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In-situ fabrication of rod shaped nano-hydroxyapatite using microwave assisted semi-interpenetrating network as a template-morphology controlled approach

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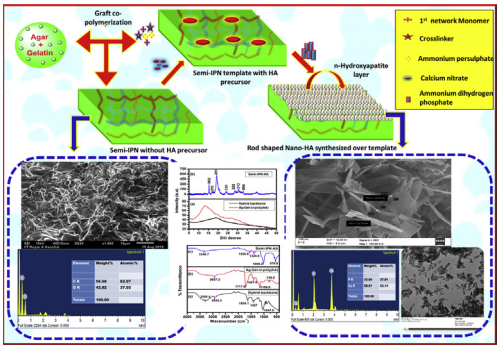
HIGHLIGHTS

- Graft copolymerization was done using microwave radiations to produce semi-IPN.
- Semi-IPN synthesized has 4750% swelling and antibacterial property.
- Sample characterization, biodegradation, salt resistant and moisture retention studies.
- Morphology controlled approach to grow rod shaped hydroxyapatite using semi-IPN.
- Nano-hydroxyapatite showed crystallite size of ***50-100 nm*** (l) and ***15–30*** *nm* (dia).

GRAPHICAL ABSTRACT



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ABSTRACT

The present work deals with the fabrication of decomposable, agar-gelatin based semi-IPN superabsorbent which was cast-off as template for the synthesis of bioactive nano-hydroxyapatite. Microwave assisted free-radical polymerization technique was used and the optimized reaction parameters were: initiator (mol/L): 0.021; time (sec): 15; microwave power (%): 100; AA (mol/L): 6.994; MBA (mol/L): 6.4×10^{-3}; solvent (ml): 12.5 and pH: 7.0. Semi-IPN could imbibe water to a large extent and maximum percentage swelling found was ***4750%***. The synthesized semi-IPN was used as mineralization matrix for the fabrication of rod-shaped nano-hydroxyapatite crystals which showed Ca/P ratio ***1.665*** similar to natural bone (1.67) with average crystallite size ***50–100 nm*** (length) and ***15–30 nm*** (diameter) respectively. Further soil water retention studies revealed that synthesized semi-IPN template could retain moisture for about ***55 days, 40 days*** *and* ***31 days*** in clayey, mix-soil and sandy soil, respectively and degraded upto ***84%*** in ***70 days*** using composting and ***80%*** using vermi-composting method of biodegradation. Thus, synthesized device being antibacterial, biodegradable and ecofriendly could have a contribution in ever-green revolution also used as template for the morphology controlled synthesis of n-HA.

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Abbreviation: n-HA, Nano-Hydroxyapatite; AA, Acrylic acid; APS, Ammonium persulphate; MBA, N,N'methylene-bisacrylamide; semi-IPN, Semi interpenetrating network; P_s, Percentage swelling; P_g, Percentage grafting.

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1. Introduction

Hydrogels from natural polymers such as gelatin, agar and β-carrageenan have gained a particular attention due to their natural origin, very low cost and high biocompatibility [1–3]. The weak mechanical properties and easy degradation limit the uses of biopolymers in biomedical field. Their properties can be improved by using different chemical and physical methods. The improved biopolymers have immense applications such as biomedicals, tissue engineering, textiles and nano-biotechnology. Homo-polymers although find extensive applications in drug delivery devices [4,5] and as food additives in food industry, but hydrogel formed of single polymer cannot meet the all desired demands in terms of both properties and performances. Therefore blending is an important method to bring the advantages of different polymers together. The polymer blends may show synergistic physical and mechanical properties [6]. Batorskey et al. (2005) incorporated collagen into agar and used to manage cell functions and to induce differentiation in stem cell therapy [7]. Blending of agar with gelatin is more economical as compared to collagen or other common extra cellular matrices derived oligopeptides. Previous studies on agar–gelatin conjugates have shown good results, which makes this combination a good candidate for cell enclosing capsules and for tissue engineering [8]. Agar-gelatin blends with different concentrations were evaluated for cell encapsulation by Rana Imani et al. (2012) [9]. Owing to a large variety in chemical structure and crosslinking methods, various hydrogels have been reported which are commonly used in clinical practice and experimental medicine for a wide range of applications including tissue engineering and regenerative medicine, diagnostics, immobilization, separation of biomolecules and as barrier materials to regulate biological adhesions [10,11].

