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A Study on Spray Dried Gelatine: Effect of Feed Concentration

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Abstract: The objective of this study was to study the performance of a pilot-scale spray dryer and observed the effect of feed concentration during spray drying of gelatine. The spray drying conditions were manipulated in terms of feed concentration, inlet temperature and feed flow rate. In all experiments, the atomizer rotation and air flow rate were fixed at 23500 RPM and 66 m^3/h. The result showed that if feed concentration of gelatine solution 10 and 15% (w/v), the product in the form of powder would not be obtained. So, the feed concentration 5 and 6% was chosen to analyze the powder of gelatine from spray drying. The quality of spray dried gelatine was compared with the Industrial Food Grade gelatine (bloom strength 151-160).

Key words: Gelatine, spray drying, denaturizing, feed concentration

INTRODUCTION

Gelatine is a protein which is manufactured by denaturizing and partial breakdown of the animal protein collagen. Collagen is the chief protein component in skin, bones, hides and white connective tissues of the animal body (Mark and Kroschwitz, 2003; Othmer, 1979). There are two types of gelatine namely type A and B. Type A gelatine produced by acid processing of collagenous raw material and type B produced by alkaline or lime processing (Mark and Kroschwitz, 2003). Because it was obtained from collagen by a controlled partial hydrolysis and does not exist in nature, gelatin was classified as a derived protein (Mark and Kroschwitz, 2003). On a dry weight basis, gelatine consists of 98 to 99% protein. The molecular weight of these protein structures typically range between 20 to 250 kD (Grobben *et al*., 2008). Gelatine as a product has been used in many food and pharmaceutical applications (Schrieber and Gareis, 2007). For this experiment, the gelatine used was type B from cattle bones. The manufacturing of gelatine started from the slaughter house to the final process of drying. For drying process, the spray drying technique is an alternative used to get the powder form of gelatine. Before this, in the industrial scale of producing gelatine, spray drying technique is rarely used in the industry because it might be lead to lower quality of the gelatine. Thus this study was carried out to investigate the potential of using spray drying technique to produce good quality gelatine and also find the suitable operating conditions in spray drying process. The quality of spray dried food is very much dependent on the spray dryer operating parameters (Chegini and Ghobadian, 2005).

Spray drying was an industrial process used for the continuous production of dry solids in powder, granulate or agglomerate form. Namely, a feed in the form of a solution, suspension, slurry or paste was atomized to form a spray of droplets which were contacted with a hot gas (usually air) flowing in either a co-current, mixed or counter-current direction within a tower (Walton, 2000; Walton and Mumford, 1999). Evaporation of moisture from the droplets proceeds under controlled temperature and airflow conditions to form a dry powder which was continuously discharged from the base of the tower (Walton, 2000). Operating conditions and dryer design depend upon the drying characteristics of the product and required powder specification. The objective of this study is to study the effect of feed concentration of gelatine in spray-dried of gelatine. Gelatine was diluted in hot water at certain concentration. The gelatine solution was then spray dried to get powder of gelatine. The quality of gelatine was then analysed.

MATERIALS AND METHODS

Gelatine solution: For the experiment, the readymade industrial gelatine from Halagel Food Grade with bloom strength 151-160 was used. The gelatine was dissolved in hot water at certain concentration before being dried with spray drying. From literature, Walton (2000) and Walton and Mumford (1999), using a single droplet drying apparatus, the feed concentration was fixed at 15% w/v and dried at a temperature 200°C. For this experiment, firstly, three runs at different concentration were used.

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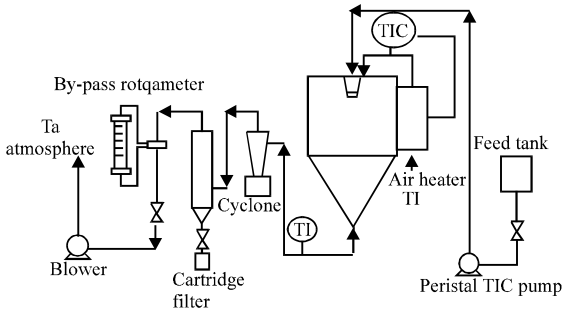

Fig. 1: Schematic of the spray dryer unit (Woo *et al*., 2007)

First run with gelatine concentration 5% (w/v) at 100°C, second run with concentration 10% (w/v) at 100°C and third run with concentration 15% (w/v) at 150°C. The experiments then continued with 5% and 6% (w/v) concentration of gelatine.

