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EFFECTS OF FILTER ADDITIVES ON CAKE FILTRATION PERFORMANCE

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Abstract – Effects of various additives, such as diatomaceous earth, inorganic adsorbent, and polymeric flocculant, on cake filtration performance were evaluated. By performing the standard vacuum filtration experiments under a constant pressure condition, we quantified their ability to reduce cake compressibility and to remove fine particulate matters. From the flux decline curves obtained, we used modified Ruth equation to determine the cake compressibility index. The filtrate clarity was quantified by measuring its turbidity spectrophotometrically. Diatomaceous earth filter aid was very effective in reducing the compressibility thereby improving filtrate flux. Calcium phosphate adsorbent was more effective in clarity improvement than in flux enhancement, whereas an anionic flocculant was effective in both aspects. When these additives were used together, the expected additive, synergic effect was not realized. However, about 3.5 times higher initial flux and 6-fold improved clarity were obtained as compared to the control experiment. Experimentally obtained flux decline curves were perfectly fitted into one of the Hermia's blocking filtration law equations. Also, it was experimentally observed that the compressibility reduction improved filtrate clarity in a linear fashion. This type of approach is useful to quantitatively determine each additive's characteristics and thus to maximize the performance of cake filtration operations.

Key words: *Cake Filtration, Filter Additives, Cake Compressibility, Filtration Flux*

INTRODUCTION

Cake filtration is widely used in bioprocessing industry either as a primary separation step to remove or recover biomass solids and other cellular materials or as a final polishing step to remove fine particulate matters. Filtration flux and filtrate clarity are two major parameters that determine the overall performance of cake filtration process. Various additives are often added to enhance the flux and to improve the clarity. They can be classified into three categories, i.e., filter aids of diatomaceous earths, flocculants of synthetic polymers, and coagulants of inorganic salts. Working mechanisms of these additives are well known. For instance, diatomaceous earths are known to adsorb fine particles onto its surface to reduce the compressibility of the biomass particles, and thus render the cake layer more porous [Nakanishi et al., 1987]. Flocculants and coagulants increase the particle size and density by forming three dimensional matrices through ionic interactions [Kimand Rha, 1987].

However, research literatures on the quantitative evaluation of these additives' effects on the flux and the clarity are relatively scarce. In this study, using corn gluten hydrolysate as a model slurry the effects of filter aid (Perlite), calcium phosphate coagulant, and cationic polymer flocculant are separately and combinatively evaluated in terms of filtration flux enhancement and filtrate clarity improvement.

MATERIALS AND METHODS

Dried corn gluten meal was gifted from Doosan Foods Inc. (Ichon, Korea). It was enzymatically hydrolyzed by using alkaline protease from *Bacillus licheniformis* at pH 9.0 and 50 °C. Other details of the hydrolysis process can be found elsewhere [Kim et al., 1995]. Solid content of the corn gluten hydrolysate was determined after oven-drying at 70 °C for 24 hours. Density was measured by a densitometer and viscosity by Ostwald's method at 25 °C. Filtrate clarity was evaluated spectrophotometrically by measuring the absorbance at 600 nm. Cake filtration experiments were performed in a standard vacuum filter apparatus using Nylon cloth as a filter medium. Perlite, calcium chloride, and monopotassium phosphate were purchased locally, and cationic flocculant made of polyacrylamide was gifted from Hansu, Inc. (Ansan, Korea; product number=CP-911.)

