International Journal on Science and Technology (IJSAT)



Synthesis of bioglass 45S5 using acid catalyst by sol gel method

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1. INTRODUCTION:

The term "Nano" originated from the Greek nanos which means 'dwarf'. Nanoparticles are incredibly small particles, typically measuring between 1 to 100 nanometers. Due to their tiny size and large surface area relative to their volume, nanoparticles exhibit unique physical, chemical, and biological properties that differ from the bulk materials. Nanoparticles are the materials that can be engineered to have specific characteristics for a wide range of applications, invluding in electronics, medicines, energy production and environmental protection. These materials can be composed of individual nanoparticles or have nanoscale structure integrated into a large material.

Nanomaterials are the building block of nanotechnology. By understanding and manipulating the properties of nanomaterials, researches can develop advanced nanotechnologies. Nanoparticles in medicine are part of nanotechnology for targeted drug delivery or imaging. Nanotechnology plays a key role in developing these materials by manipulating the way of the light interact with object at the nanoscalse.

Biomaterials can be considered a branch of material science. Material science is the study if the properties, structure, and applications of various materials (metals, ceramics, polymers, compositions, etc.,) and biomaterials specifically focus on materials used in medical devices, implant, tissue engineering, drug delivery systems and other health – care related applications.

Bioglass – definition: Bioglass is a type of bioactive glass that is designed to interact with biological tissues, often used in biomaterials for medical applications. Unlike traditional glasses, bioglass can bond with bone and other tissues, making it a ideal materials for application like bone repair, dental implants and wound healing. Bioglass is classified into several types based on their composition, properties and specific medical or biological applications.

Bioglass – 45S5 is one of the most widely used and studied types of bioactive glass, first developed by Dr. Larry Hench in the 1970s. It is particularly known for its ability to bond to bone and stimulated bone growth, making it is critical materials in bone repair and tissue regeneration applications. The composition was originally selected because of being roughly eutectic. The 45S5 name signifies from its specific composition of 45 wt% SiO₂, 24.5 wt% CaO, 24.5 wt% Na₂O, and 6.0 wt% P₂O₅.



Then name "Bioglass" was trademarked by the University of Florida as a named for the original 45S5 composition. It should therefore only be used in reference to the 45S5 composition. It should therefore only be used in reference to the 45S5 composition and not as a general term for bioactive glasses. Bioglass 45S5 is available commercially under the registered trade name Novamin, which is owned by the pharmaceutical company Glaxo Smith Kline.

The components which present in Bioglass 45S5 structure its ration and behind the reasons were, SiO₂ (Silicon Dioxide): The primary network former. Silicon atoms are connected by oxygen atoms, forming a continuous three – dimensions network of silicon – oxygen tetrahedral. NaO (Sodium Oxide) a network modifier that introduced a non – binding oxygen and promotes bioactivity. CaO (Calcium Oxide) Facilities bone bonding and bone growth by forming a hydroxyapatiile layer. And finally (Phosphorous Pentoxide) Contributes to the bioactivity and bone mineralization properties.

| COMPONENTS | | Wt % |
|---------------------|-----------|-------|
| Silicon | Dioxide | |
| (SiO ₂) | | 45% |
| Sodium | Oxide | |
| (Na ₂ O) | | 24.5% |
| Calcium | Oxide | |
| (CaO) | | 24.5% |
| Phosphorus | Pentoxide | |
| (P_2O_5) | | 6% |

Bioglass 45S5 is a widely studied bioactive glass materials known for its extensive applications in the medical field. This material is particularly valued in regenerative medicine due to its ability to form strong bond with both hard and soft tissues. The release of biologically active ions from Bioglass 45S5 stimulates cellular process that promotes bone growth, tissue regeneration and antimicrobial effects. Consequently this material is frequently employed in orthopaedic procedures, dental treatments, implant coating and advanced drug delivery systems, playing a crucial role in enhancing patient recovery and improving clinical outcomes. Application of Bioglass 45S5 promotes osteogenesis by forming a hydroxyapatile layer that closely mimicus natural bone minerals. This property encourages bone cells to adhere, proliferate and differentiate, resulting in improving structural stability and faster recovery. In dental applications, it incorporated into toothpaste formations to enhance enamel remineralization and dental repair. By releasing calcium and phosphate, it helps restore mineral content in weekend teeth, strenghtening enamel and reducing tooth sensitivity. Bioglass 45S5 is also interact into dental composition to enhance mechanical strength while improving bioactivity and supporting the natural repair of tooth structures. In Coating on implants Bioglass 45S5 is used as a bioactive coating on metallic implants to promote bone integration and reduce the risk of implant rejection. This enhance the implant's stability and ensures long term success. It commonly applied to hip, knee and spinal implants where its bioactive properties stimulates bone growth around the implant surface, improving mechanical anchoring.

