Thermophysical Study of Chitosan-Starch-Glutaric Acid Film in Acetic Acid-Water Mixtures

Virpal Singh *

Department of Chemistry, Constituent Government Degree College Bhadpura Nawabganj, Bareilly M J P Rohilkhand University, Bareilly - 243006 (U.P.), India

Editor's note: Crosslinking chitosan/starch blends using glutaric acid can result in the formation of films with a variety of potential applications, including the removal of heavy metals. Singh examined the effects of crosslinking on the thermal stability of chitosan/starch blends prepared with various concentration ratios. According to the reported DSC and TGA thermograms, both pure starch and pure chitosan, as well as their blended films, show lower stability compared to the crosslinked blended films, which demonstrate significantly greater stability.

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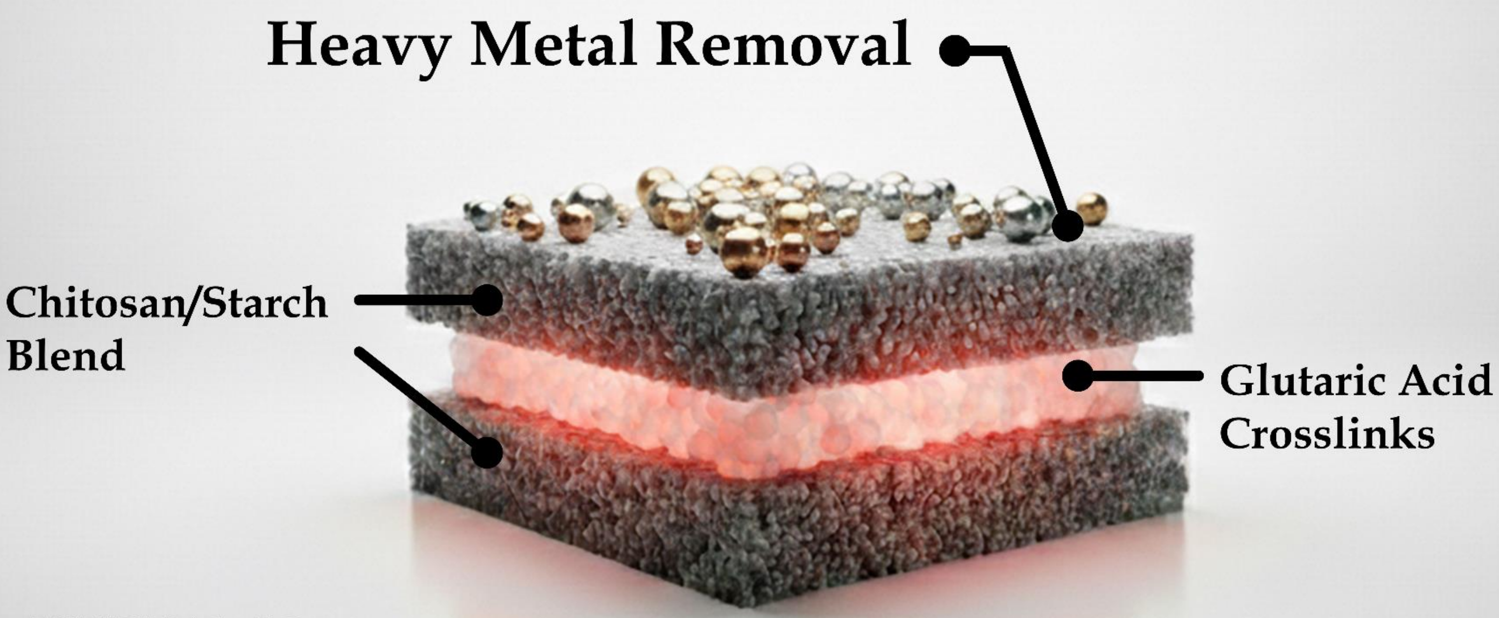

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Original Research

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Virpal Singh *

Department of Chemistry, Constituent Government Degree College Bhadpura Nawabganj, Bareilly M J P Rohilkhand University, Bareilly - 243006 (U.P.), India