Applying the Poiseuille's equation to a filtration process, the following equation can be derived [McCabe et al., 1967]:

$$\frac{dV}{Ad\theta}=\frac{\Delta P}{\mu[\alpha(W/A)+r]} \tag{1}$$

Here, V=filtrate volume, A=filter area, θ=time, ΔP=pressure (or vacuum) applied, μ=filtrate viscosity, α=average specific cake resistance, W=mass of dry cake, and r=filter medium resistance. And, α is related to the applied pressure by the following expression:

$$\alpha=\alpha'\Delta P^{S} \tag{2}$$

Here, α' is an intrinsic constant of a cake determined largely by the size of the particles forming the cake, and S, the compressibility index value, is a measure of cake compressibility varying from 0 for rigid, incompressible cakes to 1 for

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Table 1. Experimental conditions used in filtration of corn gluten meal hydrolysate

Expt no.	Additives added to the Hydrolysate [All %s are %(w/v)]
#1	Hydrolysate only (Control)
#2	Perlite, 0.1 %
#3	Perlite, 0.5 %
#4	Perlite, 1.0 %
#5	Perlite, 2.0 %
#6	Perlite, 5.0 %
#7	$CaCl_2$, 0.5 %+KH_2PO_4 0.25 %
#8	$CaCl_2$, 1.0 %+KH_2PO_4 0.5 %
#9	$CaCl_2$, 2.0 %+KH_2PO_4 1.0 %
#10	CP-911, 0.2 %
#11	CP-911, 0.5 %
#12	CP-911, 1.0 %
#13	CP-911, 2.0 %
#14	Perlite, 2 %+$CaCl_2$, 0.5 % + KH_2PO_4, 0.25 %+CP-911, 1 %

soft, very compressible cakes. The S value is in the range of 0.1 to 0.8 for most industrial cakes [Perry and Chilton, 1973]. Eq. (2) indicates that cake compressibility plays an important role in filtration of relatively compressible biomass solids in such slurries as fermentation broths, biomass hydrolysates, etc. Combining Eq. (2) and Eq. (1) and considering that in the usual range of operating condition r is negligible, we could modify the classical Ruth equation as follows:

$$V^2=K\theta \tag{3}$$

where K is defined as $(2A^2/\ C\alpha'\mu)\Delta P^{1-S}$, and C denotes mass of cake deposited per unit volume of filtrate and thus is related to the solid content of feed slurry. Since A, C, μ, and α' are constants, by maintaining ΔP constant we can determine the S value from a plot of V (x-axis) vs. θ/V (y-axis). By measuring the filtrate volume with elapsed time, the S value under each condition was experimentally determined and compared.

Table 1 shows 14 different sets of conditions used in our experiments. Expt #1 was the control experiment where no additives were added. In the Expt #14 all three additives were added to evaluate any synergic, combined effect. All the filtration experiments were performed at room temperature. By applying varying levels of vacuum pressure (100, 300, 500, and 700 mmHg) in the Expt #1, α values were determined and plotted against ΔP. From the y-intercept of that plot α' value was estimated and then used to calculate the S values in the Expt #2 through #14.

RESULTS AND DISCUSSION

Physical properties of the corn gluten hydrolysate (pH 9.0) was determined as follows: solid content was 11.7% (0.117 g solid per gram of hydrolysate), density of the hydrolysate and the filtrate was 1.037 and 1.033, respectively, and the filtrate viscosity was 1.03 cp. Fig. 1 and 2 show the variations in the S value and the filtrate turbidity as determined under each condition. From the control experiment (Expt #1),

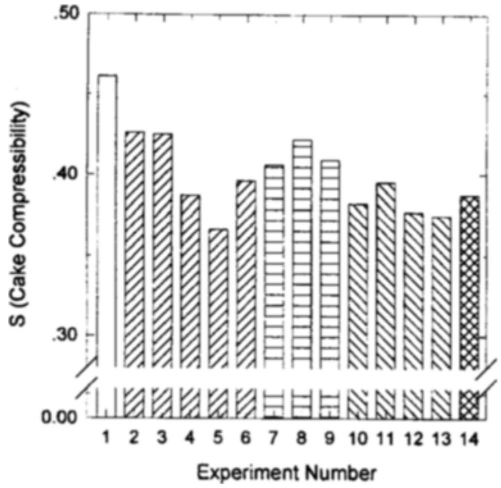

Fig. 1. Changes in cake compressibility.

the S value of 0.46 and the filtrate absorbance (at 600 nm) of 1.31 were obtained. The S value was very close to 0.45, which was obtained from the cross-flow membrane filtration of Baker's yeast cells [Nakanishi et al., 1987].

