

Preparation and characteristic of gelatine/oxidized corn starch and gelatin/corn starch blend microspheres



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ABSTRACT

Combinations of gelatin (G) and oxidized corn starch (OCS) were explored as a new microcapsule composite for single droplet spray drying. The blending solutions property, gel time, transparency and viscosity of G/CS (corn starch) and G/OCS blend solutions were compared at different ratios (10:0;9:1;8:2;7:3;6:4;5:5) and concentrations (1% wt; 3% wt; 5% wt). The drying and dissolution behaviors of composite droplet have been studied using the single droplet drying technique. Possible reaction mechanisms in the composite blend were elucidated by SEM and FTIR techniques. Blends solutions of G/OCS showed longer Gel time, higher transparency and lower viscosity; further displayed faster dissolution rate than that of G/CS under similar conditions. This was attributed to the formed Schiff base between the aldehyde group of OCS and amino group of G which improved the compatibility between G and OCS. All results indicated that the composites could be prepared with excellent properties by G/OCS (6:4) which would overcome some disadvantage such as thermodynamic incompatibility and phase separation by G/CS.

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1. Introduction

Gelatin is a typical wall material for microencapsulation. It can retain the encapsulated core materials from loss and chemical damage during manufacture, storage, and handling process [1,2]. However, the higher price of raw materials used in the production of gelatin when compared with other biopolymers, especially starch and Cellulose [3–6] and the rapid solubility have limited the application of gelatin [7,8]. It is well known that gelatin exhibits good film-forming and gas barrier properties, namely, CO₂ and O₂ [8,9] which is not so feasibly replaced by gelatin-free capsules. Crosslinking agents such as lactic acid, tannic acid, calcium chloride glutaraldehyde, and triethanolamine have been added to gelatin films to increase the water-resistance but the resulting films have been found to be less flexible and transparent [10]. Examining other biopolymers, starch is a popular food ingredient, and it has good film forming behavior [11,12], especially from the viewpoint of cost, environmental concerns and safety. Both gelatin and starch have been widely used, separately, to prepare edible films, so it will

be of commercial and scientific research to access if the composite of the two materials can be used to arrive at a cheaper composite material but yet maintain the film property [13].

Previous research has shown that gelatin and starch are immiscible and that phase separation affects the rheological, processing and mechanical properties of their blends [14,15]. It is well known that starch is a heterogenous material containing two microstructures including amylose (linear) and amylopectin (branched) [16–18]. The hydrophilic OH groups of the native starch molecule tend to form intermolecular and intramolecular hydrogen bonds which limits the usefulness of the materials for certain applications because of instability of viscous solution, low penetration and swelling property [19,20]. Therefore, lower cost gelatin-based blends composites production with conventional technology and without the shortcomings of pure gelatin have both scientific and commercial importance. One possible method to make starch and gelatin more compatible is to increase the carboxyl and carbonyl groups in the starch by oxidation. Oxidized starch has lower gelatinization point, higher transparency, lower viscosity and better stability than that of native corn starch which has been studied in many filed application such as plastic, rubber, film, etc [21,22]. During the oxidation process, the starch is extensively depolymerized and the hydroxyl groups on the carbon rings are oxidized to aldehyde and carboxyl groups [23,24], resulting in changed

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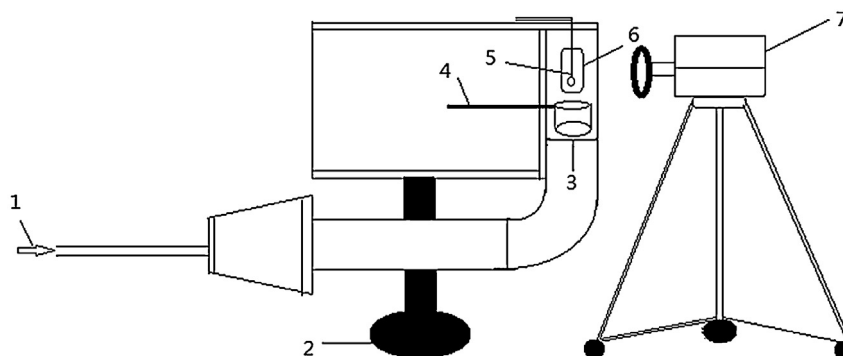


Fig. 1. Glass-filament single droplet drying and dissolution process diagram: (1) air flow (dehumidified air flow with controlled flow rate); (2) solid iron plate support; (3) column area for placing steel meshes; (4) water bath heater; (5) glass filament; (6) drying and dissolution chamber; (7) video camera.

physical and chemical properties. The oxidized starch with the aldehyde and carboxyl groups can then cross link with gelatin improve the characteristics of the overall microstructure.