Spray drying equipment: Spray drying experiment was undertaken using the APV Pasilac Anhydro AS pilot-scale cyclinder section-on-cone spray dryer unit available at UKM. The cylindrical section is 1 m in diameter and 0.8 m in height. Height of the conical section is 1 m with a 2 inch bottom outlet. The rotating disc atomizer used has four annular orifices (1.7 mm) with a disc diameter of 63.1 and 8.5 mm plate thickness. This atomizer is driven by a rotating motor (Bosch: Scintilla SA) in which the rotation is transmitted to the plate through a set of gears. The feed flow rate is controlled by a peristaltic pump (Heidolph, Pump Drive: PD5201). Powder products are collected in a bottom container from a small cyclone and the top outlet from this cyclone is connected to a cylindrical cartridge filter prior to release into the atmosphere. Hot air is driven by vacuum from a single blower at the outlet and is heated through an electric heater. This air heating setup allows the inlet air temperature to be automatically regulated at the desired set point. However, slight oscillation of the temperature about the set point is inherent in the temperature control. The hot air enters the drying chamber at the top near the atomizer. Temperature sensors were installed at the heater and the outlet pipe after the cyclone to detect the corresponding temperatures. The drying air flow rate is displayed on a bypass rotameter and can be manually controlled by a valve at the inlet of the blower. Figure 1 show the schematic of the spray dryer unit and Fig. 2 shows the picture of spray dry equipment.

Yield calculations: The mass of the dried gelatine powder collected for each experiment from the spray dryer was weighed using a balance. Yield measurements were then calculated based on the mass of dried gelatine powder collected from the spray dryer compared with the solids content of the feed gelatine.

Fig. 2: Gelatin inside the drying chamber

Gel test: The 0.5 g of gelatine was dissolved in 10 mL of hot water (60°C) in a suitable flask and was cooled in refrigerator (5-10°C) for 5 h. Then, the gelatine was check whether there was any formation of gel.

Solubility: The solubility of spray dried powder was carried out by adding 0.5 g of gelatin in 10 mL of distilled water at 60°C. The mixture was agitated in a beaker and then the solubility of the powder in solution was observed whether the powder have low or high solubility.

Scanning electron microscopy: Samples gelatine powders products for SEM analysis were gently tabbed onto aluminium stubs prepared with double-sided tapes and then coated with gold. The images of the spray dried samples were examined using Scanning Electron Microscope (SEM) (SURPA 55VP, Zeiss).

RESULTS AND DISCUSSION

Spray drying experiment: For each of the experiments, the spray drying conditions were manipulated in terms of feed concentration, inlet temperature and feed flow rate. The atomization rotation and air flow rate were fixed at 23500 RPM and 66 m^3 h^{-1}. Table 1 shows the 7 runs undertaken to determine the operational condition to obtain powder of gelatine.

From these experiments, it showed that if the feed concentration of gelatine 10% and 15%, the product outlet was not form in powder form. The product will coagulate and the water cannot evaporate completely. For these concentrations, heavy dripping was observed and the gelatine inside the drying chamber form in fiber or looks like spider nest and these can see in Fig. 2. These happen

Table 1: Operating conditions

Run	Feed concentration % (w/v)	Inlet temperature (°C)	Outlet temperature (°C)	Feed flow rate (mL min^{-1})	Dripping condition	Product form
1	15	150	120	13.90	Heavy	Coagulated
2	10	100	70	13.90	Heavy	Coagulated
3	6	100	70	17.77	Slight	Powder
4	6	120	90	17.77	Rim	Powder
5	5	90	60	17.77	Slight	Powder
6	5	100	70	13.90	No	Powder
7	5	150	100	17.77	No	Powder

Atomization rotation: 23500 RPM. Air flow rate: 66 m^3 h^{-1}

Table 2: Result for gel test and yield

Feed concentration % (w/v)	Inlet temperature (°C)	Outlet temperature (°C)	Feed flowrate (mL min^{-1})	Gelling	Colour	Solubility	Yield (%)
15	150	120	13.90	Yes	White	Low	6.01
10	100	70	13.90	Yes	White	Low	20.18
6	100	70	17.77	Yes	Yellowish	High	23.83
6	120	90	17.77	Yes	Yellowish	Medium	32.22
5	90	60	17.77	Yes	White	High	20.20
5	100	70	13.90	Yes	White	Low	20.37
5	150	100	17.77	Yes	White	Medium	20.93

