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A novel method to intensify the foam fractionation process by operating the column under reduced pressure was reported in this paper. It was found that a poorer foam stability, a bigger mean bubble size and a wider bubble size distribution in the foam layer, a less upward liquid flux and volumetric liquid fraction at the top of the foam layer, and a higher enrichment ratio but a lower mass recovery of the foam fractionation process were obtained under reduced pressures compared to the ones under atmospheric pressure. The most important effect of the reduced pressure on the foam layer during the foam fractionation process is the encouraged bubble coalescence process which was exhibited by those unusual foam properties discussed above. The limitations of this new method were also discussed.

Keywords bubble size; coalescence; foam fractionation; reduced pressure

INTRODUCTION

Foam fractionation is a low-cost separation technology and many kinds of useful substances have been successfully enriched through this technology (1–4). The bubble size not only determines the interficial area where protein adsorption occurs, but also relates coalescence and drainage in the foam phase (5). Rand and Kraynik (6) reported a qualitative relationship between the bubble size distribution and the rate of drainage. They found that smaller bubbles are correlated with a decrease in drainage. Stevenson (7) has proposed an expression for the liquid superficial drainage rate from a foam expressed as a Stokes-type number, as a function of liquid fraction only. The expression indicates that the liquid drainage rate must be proportional to the mean bubble size squared. It seems that larger bubbles tend to hasten the liquid drainage and give less liquid holdup. When foams are a desired phase, the bubble size becomes very important to the effectiveness of the foam fractionation, as this is related to the liquid content. Uraizee and Narsimhan (8) observed that the enrichment of BSA is higher for larger bubble sizes in the foam layer during the foam fractionation process.

Coalescence affects the bubble size in the foam phase and plays a key role in the enrichment of the target product by enhancing the internal reflux. Lemlich (9) pointed out that bubble coalescence destroys the surface and so releases the adsorbed material which flows back down through the rising foam and this rich drainage acts as internal reflux, which enriches the foam. During foam fractionation, the evolution of the bubble size distribution along the foam height is a reflection of the coalescence process. Ireland (10) attempted to characterize the coalescence process in terms of the mean bubble size and the vertical profile of the bubble size was presented. Hence, increasing the bubble size in the foam phase provides a way to improve the efficiency of foam fractionation.

In this paper, the effect of reduced pressure imposed to the top of the foam column on the foam layer during the foam fractionation process was studied. First, the effect of reduced pressure on the foam stability was studied by recording the complete foam collapse time under different pressures. Second, the mean bubble size and bubble size distribution in the foam layer during the foam fractionation process at atmospheric pressure and reduced pressure were presented to illustrate the enhanced coalescence process. Third, the effect of reduced pressure on the hydrodynamic of the rising foam was studied by evaluating the relationship among superficial gas velocity j_g, and the upward liquid flux j_f and volumetric liquid fraction in the foam f under atmospheric and reduced pressures. Finally corresponding to the previous experimental results, the effect of reduced pressure on the enrichment ratio and mass recovery of the foam fractionation process was presented to show the potential industrial value of this novel method.

MATERIAL AND METHODS

Material and Apparatus

Bovine serum albumin (BSA) was purchased from Lian Xing Biotechnology Corporation (Tianjin, P. R. China) with analysis purity. The solution was prepared by dissolving a given amount of BSA solid in distilled water to give

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the desired concentration of 50 mg/L in the feed and then the solution was adjusted to pH 7.0 (in all experiments). The foam fractionation column used in this study had an inside diameter 3.8 cm and length 120 cm and was manufactured from glass shown in Fig. 1.

The maximum capacity of the vacuum pump was to reduce the absolute pressure from 1 bar to 0.15 bar. The reduced pressure was imposed at the top of the foam column and the pressure was controlled by the discharge valve shown in Fig. 1. The foam was generated pneumatically by sparging air through a pool of BSA solution at the base of the column. A rotameter was used to control the airflow rate into the column. The gas sparger used was a stainless steel disc of $150 \pm 10\,\mu m$ porosity. In all experiments, the bulk liquid pool was held at 20 cm in height and the feed temperature was kept at 20°C.