Abstract

This study focuses on the synthesis of crosslinked films using chitosan and starch, with glutaric acid serving as the crosslinking agent. The concentrations of chitosan and starch varied simultaneously, ranging from 0.1/0.9 to 0.9/0.1, while the crosslinking concentration of glutaric acid was fixed at 1%. The crosslinked films were characterized using Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (TGA). These blended films are suitable for controlled drug delivery and mass transport processes, as both polymers are biodegradable. The DSC and TGA thermograms reveal that pure starch and pure chitosan, as well as blended chitosan/starch films, exhibit lower stability compared to the crosslinked blended films, which demonstrate greater stability. Additionally, the analysis indicates that the crosslinked blends are miscible and have intermediate thermal degradation properties. Overall, the results from the DSC and TGA analyses suggest that the crosslinked blended films exhibit improved thermal stability, particularly with higher compositions in ascending order. Thermal analysis provides valuable insights into the stability of these films, making them ideal for applications in controlled drug delivery and the removal of heavy metals.

Keywords: Chitosan; Starch; Crosslinked blended films; TGA; DSC.

1. Introduction

There are many polysaccharides in nature, such as alginate, collagen, gelatin, cellulose, chitosan, and starch [1]. Chitosan, (1,4)-[2amino-2-deoxy-β-D-glucan], is a natural derivative of chitin, obtained by its partial deacetylation. Chitosan has an amine side group, which is responsible for its polyatomic character, and formation of well-known intermolecular complexes with carboxylic acid and poly carboxylic acid. Chitosan is inert, hydrophilic, biocompatible, and biodegradable [2]. Several researchers have reported the preparation of membranes or films [3], beads [4, 5], and nanoparticles [6-8] for use in various fields such as metal-ion separation, gas separation, reverse osmosis, ultra-filtration, drug release [9, 10], wound healing [11], pervaporation, for affinity purification and packaging [12, 13]. It possesses antimicrobial properties and absorbs toxic metals.

* Corresponding author.
Email address: singh_veer_pal@rediffmail.com (V. Singh)

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Starch can be used for making blends with other polymeric materials. Starch is a water-swellable excipient in nature. The main differences between starch and chitosan are the glucoside linkage: α (1, 4) for starch and β (1,4) for chitosan, and the hydroxyl group of the second carbon is replaced by the amine group. Commercialized starches are from cereals such as corn, wheat, rice, and from tubers or roots, potato, sweet potato, and tapioca starch. The appearance and properties of the starch are varied on species. Starch, a polymer of α-D-glucose, consists of two forms of glucose polymers, linear (amylose) and branched chain (amylopectin).

Starch occurs in many plant species in the form of spherical granules. Amylose is a linear homopolymer of α (1, 4)-linked glucose with a degree of polymerization of ~1000. Amylose makes up ~35% of starch (range of 11-36% depending on plant and organ), and amylopectin is a highly branched form of "amylose" which has α (1, 6) glycosidic linkage [14]. It can form complexes with fat or iodine because the core of the amylose helices is a hydrophobic molecule. Amylopectin is large size polymer molecule of starch, which is the main component of the starch granules. It is highly branched, and 4-5% of its glucose monomers contain a 1-6 linkage [15, 16]. Keeping in view the above aspects, the present work aims to synthesize chitosan-starch films using glutaric acid as a crosslinking agent.

2. Experimental

2.1. Materials

Chitosan, high molecular weight, having viscosity 800 cp, is supplied by Sigma Aldrich (Germany). Corn starch is procured from Himedia (India), and acetic acid (99.5%) is purchased from Merck (Germany). Glutaric acid is purchased from Loba Chemie (MW= 132.11, purity 99%, and melting point 95-98 ℃). Double-distilled water is used for the preparation of solutions.

2.2. Preparation of crosslinked chitosan-starch films

For the preparation of films, the casting/solvent evaporation technique is employed. The solutions of chitosan/starch are prepared in different weight ratios (0.1/0.1 to 0.9/0.1), separately. A known quantity of chitosan is weighed using a Citizen balance with a precision of 10^{-1} mg and dissolved in 2% acetic acid solution at room temperature (25 ± 2 ℃) with continuous stirring for three hours. Whereas, starch solution is prepared by dissolving it in distilled water at 95 ℃ with constant stirring for 20 minutes. The resultant solution is cooled to room temperature (25 ± 2 ℃). Chitosan- corn starch solution with constant stirring for one hour to obtain a homogeneous solution. 1 ml of 1% glutaric acid solution was used as the crosslinking agent. The resultant solution was cast onto a flat, levelled, non-stick tray to set. Once set, the crosslinked film is dried at room temperature for 72 hours before peeling the film off the tray. The film samples are stored in plastic bags and kept in desiccators at 60% relative humidity for further use. Crosslinked films are designated by four digits (CS19 to CS91). The thickness of the films is measured by a screw gauge. The thickness of the film was found to be 0.105±0.005 mm [17]. The formulations of different synthesized crosslinked films are presented in Table 1.