Among the Perlite concentrations tested, 2.0 % resulted in the lowest cake compressibility (see Expt #5 in Fig. 1), whereas 1.0 % yielded the lowest turbidity (see Expt #4 in Fig. 2). Relatively higher compressibility and turbidity were observed at 0.1 and 0.5 %. Thus, it can be concluded that the Perlite concentration should be higher than 1 % to improve both flux and clarity. At 5 % the S value was seen to increase, which indicated the flux was reduced because of excessively increased cake thickness. Fig. 3 shows the flux profiles of the Perlite-added hydrolysates. As the Perlite concentration increased higher flux was obtained. However, at 5% the initial flux was not as high as that of 2 % and the flux declined more abruptly, which could be attributed to increased cake thickness. The highest initial flux was observed at 2 % which was approximately 4 times higher than that of the control experiment. At this condition, the S value was decreased by ca. 22 % to 0.36 (from 0.46 of the control experiment.). This reduction was in very good agreement with

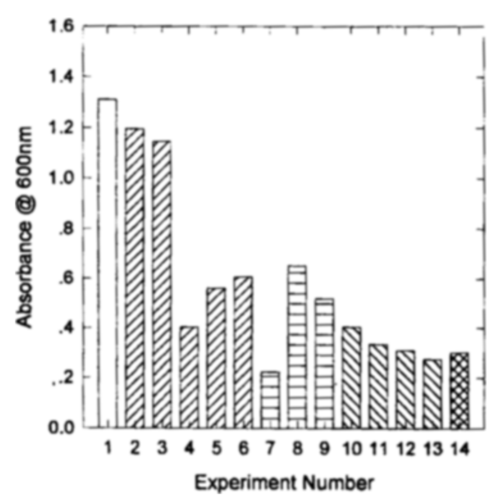

Fig. 2. Changes in light absorbance of filtrate.

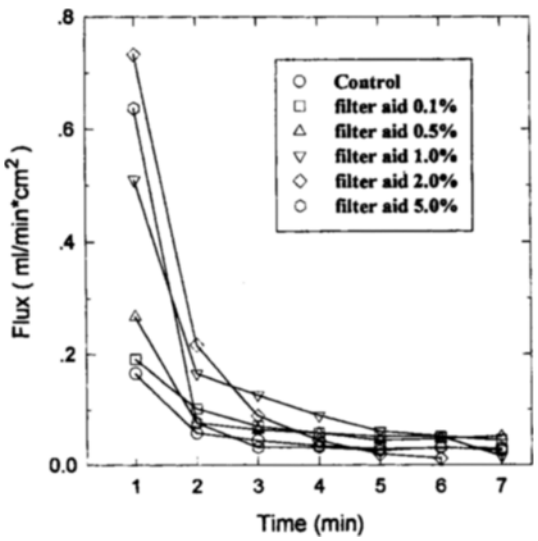

Fig. 3. Effect of filter aid addition on flux.

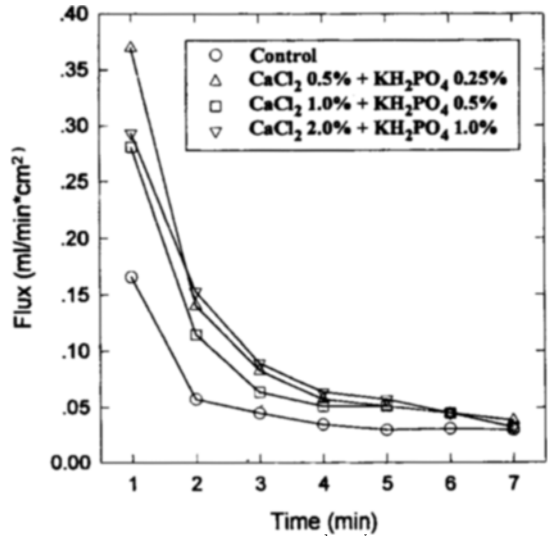

Fig. 4. Effect of calcium phosphate treatment on flux.

a previous report by Nakanishi et al. [1987], in which they reported about 25 % reduction in the compressibility index of Baker's yeast cake with 10 % (weight of filter aid per weight of dry solids) filter aid addition. Note that 2 % filter aid in our experiment was equivalent to 17.1 % in their experiment.

