



Proceeding Paper Preparation and Characterization of a Three-Component Composite Based on Polymethylmetacrylate Applied as Bone Cement⁺

Saeedreza Kordbache, Edris Jamshidi and Faranak Manteghi *

* Correspondence: f_manteghi@iust.ac.ir

+ Presented at the 25th International Electronic Conference on Synthetic Organic Chemistry, 15–30 November 2021; Available online: https://ecsoc-25.sciforum.net/.

Abstract: A composite based on three components including PMMA (polymethyl metacrylate), HA (hydroxyl apatite) and LDH (layered double hydroxide) was prepared, characterized and applied as bone cement. Bone cement is a biomaterial used in clinical applications to fix joint replacements in hip and knee joints. There are four types of bone cement, two of which are polymer-based and applied in dentistry and orthopedics, including: (a) acrylic cements based on PMMA; (b) preparation cements based on PPF (polypropyl fumarate). LDH, with the general formula of $[M(II)_{1-x}M(III)_x(OH)_2]^{x+} \cdot (A^{n-})_{X/n} \cdot mH_2O$, has M(II) and M(III) cations and an A^{n-} inter-layer anion. The importance of LDH is its planar layered structure. HA is a thermodynamically stable calcium phosphate similar to human hard tissues in morphology and composition. In this work, we prepare a Zr-CoLDH with a green method and mix it with PMMA and HA in a given ratio. In order to characterize the composite, XRD, FTIR and SEM analyses are performed.

Keywords: PMMA; HA; LDH; bone cement

1. Introduction

The application of PMMA as a biomaterial was preceded by extensive research in the chemical field. Reportedly, 1843 marked the discovery of 'acide acrylique'. This is derived from 'acreolan', the latin word for vinegary, acid or acrid, and it refers to the penetrating smell of the monomer. It was found in 1936 that mixing ground PMMA powder with a liquid monomer results in the formation of a doughy substance. This is due to the partial dissolution of the PMMA in its monomer. Polymer chains from the PMMA become available for free radical polymerization, and entanglements of these chains with newly formed chains lead to an intimate connection between the newly formed PMMA and that which was already present [1].

Layered double hydroxides (LDHs), which are a type of layered material and are also known as anionic clays, are promising layered materials due to some of their interesting properties, such as ease of synthesis, unique structure, uniform distribution of different metal cations in the brucite layer, surface hydroxyl groups, flexible tunability, intercalated anions with interlayer spaces, swelling properties, oxo-bridged linkage, high chemical and thermal stability, and the ability to intercalate different types of anions [2].

The general formula for these LDHs is $(Mg_{1-x}Al_x(OH)_2)^{x+}(A^-)_x \cdot nH_2O$, where "A" represents the interlamellar anion that restore the electroneutrality of the compound. We call these the II–III LDHs. We recently reported that the preparation of Zn-Ti LDH and Co–Ti LDHs consisting of di- and tetra-valent cations is possible [3]. The present work examines the possibility of the preparation of another LDH example consisting of bi- and tetra-valent cations, using a mechanochemical route that has been known as a green method.



Citation: Kordbache, S.; Jamshidi, E.; Manteghi, F. Preparation and Characterization of a Three-Component Composite Based on Polymethylmetacrylate Applied as Bone Cement. *Chem. Proc.* 2022, *8*, 66. https://doi.org/10.3390/ ecsoc-25-11678

Academic Editor: Julio A. Seijas

Published: 13 November 2021

Publisher's Note: MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.



Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/).

Department of Chemistry, Iran University of Science and Technology, Tehran 1684613114, Iran; sm139318@gmail.com (S.K.); edj439@gmail.com (E.J.)

2. Experimental Method

2.1. Synthesis of Co-Zr LDH in a Mortar

Co-Zr LDH was prepared using a mechanochemical route with a simple and green mechanical grinding method. NaOH pellets were added to a powder mixture of Cobalt (II) nitrate hexahydrate and zirconium chloride and manually ground to a paste. The paste was washed four times with deionized water (20 mL), dried under a vacuum, powdered, and analyzed [4].

2.2. Synthesis of the Three-Component Composite

The samples were prepared with 3% (w/w) hydroxyapatite (HA) and 5, 7, and 10% (w/w) LDH. In the first step, HA, PMMA and LDH powder were mixed well with the specified weighted percentages. Then, the mixture was introduced to MMA liquid. The mixture was kept in vacuum state to let gases exit from it. To prepare the composite tablets, the mixture was added to a cast until it became hard.

