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# Pilot study of recovery of whey soy proteins from soy whey wastewater using batch foam fractionation



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## ABSTRACT

For industrializing foam fractionation of whey soy proteins (WSP) from soy whey wastewater, a pilot study of 120 L in liquid loading volume was conducted. Firstly the effects of sodium bisulfite concentration, superficial gas velocity and temperature were investigated for increasing the WSP recovery. Furthermore the protein composition of WSP before and after foam fractionation was analyzed. Then the effects of foam fractionation, thermal sterilization and spray-drying on the fraction of soluble proteins, foaming properties, and emulsifying properties of WSP were examined. Finally the up-scaling effect of foam fractionation was analyzed. The results show that the end product of WSP with an effective recovery of  $30.6 \pm 1.5\%$  had Kunitz trypsin inhibitor (the major ingredient), lipoxidase,  $\beta$ -amylase and lectin. The processing procedures, particularly foam fractionation and thermal sterilization, resulted in about 40% decrease in the levels of the food-related properties. The up-scaling effect slightly decreased the WSP recovery and intensified their denaturation.

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# 1. Introduction

Soy whey wastewater (SWW), containing various useful compounds such as proteins (Singh and Banerjee, 2013), polysaccharides (Ray and Rousseau, 2013) and soy isoflavones (Liu et al., 2003; Liu et al., 2013a), is the residual liquid generated after isoelectric precipitation of soy protein isolate (SPI). In industry, the wastewater, yielded at 20 m<sup>3</sup> or more per ton SPI (Liu et al., 2013b), is discharged to the sewage treatment plant in which the compounds are translated into sludge by a biological method. This strategy considerably increases the cost for the production of SPI. However, if the valuable compounds are recovered by using economical methods and reused, then SWW will become a new source of profit, which will have industrial implications to the SPI-producing plants.

In SWW, the proteins called whey soy proteins (WSP) are considered to be the firstly recovered compounds. They possess high solubility in wide pH range, good foamability and good emulsibility (Sorgentini and Wagner, 2002; Palazolo et al., 2005), indicating their promising prospect in food industry. In addition, their first removal from SWW will facilitate the subsequent recovery of soybean polysaccharides which also have wide applications in food industry (Furuta et al., 1998; Nobuhara et al., 2014).

Foam fractionation has great potential in industrial recovery of WSP from SWW. This is because of the fact that WSP, mainly consisting of Kunitz trypsin inhibitor and lectin, have high surface activity and low concentrations in SWW (Wang et al., 2013; Liu et al., 2013a,b) whilst foam fractionation is much more cost-effective in separating a surface-active compound from its highly diluted aqueous solution than other separation methods (Burghoff, 2012; Chen and Parlar, 2013; Khalesi et al., 2013). In addition, a series of experimental researches have confirmed the feasibility of foam fractionation of WSP from SWW (Mukhopadhyay et al., 2010; Jiang et al., 2011; Wang et al., 2013). For instance, Jiang et al. (2011) presented that at the initial WSP concentration of 4.0 g/L, the enrichment ratio reached 8.5 with a high total recovery of 80% using a two-stage foam fractionation technology. Until now, most of the researches have been limited to lab scale and none of up-scaling studies have been reported.

In fact, it is highly necessary to conduct a pilot study to evaluate the efficiency of foam fractionation and the reusability of the product. The two indicating parameters are closely related to the feasibility of industrial recovery of WSP from SWW, but they have not been investigated in most lab-scale experiments in which recovery percentage and enrichment ratio were used as the major indicating parameters (Yang et al., 2008; Mukhopadhyay et al., 2010; Jiang et al., 2011). In up-scaling process, the process efficiency will be

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affected by the enlarged cross-sectional area of the foam fractionation column which will slow down foam drainage (Papara et al., 2009). It will also be affected by the increased air pressure out of a high-power air compressor which will enhance foam drainage via increasing bubble size but decrease the area of bubble surfaces for protein adsorption. In addition, the quality of WSP will be lowered by their denaturation enhanced by the prolonged operation time for air bubbling (Maa and Hsu, 1997). As a conclusion, the pilot-scale experiments of foam fractionation of WSP will be very different from the lab-scale investigation. Based on the above reasons, we have decided to conduct a pilot study of foam fractionation of WSP from SWW in the present work.

In the pilot study, the effects of superficial gas velocity and temperature will be investigated because the enrichment ratio of WSP is highly dependent on the two parameters (Jiang et al., 2011). For saving costs, the pH and the WSP concentration of SWW will not be changed in all the experiments. Bad smell was observed in labscale experiments of foam fractionation, so sodium bisulfite, as a food preservative, will be added to SWW before foam fractionation to keep the product good quality. However, sodium bisulfite will change the ion strength of SWW, by which the foam fractionation performances will be affected (Lockwood et al., 1997), so the effect of its concentration will also be investigated. Subsequently, the variation of proteins composition of WSP in foam fractionation will be analyzed to determine the composition of the end product. In addition, in foam fractionation, the structure of a protein will be changed and irreversible under certain circumstances when the protein adsorbs onto the bubble surface, resulting in the protein denaturation (Barackov et al., 2012). Thermal setrilization and spray drying are indispensable operation units in food processing, but it has have been widely reported for the two thermal processes to affect the properties of the proteins (Wang et al., 2004; Shen and Quek, 2014). Thus in terms of WSP, the effects of foam fractionation, thermal setrilization and spray drying on their food-related properties will be investigated using native WSP as a reference to evaluate the reusability of the end product. The food-related properties will include the fraction of soluble proteins, foaming properties and emulsifying properties, which are essential properties for food proteins, and thus greatly important for the prospect of WSP in food industry (Lam and Nickerson, 2013; Raikos et al., 2004). Finally, the comparison between the end products from pilot scale and lab scale will be made to analyze the up-scaling effect in foam fractionation of WSP from SWW.