The aim of this work was to blend gelatin with oxidized corn starch to develop one composite material with better compatibility and stability. Experiments were carried out in a glass-filament single droplet rig, and the established technique was extended to observe not only the drying behavior of a single droplet but also the dissolution behavior of the drying particle. Mechanistic interaction between the G/CS and G/OCS was further examined by flouirier transform infrared (FTIR) analysis and scanning electron microscopy (SEM).

2. Experimental

2.1. Materials

The corn starch (CS), hydrogen peroxide (H_2O_2), hydroxylamine reagent and gelatin (G) were purchased from Sigma Aldrich in Australia. 0.1 mol/L sodium hydroxide (NaOH, Sigma Aldrich) and 0.1 mol/L hydrochloric acid (HCl, Sigma Aldrich) were used to adjust the solution to the desired pH.

2.2. Preparation of oxidized starch

The oxidized corn starch (OCS) was prepared as follows: One known concentration (30% wt) of slurry was prepared by mixing corn starch and distilled water with the pH adjusted to 4.0 [25]. The reactants were heated at 80 °C for 1 h with gentle stirring. After that, the temperature of reaction solution was kept at 50 °C under continuous stirring. Hydrogen peroxide was then added to the reactants with the amount equaling 30% of the weight of the corn starch. After 6 h, the slurry was separated by high speed centrifugation immediately and the obtained oxidized corn starch was dried at 50 °C for 24 h using vacuum oven [26]. The carboxyl group content was estimated based on one chemical titration method [27] without modification with 10% carboxyl group. It was determined by titrating a sample solution with a standard NaOH solution. While the carbonyl group content was determined by reacting carbonyl groups with hydroxylamine reagent and then back-titrating with an HCl solution [28] containing 50% carbonyl group.

2.3. The preparation G/CS, and G/OCS solutions

Solutions of different concentration and ratios of G and CS, G and OCS were prepared with distilled water. Different ratios between G and CS, G and OCS (10:0, 9:1, 8:2, 7:3, 6:4, 5:5) and total concentrations of 1 wt%, 3 wt% and 5 wt%, were prepared. Each solution was

made to 10 g in total mass. The mixed materials were dissolved in distilled water at 80 °C for an initial 30 min at a 100 rpm stirring speed, then for a further 30 min at 500 rpm stirring speed.

2.4. Characterization of G/CS and G/OCS blend solutions

2.4.1. The phase properties of G/CS and G/OCS blend solutions

The phase photos of the G/CS and G/OCS solutions were observed at indoor temperature by visually observing the appearance of samples stored quietly for two weeks in constant temperature and humidity box drying oven. The phase photos were obtained after 2 days and subsequent of 1 week and 2 weeks.

2.4.2. Gel time determination

All samples were prepared according to 2.3. Gel time is conventionally defined to be the period of time from the initial mixing of flow reactants in which the material becomes too viscous to flow or be tooled caused by crosslinking to a pre-cure elastomeric state [29]. The gel time for the G/CS and G/OCS samples solutions were determined by manually tilting the container of the sample at different time intervals; the gel time took as the time duration in which the samples did not flow anymore. It is noteworthy that the intervals for the gel time determination were not a fixed parameter.

2.4.3. Transparency determination

All samples were prepared according to 2.3. A UV spectrum (Spectra Max M2e, Molecular Devices) was used to determine the transparency of varied composites solutions. The tested solution was placed in a 10 mm × 10 mm plastic square sample container and scanned between 300 and 800 nm. When the wavelength was 311 nm, all samples appear the highest adsorption peaks. So the transparency of every solution was measured at the 311 nm wavelength and the detected transmitted signal was presented as%.

2.4.4. Viscosity determination

All samples were prepared according to 2.3. The viscosity of every composite blend was measured using (DV-I Prime, Viscometer, Thermo Fisher Scientific, Australia) at indoor temperature operated at 100 rpm rotation for all samples. The viscometer spindle was immersed into the solution for about 5 min to reach the stable conditions prior to measurements. Each measurement was undertaken in triplicate and the average values were reported here.

2.4.5. Drying and dissolution of the composite particle

The schematic representation of the single droplet spray drying equipment was showed in Fig. 1. The droplet morphology change during drying and dissolution was recorded using a camcorder (Sony PCR-HC36 Camcorder Sony Corporation, Japan) equipped

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