Methods

The foam fractionation process was operated in a batch mode. The reduced pressure was achieved with the help of a vacuum pump. When the foam was discharged from the top of the column, it was collected and collapsed. The collapsed foam (foamate) was analyzed for BSA concentration by observing the UV absorbance of the sample with a spectrophotometer (Jingke, China) at a wavelength of 280 nm. The foam produced was collected until none was seen during the batch type foam fractionation experiment. The foaming time for a run was defined as the period between the time when the first drop of the foam entered the foam collector and the time when no more foam was seen. The enrichment ratio (E_r) and mass recovery (R) were served as two separation performance criteria in this paper. They were calculated by the following equations:

$$E_r = \frac{C_f}{C_b} \tag{1}$$

$$R = \frac{C_f V_f}{C_b V_b} \tag{2}$$

where C_f and C_b are the concentration of BSA in foam (mg/L) and the initial bulk solution (mg/L), respectively; V_f and V_b are the liquid volume (L) of the foamate and initial bulk solution, respectively. In batch foam fractionation experiments in which the enrichment ratio and mass recovery were obtained, the range of superficial gas velocities of atmospheric and reduced pressure are both from 2.9 to 8.2 mm/s (2.9, 5.0, 6.9, and 8.2 mm/s).

The foam stability was evaluated by the complete foam collapse time. The foam layers with the same height (20 cm) were generated under pressure of 1 bar and then the absolute pressure inside the column was adjusted to 1, 0.8, 0.6, 0.4, and 0.2 bar, respectively. Every complete foam collapse time corresponding to a given absolute pressure was recorded.

The bubble size was measured at ten locations of the foam phase (from liquid-foam interface to 90 cm above the interface at intervals of 10 cm) during the foam fractionation process. A digital camera (Nikon CooLPIX P80) was used to capture the bubble images (examples shown in Fig. 2). Images from the camera were transferred to the computer for analysis. All the bubble images that were captured are from the single bubble layer which is against the transparent column wall. Now the question arose as to whether the bubble size distribution within the bulk of the foam could be well-represeneted by images taken at the column wall or not. Cheng and Lemlich (11) identified several sources of error when determining the bubble size distribution by a photographic method including statistical planar sampling bias and the fact that small bubbles can wedge big bubbles away from the column wall. They thought the latter source of error was able to cancel the former one. With no way of quantifying this segregation, the effect of statistical planar sampling bias and

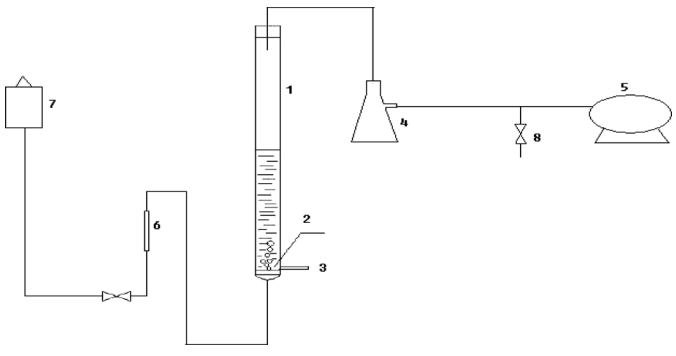
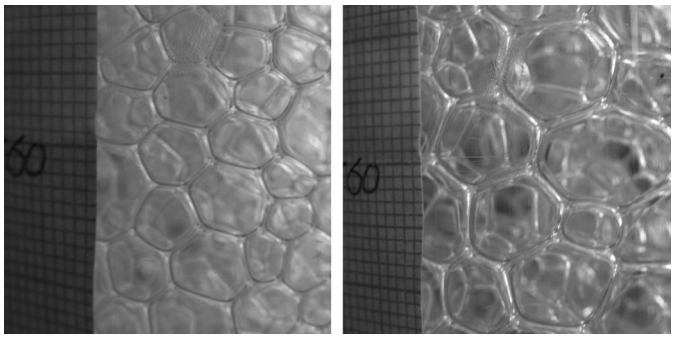


FIG. 1. Schematic diagram of the experimental equipment: (1) Foam fractionation column; (2) Gas distributor; (3) Outlet; (4) Foam collector; (5) Vacuum pump; (6) Rotameter; (7) Air pump; (8) Discharge valve.



