



Guar gum based biodegradable, antibacterial and electrically conductive hydrogels



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ABSTRACT

Guar gum-polyacrylic acid-polyaniline based biodegradable electrically conductive interpenetrating network (IPN) structures were prepared through a two-step aqueous polymerization. Hexamine and ammonium persulfate (APS) were used as a cross linker-initiator system to crosslink the poly(AA) chains on Guar gum (Ggum) backbone. Optimum reaction conditions for maximum percentage swelling (7470.23%) were time (min)=60; vacuum (mmHg)=450; pH=7.0; solvent (mL)=27.5; [APS] (mol L^{-1})=0.306 $\times 10^{-1}$; [AA] (mol L^{-1})=0.291 $\times 10^{-3}$ and [hexamine] (mol L^{-1})=0.356 $\times 10^{-1}$. The semi-interpenetrating networks (semi-IPNs) were converted into IPNs through impregnation of polyaniline chains under acidic and neutral conditions. Fourier transform infra-red spectroscopy (FTIR), thermogravimetric analysis (TGA) and scanning electron microscopy (SEM) techniques were used to characterize the semi-IPNs and IPNs. Synthesized semi-IPNs and IPNs were further evaluated for moisture retention in different soils, antibacterial and biodegradation behavior.

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1. Introduction

Graft copolymerization of vinyl monomers onto polysaccharides is an efficient method for the preparation of super-absorbents. Several attempts have been made in the past to attain the best properties by grafting synthetic polymers onto natural backbones [1,2]. A superabsorbent is the materials that can uptake and conserve large volumes of aqueous fluids owing to its unique 3D hydrophilic network even under pressure. Super-absorbents have attracted attention from all over the world and found extensive application in various fields, such as agriculture [3,4], hygienic products [5], wastewater treatment [6,7], catalyst supports [8] and drug delivery systems [9]. Smart polymeric materials have significant roles in health care especially for wound treatment/protection because of hydrophilicity, biocompatibility, non-toxicity and biodegradability. Such biomaterials can be used to replace certain parts of living system or to function in intimate contact with living tissues [10]. Because of such properties biomaterials are of great importance in tissue engineering. Since the presence of pathogenic microbes like bacteria (*Staphylococcus* sp) and fungi (*Candida* sp) are quite natural on wound infection, wound burn or fresh skin wound, therefore, it

is important to evaluate the biocompatibility of the materials and efficacy for their antimicrobial behavior before their recommendation for health care purposes. Smart dressings have become a very interesting research field from healthcare point of view. Numerous studies have proved that moist wound dressings are best for wound healing [11–14].

In modern agriculture and horticulture, hydrogels are used to enhance both the nutritional and water status of plants. Sandy soils are characterized by low water-holding capacity. Excessive drainage of rain and irrigation water below the root zone leads to poor soil-percolation of water and fertilizer gets washed away. Low soil moisture content critically restricts the seed germination and plant development. Hydrogels have been successfully used as soil amendments in agriculture and horticulture industry in order to improve the water-holding capacity and/or nutrient retention of sandy soil [15,16] as comparable to clay or loam [17]. Superabsorbent hydrogels have the potential to influence soil permeability [18], density, structure, texture [19], evaporation [20] and infiltration rates of water through the soils [21].

Guar gum known as Indian gum is a non-ionic galactomannan gum and is superior to non-degradable matrices. The biodegradability of most matrices is dependent on hydrolytic degradation [22] and enzyme catalyzed breakdown [23].

Because of high water content, flexibility and biocompatibility, hydrogels resemble human tissues and are used in wide

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variety of biomedical applications. Since there exists some electrical impulse in all living beings, therefore, the purpose of present study is to develop a biomaterial with electro-conductive properties and better biocompatibility. The present communication includes the study of moisture retention, biodegradability and antibacterial behavior of synthesized hydrogels. The main novelty of present work is to explore the use of natural resources by chemical modifications and their usefulness in broader prospective.

2. Materials and methods

2.1. Materials

Guar gum was purchased from Sigma Aldrich. Ammonium persulfate (APS), hexamine, acrylic acid (AA) and acetone were purchased from Loba-Chemie Ltd. India. Aniline (ANI) and hydrochloric acid (HCl) were procured from Merck, India. *Staphylococcus aureus* (MTCC 737); Gram positive and *Escherichia coli* (MTCC 739); Gram negative were used as test microorganisms for the study of antibacterial activity. Different soils samples were collected from the Solan (H.P.) India for the study of moisture retention. In case of biodegradation studies under soil burial and composting methods, soil and compost were collected from Hostel No. 7 garden and sewerage plant of NIT Jalandhar (Punjab), respectively.

