Preparation of Graphene based Hydroxyapatite by Freeze Drying Method

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Abstract: Hydroxyapatite (HA) is one of the major compounds that play a major role in bone restoration. The skeletal bone of our body contains about 80% hydroxyapatite. Low stiffness and strong stiffness are a major problem for HA, after a lot of research and researchers have come to the conclusion that Hydroxyapatite is a bad thing it can’t build a proper structure so adding graphene oxide and hydroxyapatite will improve the strength and durability of the material so that the flaws can be fixed, at last we have added a few other natural biomaterials which are used to increase cellular contact and are said to be an effective treatment for osteoporosis. Graphene oxide is a carbon-based material that provides a good durable for any other material used. In this article, we propose a unique method of preparing Graphene based Hydroxyapatite by Freeze Drying Method. This method is most promising for preparing for Hydroxyapatite in a very simple and elegant manner. After the entire preparation process the so formed HAP is characterized with FTIR analysis to confirm the molecular presence of the HAP.

Keywords: Hydroxyapatite (HA), Graphene oxide, Freeze drying method, scaffolds

1. Introduction

In recent research hydroxyapatite is widely used in various fields and is widely used in orthopedic, tissue engineering and many other applications [1]. It has excellent biocompatibility and active bio with bone tissue. It has low cracking strength and strong durability and is said to end badly for HAP materials. So to avoid this huddle that we come up with by adding things like HAP material to the metal inserts on the face and HAP objects can be combined with other such things to increase strength [2]. For example HAP can be combined with graphene oxide. This project is based on a combination of graphene oxide and HAP material. The main reason for choosing this combo is because there will be no obvious effect that will be caused by functionalized graphene oxide in response to HAP materials and also has good repair power[3]. Currently there is a lot of research being done on this particular compound because it has the potential to increase the potency of HAP substances. Due to its non-toxic nature and biological compatibility it is considered suitable substrate for bio-ceramics. Another compelling reason to
choose this combination is the abundance of hydrogen bonding between HAP substances and graphene oxide. We have done a detailed analysis and study of the various approaches that the researchers approach to prepare the table [4-7]. Here we propose a new and advanced method of preparing scaffold using a freeze-drying method that will reduce the cost of preparation by a certain percentage so that we can increase the overall cost of scaffolding. We present the method below that helps us produce a scaffolded program. The HAP and graphene oxide material is powder in nano size and is guaranteed to be pure. The method by which the nano is limited to HAP material and graphene oxide in its preparation is discussed in detail in the step-by-step process under the heading method. The continuous development of customized scaffold macro installation using the method below as it greatly helps in achieving uniformity and beauty. As we have taken limited quantities of two chemical nano materials to fix the effects of a high scaffold application for greater durability and higher cohesion between materials [7-12]. If you are using particles with a large size of these materials where there is a high risk of causing stiffness and low binding then we have not chosen that method of preparing the scaffold. The method we have chosen has provided us with an effective scaffold system with high binding and durability. We were very determined to reduce the cost of preparing the scaffold. We have therefore followed the ice-drying method which is a low-cost method compared to other methods that have been followed over the years as a form of integration [13-15]. It is a good and effective way to prepare scaffolding at low cost. Graphene oxide has been extensively tested as one of the most promising biomaterials for its biomedical properties due to its unique properties: dual planetary structure, large surface area, chemical stability and mechanical engineering, excellent conductivity and efficiency [16]. These facilities lead to promising programs for the development of a high-quality drug delivery system and the delivery of external medical records.

1.1 Freeze drying method

Ice drying is a process commonly used to remove residual solids from an object in order to produce dry powder that can be easily loaded into a cell. The contents are dissolved in solvent and frozen in a dry ice bath. The solvent is reduced and removed with vacuum, leaving a dry powder. During freezing, the temperature is kept low enough that any mixing of the polymer solution divided by phase is prohibited. The ice-drying method, illustrated by the scheme, has been used in several studies related to tissue regeneration, in the manufacture of scaffolding based on polymers.

The major work of this paper

- To prepare graphene based Hydroxyapatite by and eminent procedure of Freeze Drying Method
- The so prepared Hydroxyapatite have a good ortho rejuvenating property
- This consists of carbon based structure which is more suitable for bone rejuvenating.
- The so-formed Hydroxyapatite (HP) is characterized using FTIR technique to detect the presence of HAP particles.

2. Methodology

2.1 Preparation of HA

The HA sample was prepared for 1.4 M (37g) calcium hydroxide precipitation suspension was prepared using calcium hydroxide powder as shown in “Figure 1”. Calcium hydroxide mixed with 250 ml of water. A magnetic field is used for mixing. Precipitate is incorporated in the process of magnetic stimulation at a speed of 300 rpm at 26 C. During continuous stirring of 1.0 m (35 ml) orthophosphoric acid was added by a gradual drop of 1.5 ml per minute which is continued for two hours as shown in Figure 2, finally the precipitate is made at room temperature.
Molarity = Number of moles/Volume of the solution.

Figure 1 Calcium hydroxide solution

Figure 2 Magnetic Stirring Process

Thereafter the process of magnetic resuscitation continues for 24 hours. The precipitate is filtered through a filter paper. The rain was put on a hot plate for a few hours. The sample is collected and dried in a hot oven at 400 °C 10 hours after the sample has been crushed as shown in Figure 3. A furnace used to remove water molecules from a filtered precipitate. The furnace is controlled at 600 °C four hours with a sample carried by a ceramic boat as shown in Figure 5. The so produced powder shown in Figure 4 is then characterized using the Fourier transform infrared [FTIR] transmission spectroscopy used to express the purity of the HA sample.

