World Applied Sciences Journal 12 (7): 929-931, 2011
ISSN 1818-4952

Preparation and Characterization of Crosslinking Monomers Grafted Gelatin Films by Photocuring

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Abstract: Thin films of were prepared by casting and its tensile properties like tensile strength (TS) and elongation at break (Eb%) were studied. The gelatin films were procured with two types of monomers such as 5% ethylene glycol (EG) and 5% ethylene glycol dimethacrylate (EGDMA) to increase the tensile properties. Five percent of monomer solutions were prepared in MeOH along with 2% photoinitiator; Irgacure-651. Soaking time and UV-radiation intensities were optimized with extent of polymer loading (PL) and tensile properties of the cured films. Comparing the properties of EG and EGDMA treated gelatin film, EG showed the best performance. The EG-cured gelatin film with 5 min soaking time showed the highest tensile strength 47.3 MPa and elongation at break 28.4%. The water uptake was determined for raw film 500.13%, EG grafted gelatin film 375.34% and EGDMA grafted film 412.87%.

Key words: Gelatin film · Ethylene glycol · Ethylene glycol dimethacrylate · Photocuring · UV radiation

INTRODUCTION

Gelatin is a heterogeneous mixture of water-soluble proteins of high average molecular mass not found in nature but derived by hydrolytic action from collagen, a protein of mammal external protective tissues, by boiling skin, tendons, ligaments, bones, etc., with water. Commercially gelatin is presented as a colorless or slightly yellow, transparent, brittle, practically odorless, tasteless sheets, flakes or coarse powder. Their uses include not only food (confectionery, jellies and ice cream) and pharmaceutical technology but also manufacturing of rubber substitutes, adhesives, photographic plates and films, matches and clarifying agent [1-4]. Ethylene glycol is a colorless, viscous and hydrophilic liquid capable of forming polymer. Multifunctional unsaturated acrylic monomer is used in the treatment of the polymer film to reduce the UV-radiation intensities to obtain optimum properties or to achieve an increased cure state at the same UV radiation intensities [5]. Photo curing provides some advantages like low energy consumption, ambient temperature operations, no emission of solvents, reduce reaction time, enhanced product quality [6]. The extent of grafting and homopolymer formation was found to depend on gelatin, amount of initiator, monomer concentration, reaction temperature and reaction time [7]. The present study focused on the preparation of thin gelatin films and its tensile properties of EG and EGDMA cured gelatin films with dissimilar soaking time and UV-radiation intensities. The grafted and raw gelatin films were then subjected to water uptake.

MATERIALS AND METHODS

Pharmaceutical grade gelatin was collected from Global Capsules of Opsonin Pharma Ltd. Bangladesh. Ethylene was supplied by Merck, Germany and was used without further purification. Photoinitiator (Irgacure- 651) was supplied from Ciba-Geigy, Switzer land. Methanol was used and obtained from Merck, Germany. Distilled water was used as reaction medium.

Preparation and Characterization of Gelatin Films: Granules of gelatin (8 gm) were dissolved in 200 ml of distilled water and heated for 2 h at 90°C at normal pressure. The gelatin solution was cast on to release film (silicon cloth) covered frame mounted on flat glass plate for film formulation and then dried in an oven for 8 h at 70°C. The dried films (about 0.30 mm thickness) thus prepared were peeled off and cut into small pieces

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Table 1: Polymer loading (PL) and Tensile properties (TS, Eb) at different soaking times against number of UV pass

		EG treated film (Soaking time in min)					EGDMA treated film (Soaking time in min)				
No. of UV passes	Properties	2	3	5	7	10	2	3	5	7	10
10	PL (%)	2.14	2.8	3.4	3.1	2.8	1.5	1.9	2.1	2.6	2.2
	TS (MPa)	28.80	32.1	36.3	32.1	31.0	26.1	26.7	28.2	29.6	25.6
	Eb (%)	7.10	11.3	16.2	13.4	9.8	6.7	9.5	11.3	14.8	11.6
15	PL (%)	2.40	3.2	4.3	3.6	3.1	1.8	2.9	3.8	5.2	4.4
	TS (MPa)	30.70	32.8	42.3	36.5	33.4	30.5	34.7	36.4	41.5	37.8
	Eb (%)	10.20	13.7	20.5	17.4	15.1	12.2	14.1	18.4	24.5	21.2
20	PL (%)	3.50	4.9	6.5	5.7	4.6	1.6	2.8	3.2	4.8	3.6
	TS (MPa)	31.80	36.5	47.3	39.2	34.8	30.3	32.6	34.3	38.2	35.4
	Eb (%)	12.40	18.6	28.4	23.7	18.5	11.4	16.5	18.2	21.3	15.6
25	PL (%)	2.70	3.8	5.6	4.8	3.9	1.4	1.9	2.8	3.9	3.2
	TS (MPa)	29.70	34.1	43.3	36.5	33.7	28.0	30.1	32.2	35.3	31.4
	Eb (%)	9.20	12.4	20.1	16.3	12.9	10.6	12.1	15.3	19.7	13.8

(50 × 10 mm) using conventional scissors. Two types of formulations; 5 % EG (F1) and 5% EGDMA (F2) along with 2% photoinitiator in methanol was prepared. The films were soaked into these formulations for dissimilar soaking times (5-60 min) and irradiated under UV-radiation of difierent intensities (10-25 passes) using UV machine (IST Technik, Germany). The polymer loading (PL) of difierent soaking formulations with the film was determined on the basis of weight gained by the film after the entire treatment process. The tensile properties of the cured films were measured with Universal Testing Machine (Hounsfield Series S Testing Machine, UK) with a load range of 500N, crosshead speed 2 mm/min, gauze length 20 mm and efficiency within ±1%. The water uptake of the EG cured and untreated films was periodically (5- 60 min) monitored.

