



Characterisation of spray dried soy sauce powders made by adding crystalline carbohydrates to drying carrier



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ABSTRACT

This study aimed to reduce stickiness and caking of spray dried soy sauce powders by introducing a new crystalline structure into powder particles. To perform this task, soy sauce powders were formulated by using mixtures of cellulose and maltodextrin or mixtures of waxy starch and maltodextrin as drying carriers, with a fixed carrier addition rate of 30% (w/v) in the feed solution. The microstructure, crystallinity, solubility as well as stickiness and caking strength of all the different powders were analysed and compared. Incorporating crystalline carbohydrates in the drying carrier could significantly reduce the stickiness and caking strength of the powders when the ratio of crystalline carbohydrates to maltodextrin was above 1:5 and 1:2, respectively. X-ray Diffraction (XRD) results showed that adding cellulose or waxy starch could induce the crystallinity of powders. Differential Scanning Calorimetry (DSC) results demonstrated that the native starch added to the soy sauce powders did not fully gelatinize during spray drying.

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1. Introduction

Soy sauce is a traditional condiment popularly produced and consumed in East Asian countries, such as Japan, Korea and China. Soy sauce has mainly been used in liquid form in the past, but recently, different types of soy sauce powders have been developed and marketed. Commercially available soy sauce powders are mainly manufactured by spray drying, although other dehydration methods, such as freeze drying and drum drying, can also be used. Powdered soy sauce was firstly used in the soup base of instant noodles. Now the application has expanded to powdered seasoning, frozen food and processed meat (Okayasu & Hamano, 2003).

For spray dried amorphous food powders, including soy sauce powders, stickiness or caking under relatively high humidity or over a long storage period is always an issue. This is because spray drying usually generates powders in amorphous states due to its short drying time. The amorphous glass will start to flow and become sticky when environmental temperature exceeds its glass transition temperature (T_g) at which glass-rubbery transition happens. The most widely used method for solving stickiness problems is to add large molecular weight carbohydrates, like maltodextrin, to increase powder T_g and reduce hygroscopicity.

Maltodextrin has a good solubility and a high T_g . Therefore, it has been widely used as the drying carrier for making instant food powders (Cai & Corke, 2000; Ersus & Yurdagel, 2007; Goula & Adamopoulos, 2008, 2010; Sablani, Shrestha, & Bhandari, 2008; Tonon, Brabet, & Hubinger, 2008). However, due to its amorphous nature, maltodextrin will also become hygroscopic and sticky when exposed to a high relative humidity environment. Identifying alternative drying carriers to address stickiness or caking issues of amorphous food powders is of both academic and industrial interest. According to Cano-Chauca, Stringheta, Ramos, and Cal-Vidal (2005), adding waxy starch or microcrystalline cellulose as a drying aid was able to produce a partial crystalline surface in spray dried mango juice powder. The semi-crystalline powders had reduced stickiness compared to the ones made using only maltodextrin.

Spray dried soy sauce powders containing maltodextrin as the drying carrier are mixtures of NaCl crystals and amorphous carbohydrates, amino acids and proteins (Wang & Zhou, 2012). The salt crystals adsorb moisture only when equilibrium water activity reaches 0.753 (Hartmann & Palzer, 2010). So salt is not likely to cause moisture adsorption and sintering of particles when exposed to low or medium relative humidity. However, the amorphous phase is metastable and liable for moisture adsorption and sintering of particles under low relative humidity. Therefore, to incorporate crystalline or semi-crystalline carbohydrates instead of maltodextrin in order to increase the crystallinity of soy sauce powder has great potential to improve stability. The crystalline carrier may produce a semi-crystalline powder and reduce

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inter-particle powder adhesions. However, due to the insoluble nature of large molecular carbohydrates (i.e. starch and cellulose), preparing feed solutions containing them, as well as reconstitution of the powders, needs to be understood and optimised.

The objective of the study was to explore the addition of waxy starch or microcrystalline cellulose together with maltodextrin as drying carriers to produce soy sauce powders. The crystallinity of soy sauce powders was studied and related to the stickiness and caking behaviour of the powders under testing conditions. The study aimed to improve the stability of spray dried soy sauce powders by providing an alternative approach to the current practice, and lay a foundation for industrial production and application of soy sauce powders.

2. Materials and methods

2.1. Raw materials

Naturally brewed soy sauce was obtained from Kikkoman Pte. Ltd. (Singapore) with a protein content of 10.3% (w/w), carbohydrates of 8.1% (w/w) and NaCl of 16.5% (w/w) and the rest being water. Maltodextrin 10 DE was purchased from Suntop Enterprise (Singapore). Microcrystalline cellulose was purchased from Sigma–Aldrich (Singapore). Corn waxy starch was obtained from National Starch Co. (Singapore).