(a) (b)

FIG. 2. Bubble images captured at the same column height (60 cm) under absolute pressure of 1.0 and 0.2 bar (Superficial gas rate $j_g = 5.0$ mm/s): (a) 1.0 bar; (b) 0.2 bar.

the ''wedging'' of larger bubbles away from the wall can only be assumed to be self-cancelling. In light of this, we left errors as it is. Since bubbles in the column have different shapes, the bubble diameter was taken as the diameter of the circle having the same area as the wall contact area of the bubble, A, the latter being measured with the help of computer aided design (CAD) software. Thus the diameter equals $2(A/\pi)^{1/2}$. The Sauter mean bubble size (d_{32}) is used to estimate the average size of bubbles. The expression of the Sauter mean bubble size is as follows:

$$d_{32} = \frac{\sum_{i=1}^{n} d_i^3}{\sum_{i=1}^{n} d_i^2} \tag{3}$$

where n and d_i are the number of bubbles and the individual bubble size, respectively. More than 200 bubbles were measured to obtain a credible mean bubble size.

The upward liquid flux j_f (mm/s) was calculated by the following equation:

$$j_f = \frac{Q}{A} \times 10 \tag{4}$$

where Q and A are the volumetric liquid overflow rate (mL/s) and the foam column cross-section area (cm^2), respectively. The volumetric liquid overflow rate Q was measured as the mass of liquid recovered from the top of the column in a known time. Because in batch mode foam fractionation process, the bottom pool will become depleted of protein and the coalescence rate will increase in time as the stability of the gas-liquid interface diminishes, the mass of the liquid was collected at the very beginning of each run of the experiment in a very short period (about one minute). Meanwhile, the bubble images were captured. Note that the mass of liquid used in calculating the enrichment ratio and mass recovery is collected during the whole foaming time. A propagation of uncertainty analysis indicates that the calculated volumetric liquid overflow rate has an uncertainty bounded by ±6 percent. The superficial gas velocity j_g (mm/s) is controlled by the rotameter. Since the object we studied is the top layer of the foam which is overflowed from the top of the foam column, there is no reflux of liquid passing through it. Hence, the volumetric liquid fraction at the top of the foam layer would be:

$$f = \frac{j_f}{\left(\frac{j_g}{1-f}\right)}. \tag{5}$$

where $\left(j_g/1-f\right)$ is the foam flow rate (mm/s). Then the Eq. (5) can be simplified as:

$$f = \frac{j_f}{j_g + j_f}. \tag{6}$$

In batch foam fractionation experiments in which the upward liquid flux and volumetric liquid fraction in the foam were obtained, the ranges of superficial gas velocities of the atmospheric and reduced pressure are from 1.5 to 10.2 mm/s (1.5, 3.6, 5.0, 8.0, and 10.2 mm/s) and from 2.4 to 8.2 mm/s (2.4, 2.8, 4.9, 6.8, and 8.2 mm/s), respectively.

RESULTS AND DISCUSSIONS

Effect on the Foam Stability

Figure 3 shows the evolution of complete foam collapse time under atmospheric pressure and reduced pressures. As the pressure decreased from 1 bar to 0.2 bar, the complete foam collapse time declined from 34 to 11 minutes. It is somewhat unexpected that the collapse time was nearly proportional to the pressure decrease, as the collapse of the foam should be related to many factors.

Film rupture and inter-bubble gas diffusion are the two main processes that affect foam stability. It is well known that the tendency of lamella to rupture increases with thinner lamella. Hence, the lamella became more vulnerable under reduced pressure due to the bubble expansion. If we assume that the air behaves as an ideal gas, then, in the limit of zero liquid fraction, the volume of the foam would increase by a factor of five when the pressure reduces from 1 bar to 0.2 bar. Thus, if we make the gross assumption that the bubbles have a monodispersive size and that they are spherical, the bubbles would increase in diameter by a factor of 1.71 (i.e., the cube root of 5).

Effect on the Mean Bubble Size of the Foam

In a foam fractionation column the bubble size at the same cross section of the column is generally considered to be uniform (5). Hence, profiles of the mean bubble size along the foam column under atmospheric pressure and