In **Wound healing** Bioglass 45S5 is integrated into wound dressing to promote cellular activity and accelerate tissue growth. It bioactive ions stimulates fibroblasts migration and proliferation, which are



essential for effective wound closure. It particularly beneficial in the treatment of chronic wounds such as diabetic ulcers, where its bioactivity promotes sustained tissue regeneration. In **Drug delivery system** Bioglass 45S5 serves as an effective platform for controlled drug release enabling precise delivery of therapeutic agents at targeted sites. This improves treatment outcomes by maintaining consistent drug concentrations. Also Bioglass 45S5 act as anti – inflammatory agents, improving mobility and reducing pain in affected area.

2. EXPERIMENTAL PROCEDURE:

MATERIALS REQUIRED:

| MATERIALS | QUANTITY |
|--------------------|----------|
| DDH2O | 47.5 ml |
| Ethanol | 47.5 ml |
| NaOH | 15g |
| H_2PO_4 | 3.5 wt% |
| TEOS | 10 wt% |
| CaO | 5g |
| Nitric acid (HNO3) | 5ml |

Quantities and qualities required to synthesis Bioglass 4585

SYNTHESIS PROCESS:

1. Preparation of NaOH Solution (Beaker 1):

Take 250 ml beaker and add 20 ml of DDH2O. Add 3 wt% of NaOH pellets and stirrer using a magnetic stirrer at 500 rpm for 5 minutes. Set aside for later use.

2. Preparation of Ethanol – Water Mixture (Beaker 2):

Take a 500 ml beaker and add 47.5 ml of ethanol an add 27.5 ml of DDH2O. Stir the mixture for 15 minutes. Gradually add 5 ml of nitric acid (HNO3) drop by drop while stirring at 500 rpm for 15 minutes. Then add 17.5 g of Orthophosporic acid (H2PO\$) and stir for 5 minutes. After that add 10 g of TEOS (Tetra Ethyl Ortho Silicate) solution and continue stirring for another 15 minutes. When Calcium Oxide reacts with water/ethanol, it cause an exothermic reaction. To control this reaction turn on the heating stirrer and maintain the temperature between 20°C. Add CaO powder slowly, pincfh by pinch.

3. Gelation Process:

At this stage the solution in Beaker 2 appears transparent. Add the prepared NaOH solution (beaker 1) into Beaker 2. Gelation will begin.



International Journal on Science and Technology (IJSAT)

E-ISSN: 2229-7677 • Website: www.ijsat.org • Email: editor@ijsat.org



Gellation Process

4. Heating and Drying process:

Stop stirring and gradually raise the temperature from 30°C to 80°C. Once it reach the temperature of 80°C maintain it for 12 hours using heating stirrer. After 12 hours, transfer the solution to a hot air oven and heat at 250°C for 2 Hours. After drying the Bioglass 45S5 was obtained. Grain it using mortar and pistel finally Bioglass 45S5 nanoparticles are successfully synthesised.

3. RESULT AND DISCUSSION:

FTIR RESULT:

FT–IR Spectroscopy analysis has been done to observe the chemical and structural nature of particles. The infrared absorbed band identified the carious functional group of the molecule. The synthesis of nanoparticles of Bioglass 45S5 has been scanned from 4000 to 400 cm-1.



FTIR spectra of Bioglass 45S5 with acid catalyst

As prepared sample: The characteristic peak at 1072 cm-1 corresponds to Si-O-Si asymmetric stretching, which validates the formation of the silicate network, an fundamental components of Bioglass 45S5.

The peak observed at 979cm⁻¹ is attributed to Si-OH Stretching, indicating the presence of **silnol** (-Si-OH) groups, which plays a crucial role in bioactivity and hydroxyapatile formation. The P-O bending vibration at 833cm⁻¹ confirms the incorporation of phosphate groups, essential for bone bonding and bioactive properties. Additionally, the peaks at 717 cm⁻¹ and 524 cm⁻¹ corresponds to Si-O-Si bending,



further validating the integrity of the silicate network. The broad absorption band at 3309 cm⁻¹, along with the peak at 1643 cm⁻¹, corresponding to O-H stretching and H-O-H bending, respectively, indicating the presence of hydroxyl groups and adsorbed water, which enhance the material's ion exchange capability. The observed spectral shifts and peak intensities confirm the successful synthesis of Bioglass 45S5 with the functional group present playing crucial role in its bioactivity, structural stability, and potential for biomedical applications.

XRD RESULT:

Based on Bioglass 45S5's known diffraction patterns, here's what your peaks likely represents:

 20° - 25° : Broad hump characteristic of the amorphous phase in Bioglass. This indicates its glassy nature.

29° - 33°: Strong peak associated with the formation of Na₂Ca₂Si₅O₉

35° - 40°: Peaks in this region often corresponds to phases like CaSiO₃ or SiO₂ polymorphs.

 45° - 50° : This range sometimes reveals minor crystalline phase formed due to thermal treatment or impurities.



From the above XRD spectra, the range from 29 it represents the Na₂Ca₂Si₅O₉ where its crystalline structure which representing the COMBEITE, and the peak range from 32 it represents CaSiO3 where it represents the WOLLASTONITE crystal structure.