2.3. Measurements

Thermogravimetric analysis (TGA) was performed from 30 to 500 0C at a heating rate of 10 °C/min under a nitrogen atmosphere. Differential scanning calorimetric (DSC) measurements were carried out in different temperature ranges depending on the composition of the films. The DSC curve was recorded under a nitrogen atmosphere by setting the heating rate to 10 °C/min.

3. Results and discussion

3.1. TGA of Glutaric acid-chitosan-starch films

TGA is a technique applied to study the thermal stability of materials like chitosan and starch films and

Table 1. Composition of the synthesized crosslinked films

Sample No.	Chitosan (g) in 20 ml of 2% acetic acid solution	Starch (g) in 20 ml of distilled water
S	0.0	1.0
CS19	0.1	0.9
CS28	0.2	0.8
CS37	0.3	0.7
CS46	0.4	0.6
CS55	0.5	0.5
CS64	0.6	0.4
CS73	0.7	0.3
CS82	0.8	0.2
CS91	0.9	0.1
C	1.0	0.0

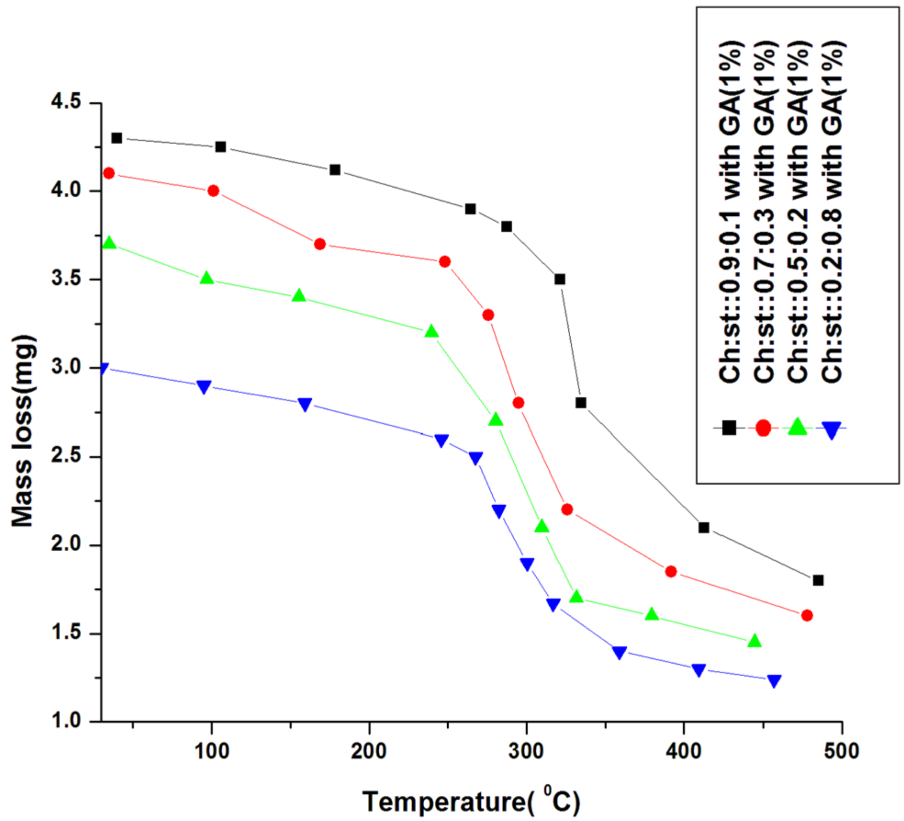

Figure 1. TGA results of starch/chitosan films.

crosslinked films by measuring their weight loss as a function of temperature. For chitosan and starch films, the TGA results in Figure 1 can reveal how temperature affects their composition and structure, indicating the temperatures at which they degrade or lose volatile components. The TGA results clearly indicate that thermal degradation of the chitosan-starch film is reduced after crosslinking. TGA indicates that crosslinked blended films have better thermal stability, which has the highest composition in ascending sequence.