Past few decades has witnessed a revolutionary advancement in the area of development of noval drug delivery system, tissue engineering, biomedical engineering and nano biotechnology. More of the focus of medical research was to explore new scientific frontiers for diagnosing, treating and curing diseases at the genetic level. Biomaterials proved to be an efficient tool to meet the requirements. The major classes of biomedical materials are metallic, ceramics, polymeric, and composites [12]. Bioceramic n-HA has been widely used about two decades as a biomaterial in biomedical science and technology. n-HA is comparatively cheap, non-hazardous, negligibly resorbed, of adequate compressive strength, and attaches fit to hard tissues without any cytotoxicity [13,14]. Various techniques are used to grow n-HA in nano range. Some of these techniques are: wet chemical deposition, biomimetic deposition, sol-gel and electro-deposition [15,16]. A cost effective, environment friendly method is required to produce crystalline n-HA at lower temperatures. One of the techniques used in our research is biomimetic deposition of hydroxyapatite layer using natural polysaccharide and protein as templates.

Present work deals with the synthesis of semi-IPN based on agar-gelatin backbone and its evaluation for water-retention capacity in different types of soils and its biodegradability was teasted. Further the synthesized semi-IPN was successfully used as a template to grow hydroxyapatite nano crystals.

2. Experimental

2.1. Materials

Agar from Hi-Media chemicals and Gelatin from CDH chemicals were purchased and used as the backbone. Ammonium persulphate (APS), N,N'methylene-bisacrylamide (MBA) and acrylic acid (AA) were procured from S.D fine chemicals (India). Calcium nitrate (Thomas baker chemicals) and ammonium dihydrogen phosphate (Merck chemicals) were used as received. Reagents NaCl, $CaCl_2$, $FeCl_3$, $SnCl_4$, HCl and NaOH used were of analytical grade.

2.2. Instrumentation

The functional groups present on the hybrid backbone, semi-IPN, IPN and hydroxyapatite were analyzed by FT-IR (Agilent technologies Carry 630 spectrophotometer) using a range of 4000 cm^{-1} to 400 cm^{-1}. FE-SEM studies have been done on ultra plus FE-SEM (Carl Zeiss, Germany Ltd.) and thermal studies were done on TG/DTA 6300, SIIEXSTAR 6000 having a heating rate of 10 °C/min. TEM Hitachi (H-7500) was used to find nano range of prepared hydroxyapatite. XRD was recorded using XRD Panalaytical X'Pert Pro multipurpose diffraction (MPD) with Cu-Kα radiation of wavelength 1.54 Å. Crystallite size (L) in nm was calculated using Scherrer Eq. (1) [17]:

$$L = K\lambda \,/\, b\cos\theta \tag{1}$$

Where $\lambda = 1.54$ Å is the wavelength of the Cu-Kα X-ray radiation used, K is the Scherrer constant (value = 1), θ is the Bragg angle and b is the FWHM of the diffraction peak.

2.3. Synthesis of semi-IPN

Semi-IPN was prepared by soaking agar and gelatin in an equal weight ratio in distilled water for overnight. This was followed by drop-wise addition of acrylic acid with continuous stirring and addition of a calculated amount of APS and MBA at 37 °C. The reaction mixture was kept in the microwave and irradiated for 15 s. Resulted semi-IPN was freed from unreacted monomer and homopolymer by giving washing with distilled water. Semi-IPN was dried and weight was noted to calculate % grafting. Optimization of different reaction parameters like APS, reaction time, volume of solvent, reaction medium pH and microwave power was done with respect to percentage grafting (P_g). However, optimization of AA and MBA was carried out with respect to percentage swelling (P_s).

The percentage (%) grafting was calculated using equation (2) [18]:

$$Pg = \frac{W_f - W_b}{W_b} \times 100 \tag{2}$$

W_f is the weight of template and W_b is the weight of hybrid backbone.

The percentage (%) swelling was calculated using equation (3) [19]:

$$Ps = \frac{W_s - W_d}{W_d} \times 100 \tag{3}$$

W_s is the weight of swollen hydrogel sample and W_d is the weight of dry hydrogel sample.

2.4. Synthesis of nano-structured hydoxyapatite using semi-IPN as template

$Ca(NO_3)_2.4H_2O$ was assimilated into the reaction mixture to

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