2.2. Sample preparation

2.2.1. Synthesis of Ggum-cl-poly(AA)

Polymer matrix composed of Ggum-cl-poly(AA) was prepared by using ammonium persulfate as an initiator and hexamine as a cross-linker under vacuum. Guar gum (1.0 g) was mixed in 20 mL of distilled water with calculated amount of APS as an initiator and acrylic acid (AA) as monomer followed by addition of hexamine with continuous stirring. Reaction mixture was continuously stirred to get homogeneity. Reaction was carried-out under vacuum for fixed time period. Different reaction parameters such as monomer concentration, initiator concentration, cross linker concentration, polymerization time, reaction temperature, amount of solvent, vacuum and pH of reaction mixture were optimized to get the candidate polymer with maximum water uptake capacity. Homopolymer was removed through extraction with water and the product obtained was dried in the hot air oven at 50 °C.

2.2.2. Synthesis of Ggum-cl-poly(AA-ipn-ANI)

Ggum-cl-poly(AA-ipn-ANI) was synthesized under vacuum using Ggum-cl-poly(AA) as a flexible matrix. Ggum-cl-poly(AA) (1.0 g) was added to the aqueous medium containing aniline in a reaction flask. The resulting solution was kept for 16 h at room temperature, which resulted in absorption of aniline monomer in the swollen Ggum-cl-poly(AA) matrix. The light brown color of the reaction solution changed to light green on slow addition of thermal initiator APS. Reaction was carried-out under pre-optimized reaction conditions (Section 2.2.1). The resulting product was washed with 1-methyl-2-pyrrolidone (C₅H₉NO) in order to remove homopolymer. Finally, the product was dried in hot air oven at 50 °C. Optimization was done with respect to aniline concentration under acidic and neutral medium and percentage swelling was calculated as per the earlier reported methods [24,25].

2.3. Swelling behavior of the sample

The swelling behavior of the synthesized sample in distilled water has been investigated for 16 h. At definite time intervals, the samples were wiped and weighed. The process was repeated till

equilibrium was achieved. The percentage swelling was calculated as per the earlier reported methods [25].

2.4. Characterization

Fourier transform infrared (FTIR) spectra of backbone and cross linked samples were recorded by Perkin Elmer FTIR spectrophotometer using KBr pellets. FTIR spectra of the samples were taken in the range of 400–4000 cm⁻¹ with 2 cm⁻¹ resolution. TGA/DTA/DTG studies of the synthesized samples were done on TG/DTA 6300, SII EXSTAR 6000 in air at a heating rate of 10 °C/min. Scanning electron micrographs of the candidate polymers were taken on a LEO-435VF.

2.5. Antibacterial property

Two bacteria were used as test microorganisms in this study i.e. *S. aureus* (MTCC 737) and *E. coli* (MTCC 739). The medium used for growing bacteria was universal nutrient agar. Briefly, agar plates were seeded with test microorganisms and kept for 30 min until medium was solidified. Test samples of particular dimensions were placed on the solidified agar and plates were incubated at 37 °C for 24 h. The inhibition zone appeared around the test samples and was measured in millimeters which were recorded as the antibacterial effect of synthesized samples.

2.6. Retention of water in different soils

Soil texture and organic matter are the key components that determine soil water holding capacity. Soil such as silt and clay consisting of smaller particle size and larger surface area has greater water holding capacity. Sand in contrast has large particle size which results in smaller surface area and low water holding capacity. In present study, different semi-interpenetrating and interpenetrating networks (1.0 g each) were well mixed with known weight (20 g) of different types of crushed and dry soils and were kept in pre-weighed ventilated paper cups. Known volume of distilled water was slowly added to each test sample and initial weight of each sample was recorded. The test samples were maintained at ambient temperature and were weighed at definite time interval i.e. after every 12 h till no detectable weight loss was observed [26].

2.7. Biodegradation of Guar gum based superabsorbent

2.7.1. Soil burial method

Guar gum and synthesized IPNs were subjected to biodegradation studies using soil burial method. Samples under investigation were buried in the soil and water was supplemented every day to replenish the drying by evaporation. Samples under investigation were kept at ambient temperature. Weights of test samples were taken at a regular interval of 7 days. Biodegradation of the test samples was monitored at different stages of breaking down using FT-IR techniques. The percentage weight loss after particular time interval was determined as follows [27]:

$$B_s = \frac{W_i - W_f}{W_i} \times 100 \quad (1)$$

where W_i is the initial weight and W_f represents the weight of IPN after each interval.

2.7.2. Composting method

Guar gum and synthesized samples were subjected to biodegradation using composting method [28]. Test samples were buried in the compost containing the varieties of actively growing and well balanced population of mixed microbial species. The microbial species in the compost were regularly fed with the aqueous

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