2.2. Spray drying

A pilot-scale spray dryer (Mobile Minor™, GEA, China) was used in the study. Co-current flow regime and a two-fluid nozzle atomizer were used for the spray drying process. The inlet air temperature was set to 160 °C and outlet temperature was maintained at 75 °C, by adjusting the feed flow rate via a peristaltic pump. The compression air pressure for atomisation was controlled at 2 bars, with an air flow rate of 4 m³/h. Dried powders were collected from the base of a cyclone separator of the drier.

Drying carriers with maltodextrin DE 10 only, mixtures of maltodextrin and waxy starch and mixtures of maltodextrin and cellulose were added into liquid soy sauce respectively with an uniform carrier concentration of 30% (w/v). For the combination of maltodextrin and cellulose/starch, specifically, ratios of cellulose/starch to maltodextrin at (1:5), (2:4) and (3:3) were applied, respectively. All the samples were spray dried into powders and stored in desiccators containing silica gel until being analysed.

2.3. Scanning electronic microscopy (SEM)

Powder samples were mounted on aluminum stubs using double-sided adhesive tape. The sample was then coated with platinum in a sputter coater. SEM was performed using a JSM-5200 SEM system (JEOL, Tokyo, Japan), which was operated at an accelerating voltage of 15 kV. The samples were observed with a magnification of 1000×.

2.4. X-ray powder diffraction

The crystallinity of the powders was identified by using the powder X-ray diffraction (XRD) method. X-ray powder diffraction (XRD, Bruker AXS D8 Advance, Germany) for phase analysis was carried out for identification of crystalline phases, by using Cu-K α radiation ($\lambda = 1.5406 \text{ \AA}$), at 40 kV and 40 mA with a step size of 0.02°. The diffraction followed $a\theta/2\theta$ Bragg–Brentano geometry and the 2θ range was varied from 5° to 50°.

2.5. Stickiness test

Stickiness was determined according to the [Chen and Hosene method \(1995\)](#). A Texture analyzer (TA.XT plus, Stable Micro

System, UK) was used in this study to provide a constant compression force and to measure the tension force. The TA.XT plus library program of dough stickiness was used. The compression force selected was at 40 g. The diameter of the plexiglass probe was 25 mm. The trigger force was set at 5 g. The compression travel speed for the probe was 2 mm/s. The probe reversing speed was 10 mm/s, which was the maximum reversing speed of the texture analyzer. The holding time was 0.1 s. The probe distance was selected as 4 mm. For stickiness measurements, the samples were mixed with glycerol in a proportion of 2 g powder to 5 ml glycerol, forming a homogeneous dough. The dough was placed in the instrument and the test was performed in triplicate.

2.6. Caking test

Caking tests were performed based on the method described previously in [Wang and Zhou \(2012\)](#). Briefly, 4 g of soy sauce powder was placed in a cylindrical plastic bottle, tapped gently to make a flat layer, and then compacted under a Texture Analyzer-XT2i (Stable Micro System, UK) with a load force of 1 kg for 1 min. After that, the compacted powder sample together with the plastic bottle was stored in a desiccator with relative humidity (RH) of 43.2%, at 25 °C for ten days. At the end of the storage period, the powder cake plug was gently taken out of the bottle. A compression test for analysing hardness of the powder cake plug was carried out by using the TA-XTplus Texture Analyzer. The test protocol included trigger force: 0.005 kg, test speed: 1 mm/s, distance: 4 mm, and cylinder probe diameter: 6 mm. The peak force prior to breaking the powder cake plug was used to compare the caking strength of the different powders.

2.7. Solubility analysis

Solubility of all powder samples was determined according to the method of [Cano-Chauca et al. \(2005\)](#) with modifications. Specifically, 50 ml of deionized water was transferred into a 200 ml beaker. Five grams of soy sauce powder was added into the beaker and then the beaker was put onto a magnetic stirrer at 800 rpm, for 5 min at room temperature. The mixture was centrifuged at 3000×g for 5 min to separate the insoluble substances. Then 25 ml of supernatant was transferred into a metal plate and oven dried at 100 °C for 5 h. The solubility (%) was calculated as the weight of dry matters in the supernatants versus the weight of dry matters in the powders.

2.8. Starch gelatinization

Starch gelatinization analysis was performed by using the method described by [White, Abbas, Pollak, and Johnson \(1990\)](#). Using a differential scanning calorimeter (Mettler-Toledo DSC822e, Switzerland), a starch suspension (10% w/w) in cold water was analysed in the temperature range of 25–90 °C with a scanning rate of 5 °C/min. In particular, the presence of a gelatinization endothermic peak was investigated.

2.9. Statistical analysis

One-way ANOVA was conducted for determination of differences between samples using the SPSS 17.0 software. Duncan's test was also employed. A probability level of $p \leq 0.05$ was considered to be significant for all statistical procedures.

3. Results and discussion

3.1. SEM analysis

[Fig. 1](#) shows the SEM micrographs of soy sauce powders produced by adding microcrystalline cellulose and maltodextrin

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