3.2. DSC Analysis of films

The thermal behavior of films (pure chitosan, pure starch, blended, and crosslinked) is analyzed by differential scanning calorimetry (DSC). The DSC thermograms are shown in Figure 2. Chitosan possesses polar groups, and hence, water is absorbed onto the film while hydrogen bonds are formed. In the DSC curve (C), two melting endothermic peaks are observed at around 80 and 170°C. The first endothermic peak corresponds to the loss of surface adhered water, and the second represents the most important loss of absorbed water and other volatile compounds present in the film. DSC scan of starch film (S) shows continuous degradation behavior for the studied temperature range of 50-250 °C. Chitosan-starch film (CS28) crosslinked with Glutaric acid exhibits uniformity of the film, and an exothermic peak corresponding to water evaporation is observed around 100 °C. The melting of film starts at 120°C, and it gets completely degraded at 250 °C. A more stable film is obtained as the percentage of chitosan increases in the blend (CS5). When the concentration of chitosan in the blend is increased, and 1% GA is added before film casting, then more stable films (CS55 and CS73) are obtained, and a similar behavior is exhibited by the DSC scans of both the films (CS55 and CS73).

4. Conclusions

The DSC and TGA thermogram shows that pure starch, pure chitosan, blended chitosan/starch films are

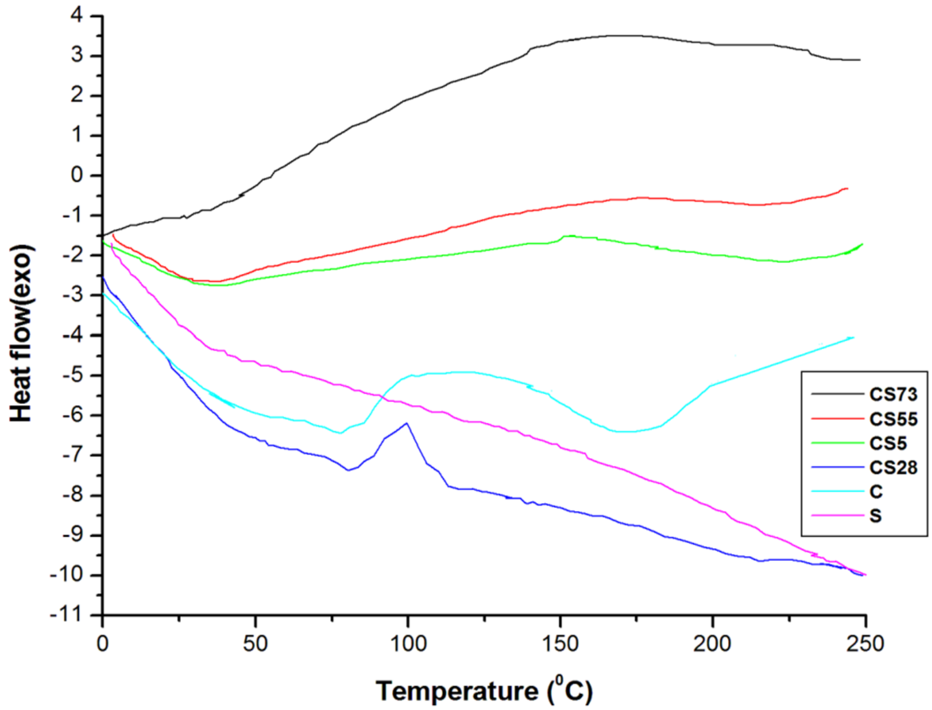

Figure 2. DSC results of starch/chitosan films.

less stable, and crosslinked blending films are most stable, and also indicate that these are miscible with intermediate thermal degradation properties. The DSC and TGA indicate that crosslinked blended films have better thermal stability, which has the highest composition in ascending sequence. The DSC and TGA indicate that crosslinked blended films have better thermal stability, which has the highest composition in ascending sequence.

Conflict of Interest

The authors declare no conflict of interest.

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