#### 2. Materials and methods

#### 2.1. Materials

## 2.1.1. Soy whey wastewater (SWW)

SWW was obtained during the production of SPI by the method of alkali-solution and acid-isolation in Scents Holding Co. Ltd., China. Its initial temperature and pH were  $50 \pm 2$  °C and 4.3-4.5, respectively. It had a total solid content of  $1.87 \pm 0.1\%$  (w/w), mainly consisting of  $16.0 \pm 0.8\%$  total proteins (namely, WSP),  $50.8 \pm 2.1\%$  total carbohydrates, and  $10.3 \pm 0.5\%$  ash. The current concentration of WSP was  $3.0 \pm 0.1$  g/L, lower than that reported by Jiang et al. (2011) owing to technology improvement for SPI production by the company.

# 2.1.2. Native WSP

Native WSP were prepared as the method of Palazolo et al. (2004) with a simple modification. SWW was adjusted to pH 8.0 and centrifuged using a high-speed centrifuge (TG16-WS, Hunan Xiangyi Centrifuge Co. Ltd., China) at 10,280g for 10 min at 20 °C. The clarified supernatant was precipitated with the addition of

ammonium sulfate to its saturation and centrifuged the TG16-WS centrifuge at 10,278g for 20 min at 20 °C. The precipitate was triply washed with water, dialyzed for 24 h at 4 °C, and freezedried by a FD-1A-50 vacuum freeze drier (Beijing Boyikang Experimental Instrument Co. Ltd., China). The protein content in the sample of native WSP was  $91.5 \pm 1.5\%$ .

#### 2.2. Pilot experiments of recovering WSP from SWW

The Schematic diagram of a technology for recovering WSP from SWW is presented in Fig. 1, and the pilot experiments were carried out in the plant of Scents Holding Co. Ltd., China.

Firstly, SWW was treated by a batch foam fractionation process using air and the column in Fig. 1. In each experiment of foam fractionation, 120 L SWW containing sodium bisulfite of a certain concentration (0 g/L, 0.05 g/L, 0.10 g/L, 0.15 g/L or 0.20 g/L) was loaded into the column and then air was bubbled into the column at a superficial velocity (0.93 mm/s, 1.23 m/s, 1.54 mm/s, 1.85 mm/s or 2.16 mm/s) when the temperature inside the column reached to a fixed value (45 °C, 50 °C, 55 °C or 60 °C) which was kept by heat tapes and heat insulating cotton. The values of superficial gas velocity and temperature were selected based on the results of Jiang et al. (2011). After the aeration of 10–16 h, the foamate where plenty of WSP precipitate was formed due to their high enrichment ratio and denaturation underwent 3 h natural sinking to obtain the precipitate-liquid mixture. Then, the mixture was centrifuged at room temperature (20-25 °C) using a LDZ5-2 centrifuge (Beijing Medical Centrifuge Factory, China) at 4200g for 5 min to obtain protein-enriched precipitate and the precipitate was diluted and ground to form slurry which was adjusted to pH 6.8-7.0 using 5% (w/v) sodium hydroxide solution in a neutralization tank. Finally, the slurry was directly heated by water vapor to 135 °C and kept at this temperature for 1-2 s for sterilization and dried by using a Mini B-290 lab spray dryer (Buchi, Switzerland) under the conditions of inlet temperature 150 °C, outlet temperature 80 °C, volumetric gas velocity 25 m<sup>3</sup>/h and feed flow rate 600 ml/min.

In this pilot study, the foam fractionation column in Fig. 1 had 2700 mm in effective length and  $300 \times 300 \text{ mm}^2$  in sectional area, 266 times larger in effective volume than that used by Jiang et al. (2011) (95 mm in length and 35 mm in inner diameter) in lab experiments. In the bottom of the column, a gas distributor made of a stainless steel plate sparger (1.0 mm thick) was installed with laser-burned pores (diameter =  $100 \pm 10 \mu$ m) arranged in equilateral triangular pattern with centre-to-centre distance of 10.0 mm.

Enrichment ratio (*E*), recovery percentage (*R*) and effective recovery percentage ( $R_{\text{effective}}$ ), defined as below, were used as the indicating parameters for the process of foam fractionation.

$$E = \frac{C_{\rm f}}{C_0} \tag{1}$$

$$R = \frac{V_{\rm f} C_{\rm f}}{V_0 C_0} \tag{2}$$

$$R_{\text{effective}} = \frac{M_{\text{t}} \varphi_{\text{s}} \eta_{\text{pr}}}{V_0 C_0} \times 100\%$$
(3)

where  $V_0$  (L) and  $C_0$  (g/L) are volume and WSP concentration of the feed SWW, respectively;  $V_f$  (L) and  $C_f$  (g/L) are volume and WSP concentration of the foamate, respectively;  $M_t$  is total mass of the proteins-enriched precipitate, g;  $\varphi_s$  is solid content in the precipitate, %, measured by using a MJ33 moisture meter (Mettler-Toledo, USA);  $\eta_{\rm pr}$  is protein content in the solid of the precipitate, %; The WSP concentration was measured by using a Kjeltec 8200 kieldahl apparatus (Foss, Sweden).

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