u>
- [5] E. Durmus, I. Celik, M.F. Aydin, G. Yildirim and E. Sur, *Evaluation of the biocompatibility and osteoproductive activity of ostrich eggshell powder in experimentally induced calvarial defects in rabbits*, J. Biomed. Mater Res. B Appl. Biomater. 86(1). 82 (2008); <u>https://doi.org/10.1002/jbm.b.30990</u>.
- [6] K Gavenis, U. Schneider, J. Groll, B.Schmidt-Rohlfing, BMP-7-loaded PGLA microspheres as a new delivery system for the cultivation of human chondrocytes in a collagen type I gel: the common nude mouse model, Int J Artif Organs 33(1), 45 (2010); <u>https://doi.org/10.1177/039139881003300107</u>.
- [7] Diana-Elena Radulescu, Ionela Andreea Neacsu, Alexandru-Mihai Grumezescu and Ecaterina Andronescu, Review Novel Trends into the Development of Natural Hydroxyapatite-Based Polymeric Composites for Bone Tissue Engineering, Polymers, 14, 899 (2022); https://doi.org/10.3390/polym14050899.

- [8] HK Kim, JH Kim, DS Park, KS Park, SS Kang, JS. Lee et al. Osteogenesis induced by a bone forming peptide from the prodomain region of BMP-7, Biomaterials, 33(29), 7057 (2012); https://doi.org/10.1016/j.biomaterials.2012.06.036.
- [9] A. Uraz, SE. Gultekin, B. Senguven, B. Karaduman, IP. Sofuoglu, S. Pehlivan et al. *Histologic and histomorphometric assessment of eggshell-derived bone graft substitutes on bone healing in rats*. J Clin Exp Dent;5(1), e23 (2013); <u>https://doi.org/10.4317/jced.50968</u>.
- [10] M. Dhonde, K. Sahu, V.V.S. Murty, S.S. Nemala, P. Bhargava and S. Mallick, *Enhanced photovoltaic performance of a dye sensitized solar cell with Cu/N Co-doped TiO₂ nanoparticles*, J. of Mat. Sci.: Mat. in Electronics, 29(8), 6274 (2018); <u>https://doi.org/10.1007/s10854-018-8605-3</u>.
- [11] J.O. Carneiro, S. Azevedo, F. Fernandes, E. Freitas, M. Pereira, C.J. Tavares, S. Lanceros and V. Teixeira, Synthesis of iron-doped TiO₂ nanoparticles by ball-milling process: the influence of process parameters on the structural, optical, magnetic, and photocatalytic properties, J. Mater. Sci., 49 (21), 7476 (2014); https://doi.org/10.1007/s10853-014-8453-3.
- [12] Asma Manzari-Tavakoli1, Amirhesam Babajani, Mohammad Hadi Farjoo, Mostafa Hajinasrollah, Soheyl Bahrami and Hassan Niknejad, *The Cross-Talks Among Bone Morphogenetic Protein (BMP) Signaling and Other Prominent Pathways Involved in Neural Differentiation*, Front. Mol. Neurosci., 15(15), 827275 (2022); https://doi.org/10.3389/fnmol.2022.827275.2022.
- [13] É. R. Oliveira, L. Nie, D. Podstawczyk, A. Allahbakhsh, J. Ratnayake, D. Lima Brasiland Amin Shavandi, Advances in Growth Factor Delivery for Bone Tissue Engineering, Int J Mol Sci. 22(2), 903 (2021); https://doi.org/10.3390/ijms22020903.
- [14] Tao Li, M. S., Liang Fang, Fengang Zheng, Xinglong Wu., *Effect of Ca deficiencies on the photoluminescence of CaTiO*₃: Pr^{3+} . Journal of Alloys and Compounds, 474(1), 330 (2009); https://doi.org/10.1016/j.jallcom.2008.06.076.
- [15] Hidekazu Tanaka, H.T., Ken'ichi Ota, and Tomoji Kawai. Molecular-dynamics prediction of structural anomalies in ferroelectric and dielectric BaTiO₃-SrTiO₃-CaTiO₃ solid solutions, Physical Review B, 53(21), 14112 (1996); <u>https://doi.org/10.1103/PhysRevB.53.14112</u>.
- [16] J.P. Wiff, V.M. Fuenzalida, J.L. Arias, M.S. Fernandez, Hydrothermal–electrochemical CaTiO₃ coatings as precursor of a biomimetic calcium phosphate layer, Materials Letters 61, 2739 (2007); <u>https://doi.org/10.1016/j.matlet.2006.06.092.</u>

Ф.К. Фархан¹, М.М. Мауват², Дж.Ф. Ода³

Ефект додавання кісткового протеїну до системи титанату кальцію

Університет науки Аль-Карх, коледж науки, Багдад, Ірак, mazin.alali@kus.edu.iq

Керамічну суміш розроблено шляхом змішування карбонату кальцію в мольному співвідношенні з нанорозмірним порошком діоксиду титану методом виготовлення порошків, при цьому здійснювали змішування та подрібнення до 72 годин у присутності неіонізованої води. Після чого порошок екстрагували, потім його сушили при температурі 100°С протягом 4 годин, обробляли при 800°С у конвекційній печі при 10°С кожні 2 хвилини. Перед додаванням синтезованого кісткового білка, отриманого з коров'ячих кісток, проводили рентгенівський дифракційний тест керамічного порошку. Скануючу електронну мікроскопію та енергетичну дисперсійну спектрометрію проводили до та після додавання ВМР. Результати дослідження показали велику різницю у властивостях сполуки з точки зору атомної структури, оскільки нові сполуки кісткових компонентів були введені в атомну структуру, а результати інтерпретовані через практичну інтенсивність дослідження рідини – розчину SBF.

Ключові слова: титанат кальцію кістковий, виробництво порошків, нанорозмірний діоксид титану, синтез кісткового протеїну.