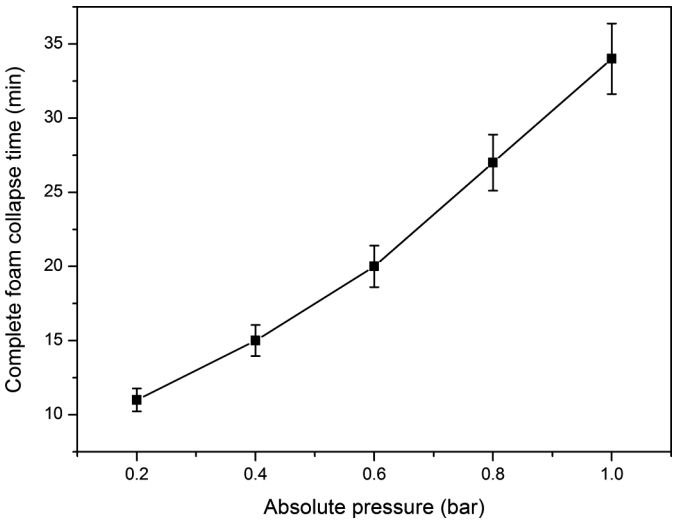


FIG. 3. The complete foam collapse time vs. absolute pressure.

reduced pressures were depicted to show the bubble size evolution during the foam fractionation process.

Figure 4 shows the vertical profiles of the mean bubble diameter under three different absolute pressures (1, 0.5, and 0.2 bar). As expected, the mean bubble diameter increases along the foam column and also when the pressure decreases. The increase in the mean bubble diameter becomes more pronounced under absolute pressure of 0.2 bar. It was noticed that towards the bottom of the foam the factor of 1.71 seems to hold approximately, whereas towards the top it does not. This is indicative of coalescence towards the top of the column. As the foam flowed up through the column, part of the interstitial liquid flowed back into the liquid phase and the liquid lamella became thinner which enhanced the bubble coalescence process. Therefore, a gradient of the mean bubble size under three different absolute pressures was observed at the top of the column.

Effect on the Bubble Size Distribution of the Foam

Since the foam generated by the sparging method contains a large amount of bubbles, the single mean bubble size does not adequately reflect the broad bubble size distribution in the foam fractionation process. Moreover, the rheology and stability of the foam are strongly influenced by the foam's bubble size distribution (12). Figures 5a–d shows the bubble size distribution, expressed as the cumulative number frequency versus the bubble diameter, under absolute pressure of 1 and 0.2 bar at four different vertical positions (liquid-foam interface, 30, 60, and 90 cm above the interface). At each position, the entire cumulative distribution curve of 0.2 bar is shifted to the right in comparison with that of 1 bar, which is in correspondence with Fig. 4. It is shown that under reduced pressure, there was a great change towards a wider bubble size distribution, especially at the top of the column (90 cm above the liquid-foam interface). Take Fig. 5c for example, the bubbles under atmospheric pressure mostly are about 3–6 mm in diameter, the largest being up to 7.5 mm, whereas the foam under reduced pressure has bubbles that were up to 12.0 mm and ranges from 4.5–12.0 mm in diameter. As can be seen from Figs. 5a–d, the bubble size distribution becomes wider as the foam flows up through the vertical column. The tendency becomes more obvious under pressure of 0.2 bar where the ranges of bubble diameter at the vertical position of the liquid-foam interface, 30, 60, and 90 cm above the interface are 1.7–4.2 mm, 2.5–8.5 mm, 4.5–12.0 mm, and 7.0–21.0 mm, respectively.

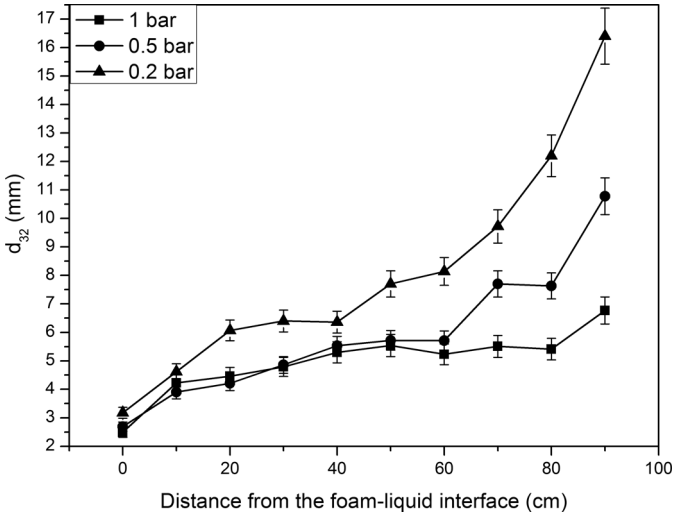


FIG. 4. Sauter mean bubble diameter vs. vertical positions, for three values of absolute pressure (Superficial gas rate $j_g = 5.0$ mm/s).

