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ORIGINAL ARTICLE

Physically crosslinked poly(vinyl alcohol)-hydroxyethyl starch blend hydrogel membranes: Synthesis and characterization for biomedical applications

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Abstract Poly(vinyl alcohol), PVA is a polymer of great importance because of its many appealing characteristics specifically for various pharmaceutical and biomedical applications. Physically crosslinked hydrogel membranes composed of different amounts of hydroxyethyl starch (HES) in (PVA) and ampicillin were prepared by applying freeze–thawing method. This freezing–thawing cycle was repeated for three consecutive cycles. Physicochemical properties of PVA–HES membrane gel such as gel fraction, swelling, morphology, elongation, tensile strength, and protein adsorption were investigated. Introducing HES into freeze–thawed PVA structure affected crystal size distribution of PVA; and hence physicochemical properties and morphological structure have been affected. Increased HES concentration decreased the gel fraction %, maximum strength and break elongation. Indeed it resulted into a significant incrementing of the swelling ability, amount of protein adsorption, broader pore size, and pore distribution of membrane morphological structure. Furthermore, an increase in HES concentration resulted in better and still lower thermal stability compared to virgin PVA and freeze–thawed PVA. The maximum weight loss of PVA–HES hydrogel membranes ranged between 18% and 60% according to HES content, after two days of degradation in phosphate buffer saline (PBS), which indicates they are biodegradable. Thus, PVA–HES hydrogel membranes containing ampicillin could be a novel approach for biomedical application e.g. wound dressing purposes.

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1. Introduction

PVA hydrogels have been previously used for numerous biomedical and pharmaceutical applications (Tanigami et al., 1995). PVA hydrogels have several advantages that make them

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proper candidates for biomaterials. Some of these advantages include their non-toxic, non-carcinogenic, biocompatible, bioadhesive characteristics, excellent film-forming, excellent transparency, and additionally their ease of processing. PVA has a simple chemical structure and its chemical modification is possible using simple reaction as well. Meanwhile, PVA gels possess a high degree of swelling in water or biological fluids and an elastic or rubbery nature structure (Chan, 1999). Because of latter advantages, PVA is capable of simulating natural tissues and can be completely accepted into the body. PVA gels have been applied in different biomedical application sites such as contact lenses, the lining for artificial hearts, wound dressing, and drug delivery applications. Peppas and Merrill (1977a,b) have revealed in their earliest work in considering PVA hydrogels as biomaterials. Generally, hydrogels were achieved by crosslinking process of polymers, which may be done by a chemical reaction (e.g. radical polymerization, chemical reaction of complementary groups, using high energy irradiation, or enzymatic reaction) or by physical reaction (e.g. ionic interaction, crystallization of the polymeric chain, hydrogen bond between chains, protein interaction, or design of amphiphilic block and graft copolymers) (Hennink and Nostrum, 2002).

In recent decades, the need of physical crosslinked gels has been potentially increased, (Van Tomme et al., 2005) to avoid the use of chemical crosslinking agents and reagents. These agents are not only often toxic compounds which can be removed or extracted from prepared gels before application, but also can affect the integrity of the substances when entrapped (e.g. proteins, drugs, and cells). Therefore, the physical crosslinking method has been chosen and preferred comparable with the chemical crosslinking method for most crosslinked polymers' preparation. Several attempts have been done to prepare crosslinked PVA-based hydrogels including radiation crosslinking, (Park and Chang, 2003) chemical reaction with glyoxal, (Teramoto et al., 2001) bifunctional reagents with glutaraldehyde, (Dai and Barbari, 1999) or reaction with borates (Korsmeyer and Peppas, 1981).