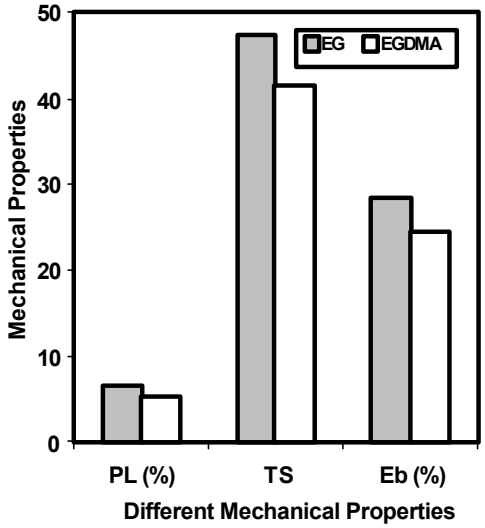

Fig. 1: Comparison of the different mechanical properties of EG and EGDMA grafted gelatin films

RESULTS AND DISCUSSION

Optimization of Soaking Time: The results of PL, TS and Eb are cited in Table 1. The PL, TS and Eb values for F1 and F2 formulations increases with increasing soaking time up to 5 and 7 min, respectively and then decrease with increasing soaking time. It is observed that the maximum PL value for F1 formulation is 6.5% at the 20th UV pass for 5 min soaking time and the maximum PL value for F2 formulation is 5.2% at 10th UV pass for 7 min soaking time. Soaking increases the cross- section area of the film by swelling. As a result more monomer can diffuse into the gelatin film. But in higher soaking time, the film becomes twisted, shrinkage [8]. For F1 formulation, the highest TS and Eb are 47.3MPa and 28.4% respectively at 20th UV pass for 5 min soaking time and for F2 formulation the highest TS and Eb values are 41.5 MPa and 24.5% at 10th UV pass for 7 min soaking time. The initial increase in TS may be due to the increased swelling and leads to an increased diffusion of the monomer into the reaction sites and the amount of curing increase. The reason behind the decrease in TS at higher soaking time may be the homopolymerization of monomer at higher concentration. The inherent character of the film appears to be superior with 20 min soaking time as compared to other soaking times.

Comparison of Tensile Properties Between the EG and EGDMA Grafted Gelatin Films: The tensile properties of EG and EGDMA grafted gelatin films are plotted in Fig. 1. It is observed that EG grafted gelatin film shows 6.5% PL, 47.3% TS and 28.4% higher Eb value than EGDMA treated film. EG is a low molecular weight compound than EGDMA. So, it is possible for EG to make a denser

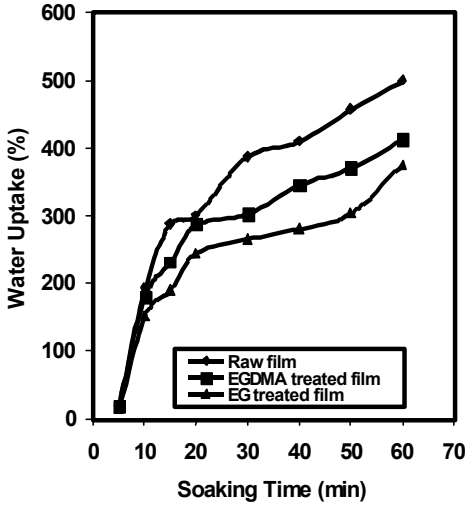

Fig. 2: Water Uptake of raw, EG and EGDMA treated gelatin films

crosslinking with gelatin than EGDMA. Another important reason behind the higher mechanical properties of EG is that the radical formation pattern of EG. In EGDMA only acrylic group goes into radical formation whereas in EG both the methylene and hydroxyl group have the chance to go into radical formation in photocuring procedure. This also helps to form more crosslining between gelatin and EG than that of EGDMA. EG of same concentration (5%) gives better mechanical properties at 5 min soaking. On the other hand same concentration of EGDMA gives the highest properties at higher soaking time of 7 min. So, it can be concluded that EG grafted film gives better mechanical properties than EGDMA grafted gelatin film.

Water Uptake Behavior: Water uptake (%) of raw gelatin, F1 and F2 treated gelatin films are plotted in Fig. 2. From the Fig. 2 it is clear that water uptake of the films is faster at fast few minutes and then it became show or either attains almost. The water uptake percent after 60 min is 500.13% for raw film, 375.34% for EG treated film and 412.87% for EGDMA treated film. The lower water absorption of cured gelatin is due to the crosslinking of monomers with gelatin. More crosslinking lowers the void space within the polymer structure to get into the water in it. EGDMA treated film showed greater water uptake than that of EG. EGDMA is a comparatively larger molecule than EG, it may provides more intra and intermolecular space in the grafted polymer structure for water absorption.

CONCLUSION

The TS and Eb of the raw gelatin film are satisfactory but the enhancement of TS and Eb of the treated gelatin film is significant. The best formulation for grafting is identified for ethylene glycol monomer. The present investigation deals with the measurement of impact of water to find out the conditions in which the developed gelatin film is completely degradable so that the treated gelatin film may be more suitable to use in all possible and desirable purposes. At the same time these products can be easily decomposed after use without posing any threat to the environment.

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