Effect on the Upward Liquid Flux and Liquid Fraction at the Top Layer of the Foam

Liquid flux and liquid fraction are two important parameters of the rising foam as they strongly influence the wetness of the foam. In the foam fractionation process, the interstitial liquid entrained by the rising foam is very harmful to the enrichment of the target product as it will make the foamate diluted. The interstitial liquid fluxes in two opposite directions in the rising foam—the upward and the downward. The upward liquid flux will be one part of the foamate whereas the downward flux is served as the drainage of the foam which will flow back to the liquid pool at the base of the foam column. The liquid fraction at the given cross-section of the foam layer is determined by the two opposite liquid fluxes according to the mass-balance assumption. As for the top layer of the rising foam, as there is no liquid drainage from the top, the liquid fraction only depends on the upward liquid flux.

Figure 6 illustrates the relationship between the upward liquid flux j_f and the superficial gas velocity j_g at the top layer of the rising foam under atmospheric and reduced pressure. The power-law relationship with an exponent of 1.769 (1 bar) and 2.344 (0.2 bar) is close to the conclusion of Neethling (13) which is a power-law relationship with an exponent of 2. Owing to the encouraged bubble coalescence process under reduced pressure, more entrained interstitial liquid in the foam released as the downward liquid flux which correspondingly diminishes the upward liquid flux j_f. Since more interstitial liquid released as the foam rises up under reduced pressure, the foam will become drier. This effect is shown in Fig. 7. To sum up, the reduced pressure makes the bubbles bigger which in turn makes the foam drier.

Effect on the Enrichment Ratio and Mass Recovery of the Foam Fractionation Process

In the previous sections, the reduced pressure at the top of the foam column will make the foam more unstable, the