$CaCl_2$ and KH_2PO_4 were added sequentially to induce *in situ* formation of various calcium phosphate salts that could function similarly to hydroxylapatite gel [Green and Wase, 1986]. Among many applications, calcium phosphate gels can be effectively used as a non-specific adsorbent to adsorb charged proteins and peptides to either calcium or phosphate sites on its surface. In our experiments, $CaCl_2$ was first added, fully dissolved, and then KH_2PO_4 was added. Compared to Perlite, it was not as effective in reducing the cake compressibility (see Expt #7-9 in Fig. 1) but quite effective in improving the filtrate turbidity, particularly at 0.5 % $CaCl_2$ and 0.25 % KH_2PO_4 (see Expt #7 in Fig. 2). Under this condition, the turbidity was reduced by more than 80 % and the initial flux was improved about twofold from the control (Fig. 4).

Polymeric flocculants in general have three-dimensional structure of branched polymers with charged functional groups distributed along the polymer backbone. Depending on the surface charge of solid particles, the charged groups on the polymer surface interact with the particles via ionic interaction for flocculation. Among the flocculant concentrations tested (0.2, 0.5, 1.0, and 2.0 %), compressibility reduction effect was comparable each other (see Expt #10-13 in Fig. 1). And the clarity improvement effect, which became somewhat stronger as its concentration was increased, was superior to that of Perlite or calcium phosphates (Fig. 2). This result confirms the observation that the charged flocculants plays a crucial role in capturing some of the fine particles responsible for the organization of a resistant layer. Initial fluxes were comparable to those of Perlite (Fig. 5).

To find any synergic, combined effect of these additives, Perlite (2 %), $CaCl_2$ and KH_2PO_4 (0.5 and 0.25%, respectively), and CP-911 (1.0 %) were added in order. The compressibility and the turbidity were much reduced but the additive, synergic effect of the additives was not realized (see Expt #14 in Fig. 1 and 2). As seen in Fig. 6, about 3.5 times higher initial flux as compared to the control was obtained.

Considering the interactions between solid particles and filter materials, Hermia presented several physical models for cake filtration [Hermia, 1982]. In the 'complete' and 'intermediate blocking' models, the flux decline pattern was expressed by a simple exponential form, i.e., $Q=Q_0 \cdot \exp(-K\theta)$. Here Q and Q_0 are the instantaneous and the intial (or time zero) flux, respectively, and K is a constant determined by the pore blocking mechanism employed in the model. To this model equation, we added a term for the asymptotic flux:

$$Q=Q_0 \cdot \exp(-K\theta)+Q_\infty \tag{4}$$

Q_∞ here is the asymptotic flux. All the experimentally obtained flux curves were well fitted into Eq. (4) with the average regression coefficient of 0.995. Table 2 summarizes the Q_0, K and Q_∞ values calculated from Eq. (4) for each

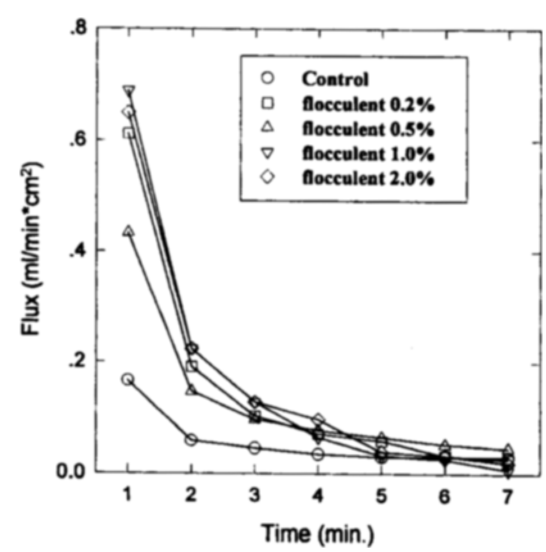

Fig. 5. Effect of polymeric flocculent addition on flux.

