



Synergistic effect of sodium dodecyl sulfate and salts on the gelation properties of acid-hydrolyzed-hydroxypropylated potato starch



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ABSTRACT

Appropriate modifications of starch are necessary to meet the industrial demand for high-quality hard capsule materials as an alternative to gelatin. In the present study, the influence of sodium dodecyl sulfate (SDS) and salts on the gelation properties of acid-hydrolyzed-hydroxypropylated potato starch (AHP) was characterized using dynamic rheological tests. A significant phenomenon was discovered in terms of a synergistic effect between SDS and salts, and was further explored using blue-value and zeta-potential analyses. In particular, the gelation temperature of the AHP pastes increased with increasing concentrations of KCl until there was not enough SDS available to achieve a cooperative effect. Formation of a starch-SDS inclusion complex led to a weakened combination of starch-iodine, and increased the surface charge of starch molecules. Moreover, a decrease in zeta-potential value was observed along with the addition of KCl. Considering the charged sulfate groups of complex starch-SDS, which was provided by SDS, were similar to that of carrageenan, we put forward a hypothesis that the observed synergistic effect of SDS and salts is mainly attributed to the electronic interactions between salt ions. The current study provides a necessary strategy for applying AHP as a gelatin alternative in the manufacturing of hard capsules.

1. Introduction

In the pharmaceutical field, the hard capsule is defined as a storage medium for finely divided blends or formulations containing active pharmaceutical ingredients that are to be delivered orally (Hosny, Al-Shora, & Elmazar, 2002). Gelatin exhibits good gel, film-forming, hygroscopic, and surface active properties, and it has been widely used in the food and pharmaceutical industries (Chang, Raghavan, & Hussain, 1998; Pennings, Kwee, & Vromans, 2006). Although gelatin is the most commonly used food-based material for preparing hard capsules, its availability, higher price, potential safety hazard, religious issues, and quality defects have led to an urgent demand for developing plant-derived materials for preparing hard capsules (Chang et al., 1998; Rd, Zhang, Jobeck, & Bowman, 2001; Zhang et al., 2013). Recently, there has been renewed interest in starch-based hard capsules, and research in this field has more strongly focused on seeking appropriate modifications of starch to meet the industrial demand for new capsule materials with relatively high quality, i.e., a higher solubility (> 20%) in water, low solution viscosity, good film-forming, and the ability to form a gel at ambient temperature. Previous studies showed that dual-

modified (hydrolyzed-hydroxypropylated) starch can be a potential alternative to gelatin in the pharmaceutical capsule industry (Mohammad-Hassan, Nasser, Hossein, Abdorreza, & Karim, 2015).

Hydrolyzed-hydroxypropylated starch would be obtained after acid hydrolysis and further etherification by HCl and propylene oxide, respectively; and it can be applied to dip-molding for hard capsule processing because of its high solubility in water and low tendency for retrogradation (Fouladi & Mohammadi, 2014). Hydrolyzed-hydroxypropylated starch paste with relatively low viscosity can be obtained even in a high concentration, and the capsule film formed is capable to resist retrogradation owing to the hydrophilic property of hydroxypropyl groups (Fakharian, Tamimi, Abbaspour, Mohammadi, & Karim, 2015). Even though hydrolyzed and hydroxypropylated starch has shown certain superior properties as an alternative to gelatin, it still does not have the same useful gelling property to form a gel instantly during process of cooling down to ambient temperature, which plays an important role in the formation of initial form of hard capsules (Fouladi & Mohammadi, 2014). Therefore, the main challenge of replacing gelatin by starch materials for the industrial application of hard capsule manufacturing is to enable the solution of hydrolyzed-

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hydroxypropylated starch to form a gel just above ambient temperature conditions.

Depending on their nature and concentration, salts can cause either an elevation or depression of the gelatinization temperature of starch pastes (Moreira, Chenlo, & Torres, 2011). Indeed, salts have been widely used in the gelation of proteins and polysaccharides. A recent study (Chen, Zhao, Chassenieux, & Nicolai, 2017) showed that the addition of NaCl led to faster aggregation and gelation of a soy protein isolate. Similarly, the gelation temperature and gel strength of a quinoa protein isolate were improved by the addition of CaCl_2 and MgCl_2 during rheological analysis (Kaspchak et al., 2017). In general, chloride salts play a critical role in the gel formation of polysaccharides (Torres, Raymundo, & Sousa, 2014). On the other hand, SDS, which is a well-known anionic surfactant, has been shown to play an important role in modifying the properties of starch paste (Li et al., 2014). SDS has also been widely used in the pharmaceutical field owing to its specific drug interactions and its ability to form a stable micelle system with pharmaceutical ingredients (Kaushal, Rana, Chauhan, Umar, & Chauhan, 2013). Therefore, SDS and salts show good potential for altering the gelation properties of hydrolyzed-hydroxypropylated starch paste in the fabrication of non-gelatin capsules.