Atomization rotation: 23500 RPM. Air flow rate: 66 m^3 h^{-1}

because the concentration was high, so the gelatine solution could not be dried completely and the atomizer cannot atomize completely. This was because fibers tend to form in the drier. These cause blockages, as the water, because its high surface tension cannot evaporate quickly enough to prevent gelling taking place (Schrieber and Gareis, 2007). So, to avoid these from happening again, the low concentration of 5% was then selected. Spontaneous drying without having to go through the gelling stage give the gelatine an amorphous form or powder form than the partially crystallized form of classical powder gelatine. For feed concentration of 6%, the product formed powder and have a slight of dripping. Three different inlet temperatures were used during the experiments with the feed concentration of 5%. All three runs for this concentration, formed the dried product in powder form. For inlet temperature 90°C, it have slight dripping condition, while for inlet temperature 100% and 150°C, there were no occurrence of dripping condition.

Yield: The yields were shown in Table 2 and were indications of wall deposition because low yield may be associated with more powder being deposited on the walls of the equipment. From the experimental result, the yields approximately varied from 6.01% to 32.22%. The data showed that the feed concentration of 6% with inlet temperature 120°C produced high yield (32.22%) from others while the feed concentration of 15% with inlet temperature 150°C produced the lower yield (6.01%). The feed concentration of 15% has the lower yield because most of the gelatine solution did not become a powder form but coagulated when dried with spray drying. The solution did not dried completely and induced low yield because most of the gelatine still left behind inside the drying chamber. For feed concentration 5% with inlet temperature 90, 100 and 150°C, yields ranging from 20.2 to 20.93%. From the result, it shows that increasing the feed concentration induced high yield. But, for feed concentration 10 and 15%, low yield was recorded because the feed concentration also affect the spray drying process. From the experiment, it shows that these concentration (10% and 15%) and condition were not suitable for spray dried gelatine.

Gel test: The objective of gel test is to identify the powder of spray dried gelatine will become gel or not. From experiments, all 7 sample powder of gelatine from spray dried become gelling. This result was compared with food grade industrial gelatine. The industrial food grade gelatine, usually is a vitreous, brittle solid that is faintly yellow to white and nearly tasteless and odourless. Table 2 shows the result for gel test. Based on these results, it shows that the condition for feed concentration of gelatine 6% was much better than others. So, this concentration can be studied further to obtain optimum condition to spray dried gelatine.

The most important properties were its gelling and its surface active properties. Gelatine may form a gel when a warm solution of it was cooled to below a certain temperature. However some gelatines, the so-called hydrolysates, do not form gel. The rigidity of the gel depends for a part on certain molecular characteristics of the gelatine; its amino acid composition and its molecular mass composition. Mammal derived gelatine containsapproximately 25% (w) amino acids, fish gelatine contains between 17 and 22% amino acid (warm water fish (tilapia) 22%, cold water fish (cod) 17%) (Grobben *et al*., 2008). Gelatine with low amino acid content has at the