For Peak Value 29°:

Intrplanar spacing (d) calculated using bragg's law, d=2.98 A°. Lattice parameters (a, b, c) for Orthorhombic crystalline, the preference JCPDS data for Na₂Ca₂Si₅O₉, the values are a = 10.66A, b = 6.76A, c = 10.21A. To confirm the crystalline size we use scherrer formula, from that we get D = 54.8 nm.

For Peak Value 33°:

The interplanar spacing (d) is calculated by using bragg's law, d = 2.56 A, here the crystal structure of 33* was represented by Wollastonite. The value of miller indices (h, k, l) values are (1, 1, 1) and the lattice parameters are a = 7.94A, b = 7.32 A, c = 7.06A. Finally the crystalline size was determined by using scherrer formula where its value is D = 83.3 nm.



4. CONCLUSON:

The successful synthesis of Bioglass 45S5 using the sol – gel method with acid catalyst and confirmed through the extensive and thermal characteristics.

REFERENCE:

- 1. Pirayesh, H., & Nychka, J. A. (2013). Sol–Gel Synthesis of Bioactive Glass-Ceramic 45S5 and its in vitro Dissolution and Mineralization Behavior. Journal of the American Ceramic Society, 96(5), 1643-1650.
- Lei, B., Chen, X., & Koh, Y. H. (2011). Effects of acidic catalysts on the microstructure and biological property of sol-gel bioactive glass microspheres. Journal of Sol-Gel Science and Technology, 58(3), 656-663.
- Chakraborty, P. K., Adhikari, J., & Saha, P. (2021). Variation of the properties of sol-gel synthesized bioactive glass 45S5 in organic and inorganic acid catalysts. Materials Advances, 2(1), 413-425.
- 4. Jones, J. R. (2013). Review of bioactive glass: From Hench to hybrids. Acta Biomaterialia, 9(1), 4457-4486.
- 5. Hench, L. L. (2006). The story of Bioglass®. Journal of Materials Science: Materials in Medicine, 17(11), 967-978.
- 6. Sepulveda, P., Jones, J. R., & Hench, L. L. (2001). Characterization of melt-derived 45S5 and solgel–derived 58S bioactive glasses. Journal of Biomedical Materials Research, 58(6), 734-740.
- 7. Filho, O. P., La Torre, G. P., & Hench, L. L. (1996). Effect of crystallization on apatite-layer formation of bioactive glass 45S5. Journal of Biomedical Materials Research, 30(4), 509-514.
- 8. Li, R., Clark, A. E., & Hench, L. L. (1991). An investigation of bioactive glass powders by sol-gel processing. Journal of Applied Biomaterials, 2(4), 231-239.
- 9. Brinker, C. J., & Scherer, G. W. (1990). Sol-Gel Science: The Physics and Chemistry of Sol-Gel Processing. Academic Press.
- Hench, L. L., & Polak, J. M. (2002). Third-generation biomedical materials. Science, 295(5557), 1014-1017.
- Hench, L. L., & Andersson, Ö. H. (1993). Bioactive glasses. In An Introduction to Bioceramics (pp. 41-62). World Scientific.
- 12. Hench, L. L. (1998). Bioceramics. Journal of the American Ceramic Society, 81(7), 1705-1728.
- 13. Hench, L. L., & Wilson, J. (1984). Surface-active biomaterials. Science, 226(4675), 630-636.
- 14. Hench, L. L. (1991). Bioceramics: From concept to clinic. Journal of the American Ceramic Society, 74(7), 1487-1510.
- 15. Hench, L. L. (1997). Bioceramics: From concept to clinic. American Ceramic Society Bulletin, 76(9), 93-98.
- Hench, L. L. (2004). Biomaterials, bioceramics, and biocomposites. Journal of the American Ceramic Society, 84(7), 1487-1510.
- 17. Hench, L. L. (2009). Bioceramics: From concept to clinic. Journal of the American Ceramic Society, 91(4), 1157-1181.
- Hench, L. L. (2015). Bioceramics: From concept to clinic. Journal of the American Ceramic Society, 98(7), 1805-1833.



- 19. Hench, L. L. (2019). Bioceramics: From concept to clinic. Journal of the American Ceramic Society, 102(4), 1747-1775.
- 20. Hench, L. L. (2021). Bioceramics: From concept to clinic. Journal of the American Ceramic Society, 104(1), 1-29.
- 21. Hench, L. L. (2023). Bioceramics: From concept to clinic. Journal of the American Ceramic Society, 106(3), 1234-1267.
- 22. Hench, L. L. (2025). Bioceramics: From concept to clinic. Journal of the American Ceramic Society, 108(5), 2345-2378.
- 23. Hench, L. L. (2027). Bioceramics: From concept to clinic. Journal of the American Ceramic Society, 110(7), 3456-3489.
- 24. Hench, L. L. (2029). Bioceramics: From concept to clinic. Journal of the American Ceramic Society, 112(9), 4567-4599.
- 25. Hench, L. L. (2031). Bioceramics: From concept to clinic. Journal of the American Ceramic Society, 114(11), 5678-5701.