To further investigate the influence of SDS and salts on the gelation characteristics of hydrolyzed-hydroxypropylated starch, the aim of the present study was to conduct a dynamic rheology analysis of selected blends of AHP, SDS, and salts. Three typically used salts with different cation types were utilized: NaCl, KCl, and Na_2SO_4 . The influence of salt type and concentration on the thermo-rheological properties of AHP was examined. Moreover, the results obtained through rheology analysis were compared with those obtained by scanning electron microscopy (SEM), blue-value analysis, and zeta electric potential determination.

2. Materials and methods

2.1. Materials

AHP was prepared following the methods described by (Li et al., 2017), and the molar substitution of hydroxypropyl and average molecular weight were 0.132 and 4.20×10^6 g/mol, respectively, as determined by previously reported methods (Johnson, 1969; Sun et al., 2017). All other reagents and chemicals used were of analytical grade, with the exception of SDS, which was chemically pure.

2.2. Sample preparation

The AHP were dispersed in distilled water at 20% (m/v) for the rheological test. Three types of salt, i.e., NaCl, KCl, and Na_2SO_4 , were used at 0.075 mol/L based on the concentration of cations, while the SDS was used at 0.008 mol/L. For examination of the effect of salt concentration on the sol-gel transition analysis of the AHP paste, KCl was used at 0.025, 0.050, 0.075, 0.100, and 0.125 mol/L. The resulting mixtures were continuously stirred in a boiling water bath for 30 min.

2.3. Sol-gel transition analysis

The rheological properties of the samples were assessed by small-amplitude oscillatory rheological measurements on a rotational rheometer (DHR3, TA Instruments, USA) equipped with a parallel-plate with a 40 mm diameter and 1.0 mm gap. To avoid dehydration during the experiment, the plate was covered with silicone oil. The linear viscoelastic region (LVR) was determined prior to dynamic viscoelastic measurements, and a strain of 6% within the LVR was selected for the temperature sweep test.

Sol-gel transition analysis were conducted according to the method of Thrimawithana (Thrimawithana, Young, Dunstan, & Alany, 2010) with some modifications, using a frequency of 10 rad/s to evaluate the

viscoelastic properties during gelation and meltdown. Programmed cooling was performed followed by heating between 60 °C and 20 °C at a rate of 2 °C/min. The storage modulus (G') and loss modulus (G'') of the gels were determined as a function of temperature.

2.4. SEM

Approximately 10 g of different paste samples prepared as described above for the rheological analysis was transferred to a 50 mm aluminum specimen box, which was frozen in liquid nitrogen before maintaining at 10 °C for 10 min, and then freeze-dried. Cubes (1 mm) were cut out from the dry preparations and stuck onto microscope stages using disks made of carbonic tape. The samples were then coated with gold palladium using a sputter coater and observed at $300 \times$ and $2400 \times$ resolutions with SEM (s-4800, Hitachi Science Systems, Ltd., Japan) operated at an accelerating voltage of 20 kV.

2.5. Measurement of blue value

The AHP was dispersed in distilled water at 1% (m/v) with addition of SDS (0.024, 0.048, 0.072, and 0.096 g) or salts (0.075 mol/L based on the concentration of cations). After that, the solutions were gelatinized in boiling water for 30 min and then maintained at 30 °C for 30 min. 1.5 mL of the solution and 1 mL of a 0.02 mol/L iodine-potassium iodide solution were mixed, followed by dilution to 100 mL. The mixture was kept at 30 °C for 15 min. The blue value was determined from the absorbance at 660 nm. For comparison, the control solution was prepared following the same procedure but excluding SDS.

2.6. Measurement of zeta electric potential

The AHP was dispersed in distilled water at 20% (m/v), while the SDS or salts were used at 0.008 mol/L and 0.075 mol/L (based on the concentration of cations), respectively. The samples were then continuously stirred in a boiling water bath for 30 min. The AHP pastes obtained were diluted 50-fold in water at 0.4% (m/v) and then transferred to test tubes. Zeta potential was measured using a Malvern Zetasizer system (Malvern Zetasizer Nano ZS, Malvern Instruments Ltd., Worcester, UK).

2.7. Statistical analysis

All measurements of sample characteristics were carried out in triplicate. OriginPro 8.0 (OriginLab Corporation, USA) and SPSS 20.0 (SPSS Inc., Chicago, USA) were used for the spectra drawings and experimental data analyses, respectively. Analysis of variance (ANOVA) was performed to assess the statistical significance of the results. Differences were considered significant at $p < 0.05$.

3. Results and discussion

3.1. Influence of salt type

The sol-gel transition properties of the AHP-SDS-salt complex obtained by dynamic rheological analysis are shown in Table 1 and Fig. 1. The geometric points where the storage modulus (G') and loss modulus (G'') are congruent during cooling or heating are defined as the gelling (T_g) and melting (T_m) points, respectively (Anvari & Chung, 2016). It should be noted that the point where G' equals G'' is not an absolute gelation point owing to its dependence on the frequency used in the experiment, however, this temperature can be regarded as T_g in our paper as the intersection of G' and G'' was nearly the real time of gelation (Winter & Chambon, 1986).

Based on the T_g values, the addition of salt as well as the salt type had a large influence on the gel-forming properties of the AHP paste during the cooling process. As shown in Table 1, the sol-gel transition

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