2.3. Results and Discussion

2.3.1. Co-Zr LDH Characterization

Figure 1 shows the XRD pattern which indicates that the Co-Zr LDH was directly synthesized. The sharp peaks at different 2Θ (19.17, 32.57, and 58.16) were referred to as (003), (006) and (009) plates, respectively [3].



Figure 1. The XRD pattern of Co-Zr LDH.

The FT-IR spectrum of Co-Zr LDH is shown in Figure 2. The widespread and intense band in the 3446 cm⁻¹ area is due to the stretch vibrations of the O–H groups present in the interlayer and the water molecules which are in the layers. The 1625 cm⁻¹ band is related to water-bending vibrations. The sharp bands at 1379 cm⁻¹ and 827 cm⁻¹ are related to the stretch and bending vibrations of the interlayer nitrate anion, respectively. However, the band seen at 1357 cm⁻¹ is related to CO_3^{2-} , which was caused by existing CO_2 in the deionized water [3]. The picks shown at 470 cm⁻¹ and 520 cm⁻¹ are related to M-O-M, which was caused by Zr or Co and oxygen-deionized water.



Figure 2. FT-IR spectrum of Co-Zr LDH.

2.3.2. Scanning Electron Microscopy

The method was selected to investigate the morphology and particle size of Co-Zr LDH. Figure 3 shows FE-SEM images which indicate that the Co-Zr LDH plates were directly synthesized.





Figure 3. The LDH FE-SEM images.

2.3.3. Three-Component Composite Images

Figure 4 shows images of three component composites with 5, 7 and 10% (w/w) LDH, respectively. The results indicate that the pores will increase by increasing the percentage of LDH, as shown in Figure 4. It is expected that the phenomena causes an increase in the concentration of tension, and a decrease in the strength of the composite [5].



Figure 4. Images of the three-component composites with 5, 7 and 10% (w/w) LDH.

3. Conclusions

Co-Zr LDH was proposed as a composite for cement bone because of the micro-sized particles of LDH and the mechanical properties of Zr. It was theorized that the micro size of LDH would prevent the concentration of tension and the decreasing strength of the composite. However, pores increased in size with increasing percentages of LDH. Thus, this phenomenon causes increases in the concentration of tension and decreases in the strength of the composite.

Author Contributions: Conceptualization, F.M.; methodology, E.J.; software, S.K. and E.J.; validation, F.M. and E.J.; formal analysis, S.K. and E.J.; investigation, F.M. and E.J.; resources, F.M.; data curation, E.J.; writing—original draft preparation, S.K. writing—review and editing, E.J.; visualization, F.M.; supervision, F.M.; project administration, F.M. and E.J. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest.

References

- 1. Hendriks, J.; van Horn, J.R.; van der Mei, C.; Busscher, J. Backgrounds of antibiotic-loaded bone cement and prosthesis-related infection. *Biomaterials* **2004**, *25*, 545–556. [CrossRef]
- 2. Mishra, G.; Dash, B.; Pandey, S. Layered double hydroxides: A brief review from fundamentals to application as evolving biomaterials. *Appl. Clay Sci.* **2018**, 153, 172–186. [CrossRef]
- 3. Saber, O. Preparation and characterization of a new nano layered material, Co–Zr LDH. J. Mater. Sci. 2007, 42, 9905–9912. [CrossRef]
- 4. Ay, A.N.; Zümreoglu-Karan, B.; Mafra, L. A Simple Mechanochemical Route to Layered Double Hydroxides: Synthesis of Hydrotalcite-Like Mg-Al-NO₃-LDH by Manual Grinding in a Mortar. *Z. Anorg. Und Allg. Chem.* **2009**, *635*, 1470–1475. [CrossRef]
- Ben, H.S.A.; Dimitrijević, M.M.; Kojović, A.; Stojanović, D.B.; Obradović-Đuričić, K.; Jančić Heinemann, R.M.; Aleksić, R. The effect of alumina nanofillers size and shape on mechanical behavior of PMMA matrix composite. *J. Serb. Chem. Soc.* 2014, 79, 1295–1307. [CrossRef]