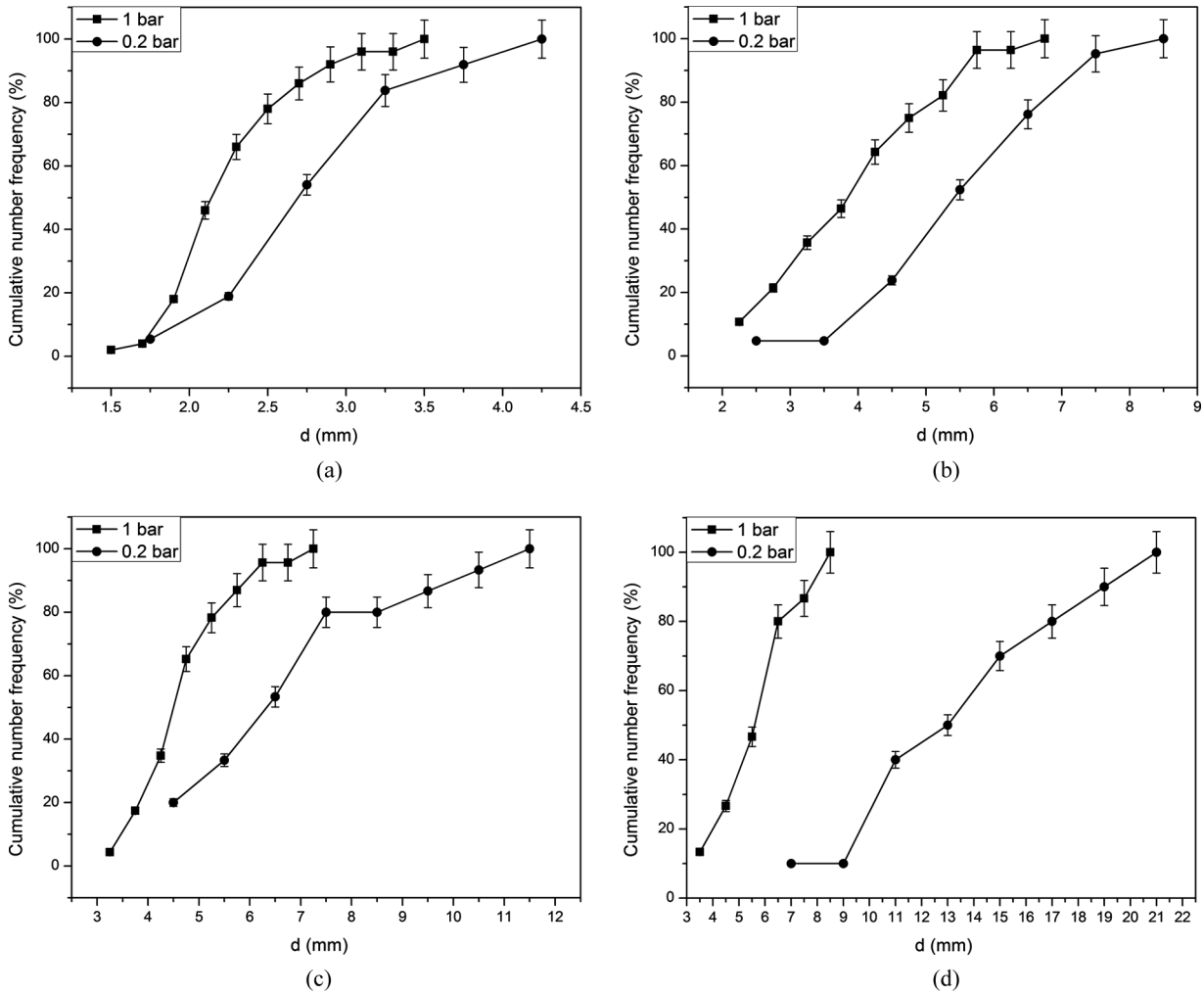


FIG. 5. Comparison between the cumulative bubble size distribution at atmospheric pressure (1 bar) and under vacuum (0.2 bar) for four different distances from the foam-liquid interface (Superficial gas rate $j_g = 5.0$ mm/s): (a) 0 cm; (b) 30 cm; (c) 60 cm; (d) 90 cm.

bubble size bigger and wider distributed, the bubble coalescence happens more easily, the upward liquid flux gets diminished, and the foam gets drier. All these effects caused by the reduced pressure will influence the enrichment ratio and mass recovery. The foaming times of each run appeared in Figs. 8 and 9 are as follows: for the experiments under atmospheric pressure, the foaming times with the superficial gas velocity j_g of 2.9, 5.0, 6.9, and 8.2 mm/s are 25, 17, 12.5, and 9 min, respectively; for the experiments under reduced pressure (0.2 bar), the foaming times with the superficial gas velocity j_g of 2.9, 5.0, 6.9, and 8.2 mm/s are 38.5, 29, 20.5, and 15.5 min, respectively. Figure 8 shows the evolution of the enrichment ratio with different superficial gas rates under atmospheric and reduced pressure. Since a higher gas rate will make the bubble retention time in the foam column reduced which hinders the bubble coalescence process and correspondingly makes the foam wetter, the enrichment ratio decreased with the increase of the gas rate. The positive effect of the encouraged bubble coalescence process under reduced pressure on the enrichment ratio of the foam fractionation process was experimentally confirmed at all gas rates (shown in Fig. 8). Figure 9 shows the evolution of mass recovery with the same superficial gas rates as the ones in Fig. 8 under atmospheric and reduced pressure. Since a higher gas rate encourages the surface flux of bubbles and reduces the retention time of foam in the column which is an important factor in the bubble coalescence process, the foam will be more stable and more protein will be recovered from the bulk solution. From Fig. 9, the mass recovery increases from 70.2% to 88.3% and from 61.5% to 81.2% with the increase of superficial gas velocity from 2.9 to 8.2 mm/s under the absolute pressure of 1 bar and 0.2 bar, respectively. Because the foam becomes more fragile under reduced pressure due to the encouraged bubble coalescence process which is experimentally verified in

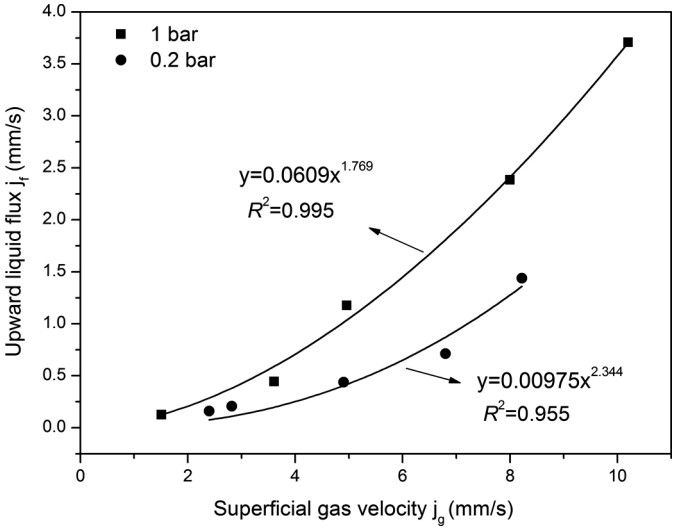


FIG. 6. Experimental relationship between the upward liquid flux j_f and the superficial gas velocity j_g at the top layer of the rising foam under atmospheric and reduced pressure.