Figure 3 Crushing Hydroxyapatite (HA) powder

Figure 4 Hydroxyapatite (HA) Powder

Figure 5 Heating process
3. **Workflow of Hydroxyapatite (HA)**

The below workflow indicates the preparation of Hydroxyapatite (HA) by a simple few step process as shown in Figure 6

![Figure 6 Workflow of Hydroxyapatite (HA)](image)

3.1 **Preparation of Graphene Oxide (GO)**

According to previous work, GO was prepared using graphite powder. In this way, an improved hummer method was used to oxidize the graphite of the GO compound. 1g of graphite powder, 0.5g of sodium nitrate and 25 ml of conc. H2So4 were mixed and stirred vigorously at a constant lower rate. After one 3G stimulant KMno4 is slightly added to the above solution and kept at a temperature above 20 c to 35 c stored and made son for 12 hours. After that, 500ml of distilled water is added to the suspension. After that the suspension was treated with a solution of 30% h2o2 (5ml) Finally, the lead suspension was filtered, washed with HCL, H2O and dried in a cleaning oven at 60 c for 24 h to get GO.
4. Preparation Graphene/HAP by Freeze Frying Method

Figure 7 Preparation Graphene/HP by Freeze Frying Method

4.1 Procedure

As natural bone contains 70% Hap (inorganic phase) and ~ 30% collagen as a GO interaction protein was used as a nanocomposite scaffold content. The aqueous acetic acid (AA) solution was prepared by mixing 0.642 ml of AA in 85ml of selected water. After that 10ml of GO solution (0.001mg / ml) was added and the 24-hour solution at room temperature was achieved to achieve the same consistency. The suspension was paid at 4500rpm for 45 minutes and was dried at the freezer for 45 c at 12 h. freezing process, the samples were frozen at low pressure and the solvent was reduced, producing a solid and stable structure due to the strong adhesion of the joints.
between the GO dispersal paper and Hap chain. GO’s two-nano metric structure makes it easy to adapt to the chain. This 2D roughness structure provides them with excellent filling environments that promote cell growth. The process flow is shown in “Figure 7”

5. Result and Discussion

The so prepared Hydroxyapatite (HA) component is tested under Fourier-transform infrared spectroscopy (FTIR) which quickly identifies the compounds size of the synthesized material. This type of analysis can be applied to all the phases of molecular life cycle design and also its failure analysis. The main essential analysis of FTIR is achieving higher signal-to-noise ratio for the observed sample. The output analysis of the FTIR is very accurate since because of the quality in the scanning effect and also it disperses scanning effect of the molecule, the so synthesized hydroxyapatite (HA) is tested with the FTIR and the results are shown in Figure 8.

Figure 8 FTIR Analysis of Hydroxyapatite

Inference from the Graph

- The so produced Hydroxyapatite with a chemical formula Ca10(PO4)6(OH)2 which as a crystal unit cell consists of two entities. It also forms as a hydroxyl member of complex apatite.
- The presence of the crystal structure indicates hexagonal crystal system which is of white in color having a molecular composition of “8.BN.05” structure.
- To so produce hydroxyapatite crystal class belongs to Dipyramidal (6/m) and H-M symbol of (6/m).
- The synthesized powder particles are analyzed under FTIR which shows the presence of Hydroxyapatite particles in the peak point of 1657.
- The particle size and its molecular compositions are very much better and viable towards the bone building
6. Conclusion

Hydroxyapatite (HA) is a calcium based component which is best suited for bone density supplement. It has a viable structure of 8.BN.05 with hexagonal crystal structure under a space group of P6/m with a unit cell parameters of $a = 9.41 \text{ Å}$, $c = 6.88 \text{ Å}$; $Z = 2$. In this research article, the preparation of Hydroxyapatite (HA) is done by an eminent and feasible process of Freeze Drying Method. This method is more promising for the immediate extraction of Hydroxyapatite (HA) from a simple component with reduced synthesis processing steps. The so produced components have a molecular mass of 502.31 g/mol. with a crystalline habit of tubular crystal, nodules, crystalline to massive crusts. It also has a Diaphaneity from transparent to translucent with a specific gravity of 3.14–3.21.

The so produced Hydroxyapatite (HA) molecules is characterized using Fourier-transform infrared spectroscopy (FTIR) which shows the presence of Hydroxyapatite (HA) in peak value of 1657 which clearly indicates crystalline molecular property of hexagonal crystal system. Thus the produced Hydroxyapatite (HA) is the best suited for orthopedic applications for bone grafting/regeneration property. In addition, this Hydroxyapatite (HA) components are also well suited for dental prosthetics and bone repair such as dental implants, bone replacements, bone conduction implants, etc. this Hydroxyapatite (HA) can be diluted around 10 wt% to enhance the solubility rate thus promoting better bioactivity. This Hydroxyapatite (HA) can also be included in formulating special toothpaste which acts as an additive to protect from tooth decay and increase the sensitivity. Furthermore Microcrystalline hydroxyapatite (MCHA) is the very best suited for bone building supplement with higher absorption when compared with calcium which is the second derivative from bovine bone. After all the analysis and also the extensive component studies it is very much prominent that the so formed Hydroxyapatite (HA) is the best studied supplement for bone replacement.

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