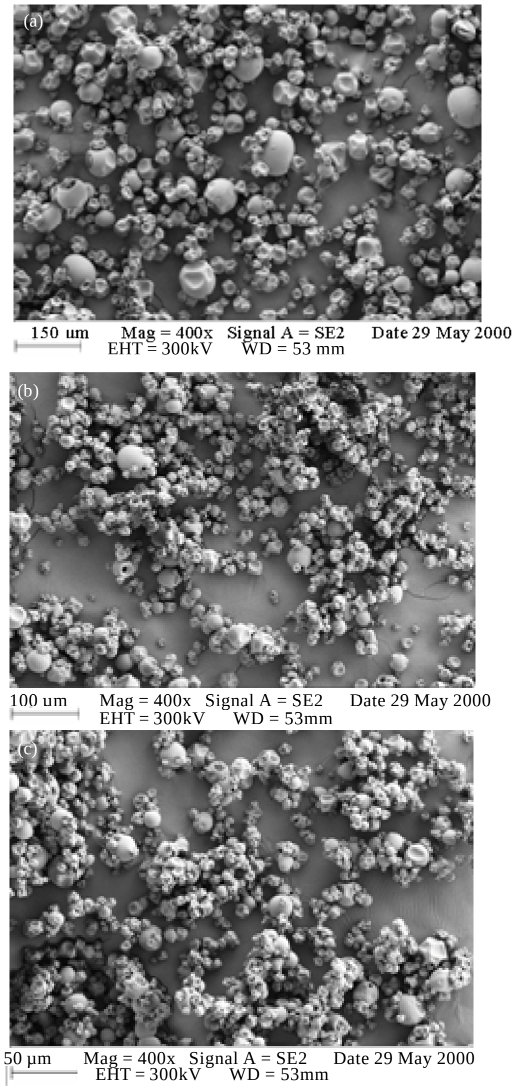

Fig. 3: SEM images of gelatine powders spray dried at 100°C. (a) Feed concentration 10%; (b) feed concentration 6%; (c) feed concentration 5%

same concentration and temperature a much lower gel-strength than gelatine with high amino acid content.

Solubility: Results of Table 2 shows that the powder from concentration of 5 and 6% has higher solubility than 10 and 15% concentration. So, from this result, it shows that the feed concentrations also affect the solubility of the powder.

Particle morphology: Scanning electron micrographs of gelatine powder samples dried at 100 and 150°C indicate that the particles are smooth skin-forming particles and hollow, whether they are prepared from different feed concentration (10, 6 and 5%), as shown in Fig. 3 and 4. In general, but with the exception of high feed concentration and high temperature, the dried particles are spherical in shape (Anandharamakrishnan *et al*., 2007). However, at the lowest temperature a large proportion of particles appear to have caved-in (Anandharamakrishnan *et al*., 2007). From the micrographs in Fig. 4, for the feed concentration of 10%, the particles are more spherical and larger than feed concentration 6 and 5%.

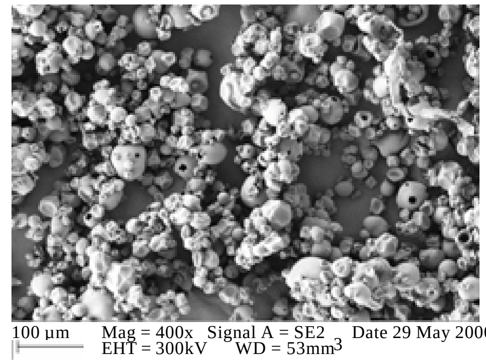

Fig. 4: SEM images of gelatine powders spray dried at 150°C for feed concentration 5%

Figure 4 show the SEM image for gelatine powders spray dried on feed concentration 5 at 150°C. Supposedly, when the drying temperature was increase, the particles will become more spherical and less shriveled (Nijdam and Langrish, 2005). When the drying temperature is sufficiently high, moisture is evaporated very quickly and the skin becomes dry and hard so that the hollow particle cannot deflate when vapor condenses within the vacuole as the particles moves into cooler regions of the dryer. However, when the drying temperature is lower, the skin remains moist and supply for longer so that the hollow particles can deflate and shrivel as it cools (Nijdam and Langrish, 2005). But, from the Fig. 4, it shows that it is not much different with the gelatine powders dried at 100°C. This happen maybe because the process condition that was used for spray dried gelatine is not suitable. So, further study will be carried out to obtain the suitable or optimum condition for spray drying the gelatine.

CONCLUSION

In conclusion, the experimental results revealed that the concentration of the solution and inlet temperature thus affect the product formation. The product in the form of powder would not be obtained if the gelatine feed concentration is of 10 and 15%. Hence, one can say that the feed concentration plays an important role in the spray dried of gelatine. If the concentration of gelatine is very high, the gelatine would not dry completely and a powder form may not be developed. Previous literature Schrieber and Gareis (2007) stated that if spray drying process is to be used, the gelatine solution should be spray dried together with additives. Otherwise low concentration should use. In order to get best result for spray dried gelatine, further study by using a laboratory-scale spray dryer can be employed because the drying characteristics of the product also depend on the spray dryer design and easier to handle rather than using the pilot-scale spray dryer.

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