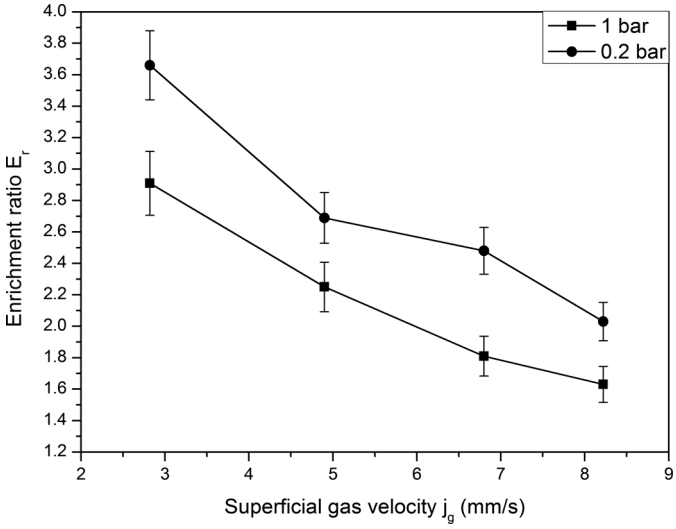


FIG. 8. Experimental relationship between the protein enrichment ratio E_r and the superficial gas velocity j_g at the top layer of the rising foam under atmospheric and reduced pressure.

section titled "Effect on the Foam Stability", higher protein concentration of the residual solution in the column is observed compared with the one under atmospheric pressure with the same gas rate. The negative effect of the encouraged bubble coalescence process under reduced pressure on the mass recovery of the foam fractionation process was experimentally confirmed at all gas rates (shown in Fig. 9). Bhattacharjee et al. (14) developed a phenomenological model to predict the separation factor (defined as enrichment ratio) obtained in concentrating protein (casien and BSA) solutions using a batch-foam column. They reported the profile of enrichment ratio of BSA (1 g/l) with the changes of three factors—pool residence time, drainage time, and foam height. Under the optimum conditions, the enrichment ratio is less than 2, whereas in our paper the enrichment ratio is up to 3.7 under reduced pressure with a relatively low gas rate. Aksay and Mazza (15) reported their enrichment and mass recovery results under optimum conditions of batch mode foam fractionation using the response surface methodology. From their paper, the highest enrichment ratio is 7.81 with the lowest recovery of 46.89% and the highest

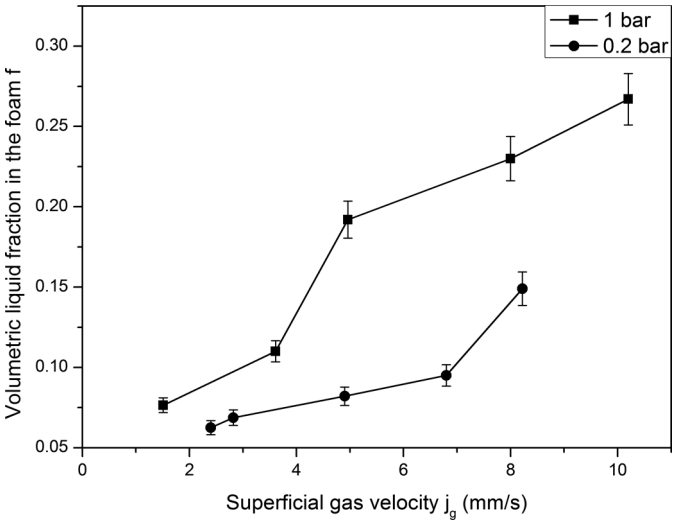


FIG. 7. Experimental relationship between the volumetric liquid fraction in the foam f and the superficial gas velocity j_g at the top layer of the rising foam under atmospheric and reduced pressure.