Although, an aqueous solution of PVA can form low strength of hydrogel upon exposure to very long storage time at room temperature, but this method did not meet any application requirements, where the mechanical properties are the most important character in hydrogel properties. The earliest attempt for crosslinking of PVA using freezing–thawing method has been pioneered by Peppas (1975). Semi-crystalline PVA gels were prepared by exposing PVA aqueous solution to repetitive freezing–thawing cycles which induced crystallization and result in a network structure, which act as physical crosslinking sites in the network. The freezing–thawing method is regarded the best and the preferred method for obtaining physically crosslinked PVA hydrogel without using any traditional toxic chemical crosslinking agent (Yokoyama et al., 1986). While, the obtained mechanical properties of physically crosslinked PVA hydrogel are tunable structure and can be adjusted by the molecular weight and concentration of PVA or the cycle number of freeze–thaw method. Many polymers have been previously blended to PVA to meet such clinical demands or sometimes to develop a polymeric system suitable for specific biomedical applications, such as drug delivery application, tissue engineering or wound dressing. The blended polymers with PVA are like PVP, (Park and Chang, 2003) chitosan, (Kim et al., 2003a) poly (N-isopropylacrylamide), (Kim et al., 2003b) carboxymethyl chitosan, (Zhao et al., 2003) alginate, (Kim et al., 2008) and dextran (Cascone et al., 1999; Fathi et al., 2011).

Hydroxyethyl starch, (HES) is a synthetic polymer prepared by reacting naturally occurring amylopectin with ethylene oxide resulting in hydroxyethyl groups being added to oxygen at different carbon positions at glucopyranose unit C2, C3, or C6 to be in the final form of α-1,4-linked D-glucopyranose residues (Kalhorn et al., 1984). HES has valuable medical applications e.g. as blood plasma volume expander polymers (Deitrich, 2001). Leukapheresis agent, as cryo-preservative (Kalhorn et al., 1984), as polymer drug delivery, (Kamoun and Menzel, 2012) and as blood isotonic electrolyte solutions, which further evidenced its non toxicity, biodegradability, and biocompatibility with the human body.(Dorothee et al., 1998) Thus, HES has been chosen to incorporate with PVA membranes due to its unique biomedical characteristics mentioned earlier, additionally its appealing intrinsic properties e.g. high hydrophilicity, abundance natural sources, and low cost polymers compared to other polysaccharides e.g. sodium alginate and dextran, which have been previously blended with PVA membranes.

In the light of such contributions, the blended HES with PVA hydrogel has not been previously reported yet, and in this work the results of PVA–HES blend membrane based hydrogels are explained in detail for the first time in the literature. PVA–HES blend gel membranes were prepared and entanglement physically using freeze–thaw cycle method at high concentrations of PVA (10%, w/w) and high HES contents (0%, 25%, 33%, 50%, 65%, and 75%, w/w). The PVA–HES blend gel membranes were characterized by Fourier transformer infrared (FT-IR), scanning electron microscope (SEM), differential scanning calorimetry (DSC), and thermal gravimetric analysis (TGA). In addition, the physicochemical properties of gel membranes e.g. gel fraction, swelling behavior, maximum tensile strength, protein adsorption, and protein release profile have been assessed for wound dressing polymeric membrane materials.

2. Experimental

2.1. Materials

PVA (typically average M_w = 72,000 g/mol; 98.9% hydrolyzed) was obtained from Biochemica, Germany. HES (average M_w = 130,000 g/mol as determined by GPC, and DS = 0.5), albumin from bovine serum (BSA, fraction V, minimum 96% electrophoresis, nitrogen content 16.2%), and ampicillin sodium salt were purchased from Sigma–Aldrich Chemie GmbH, Steinheim, Germany. Folin & Ciocalteu's phenol reagent (FC, 2 N with respect to acid), was exported from Park Scientific Limited, Northampton, UK. Distilled water was used throughout this research. All other chemicals were used without any further purification.

2.2. Instrumental analysis and measurements

2.2.1. Preparation of PVA–HES hydrogels

PVA–HES hydrogel membranes were prepared by freezing–thawing (F–T) cycle according to the reported procedure of (Peppas and Stauffer, 1991). Briefly, aqueous solution containing 10% (w/v) PVA and 1.5% (w/v) HES and 20 mg of ampi-

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