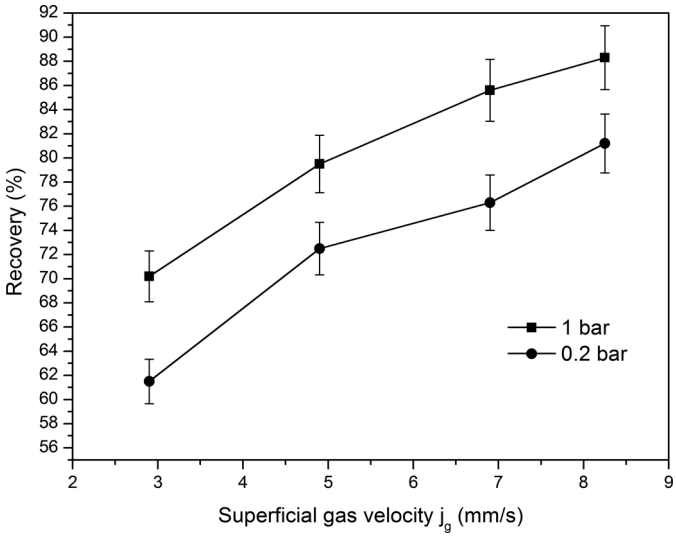


FIG. 9. Experimental relationship between the protein mass recovery R and the superficial gas velocity j_g at the top layer of the rising foam under atmospheric and reduced pressure.

recovery is 97.79% with the lowest enrichment ratio of 1.42. This contradiction between the enrichment ratio and mass recovery is the same as the results reported in our paper.

To put these research results into industrial practice, there are still some concerns which need to be discussed:

1. The energy consumption problem. Since this method demands the reduced pressure imposed on the top of the foam column, the extra energy consumption will be needed to be compared to the regular foam fractionation process. On the grounds of economic consideration, this method is only suitable for the separation of some high-valued products.
2. The product recovery problem. As discussed in the section titled ''Effect of the Foam Stability'', the foam stability is poor when the reduced pressure imposed at the top of the column. The poor foam stability will discourage the product recovery from the liquid pool since the foam is the carrier of the target product. This is experimentally verified in the section titled ''Effect on the Enrichment Ratio and Mass Recovery of the Foam Fractionation Process.''
3. The processing rate problem. The high processing rate will reduce the operation time of the foam fractionation process.

While as discussed in the sections titled ''Effect on the Mean Bubble Size of the Foam'' and ''Effect on the Bubble size Distribution of the Foam'', the bigger bubble size will be obtained when conducting the foam fractionation process under reduced pressure. This means the surface flux of bubbles rising through the foam, which determines the processing rate, will decrease. A longer operation time will be needed to complete the foam fractionation process under reduced pressure compared to the one under atmospheric pressure. This is experimentally verified through the foaming time results in the section titled ''Effect on the Enrichment Ratio and Mass Recovery of the Foam Fractionation Process.''

CONCLUSIONS

1. The foam becomes more fragile under reduced pressure due to the encouraged bubble coalescence process. As the absolute pressure decreased from 1 bar to 0.2 bar, the complete foam collapse time declined from 34 to 11 minutes.
2. The foam presents a bigger mean bubble size and wider bubble size distribution under reduced pressure. In Fig. 4, the mean bubble diameter increases from 6.5 mm to 16.5 mm at the top of the column as the absolute pressure reduces from 1.0 to 0.2 bar. In Fig. 5c, the bubbles under atmospheric pressure mostly are about 3–6 mm in diameter, the largest being up to 7.5 mm, whereas the foam under reduced pressure has bubbles that were up to 12.0 mm and ranges from 4.5–12.0 mm in diameter. Bubble expansion encourages the bubble coalescence process.
3. Under reduced pressure, the foam at the top of the column upholds less upward liquid flux and therefore becomes drier. In Fig. 7, the volumetric liquid fraction in the foam f, which is used to evaluate the wetness of the foam, ranges from 0.06 to 0.13 with the superficial gas velocities ranging from 2.4 to 8.2 mm/s under reduced pressure whereas f ranges from 0.075 to 0.22 with the comparative superficial gas velocities ranging from 1.5 to 8.0 mm/s under atmospheric pressure.
4. An enhanced enrichment ratio but lower mass recovery were obtained when the foam fractionation process operated under reduced pressure. In Fig. 8, the enrichment ratio is up to 3.7 at the lowest gas rate under reduced pressure whereas the enrichment ratio is only 2.9 at the same gas rate under atmospheric pressure. In Fig. 9, the mass recovery is 61.5% at the lowest gas rate under reduced pressure whereas the mass recovery is 70.2% at the same gas rate under atmospheric pressure.

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