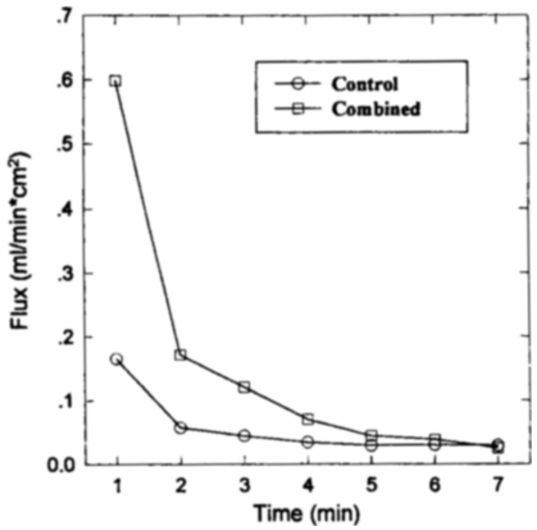

Fig. 6. Flux profile under control and combined conditions.

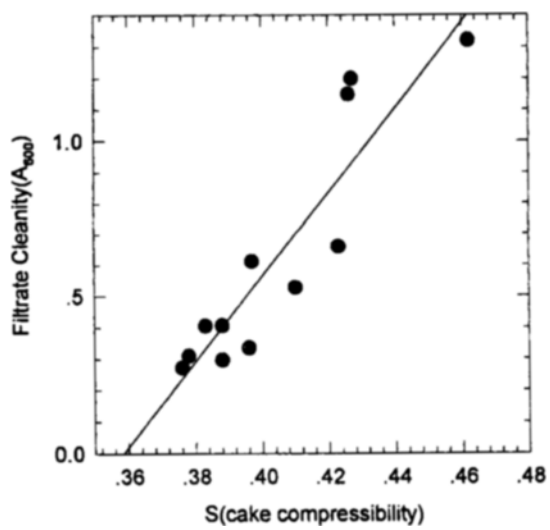

Fig. 7. Relationship between cake compressibility and filtrate clarity.

Table 2. Q_0, K, and Q_∞ values determined by using Eq. (4)

Expt. no.	Q_0 (ml/min/cm^2)	K (min^{-1})	Q_∞ (ml/min/cm^2)	r^2 (reg. coeff.)
#1	0.62	1.53	0.03	0.9952
#2	0.37	0.94	0.05	0.9987
#3	1.70	2.07	0.05	0.9954
#4	1.46	1.17	0.05	0.9780
#5	2.47	1.24	0.02	0.9991
#6	7.72	2.54	0.03	0.9999
#7	1.04	1.17	0.04	0.9965
#8	0.77	1.17	0.04	0.9983
#9	0.56	0.18	0.04	0.9971
#10	2.01	1.26	0.04	0.9957
#11	1.43	1.34	0.06	0.9945
#12	1.97	1.09	0.03	0.9945
#13	1.78	1.07	0.04	0.9916

experimental condition.

Cake compressibility is known to affect the filtrate clarity also. In general, fine particles are more easily captured by less compressible or more rigid cake layer. In Fig. 7, the S values determined are plotted against the filtrate absorbances. It strongly suggests that a small reduction in cake compressibility could yield much clearer filtrate.

CONCLUSION

The effects of diatomaceous earth as a filter aid, calcium phosphate gel coagulant, and cationic polymer flocculant on cake filtration performance were evaluated focusing on the additive's ability to reduce the compressibility of biomass solids and to remove fine particles from the filtrate. The Perlite filter aid was very effective in reducing the compressibility thereby improving filtrate flux. Minimum 1% (w/v) was needed for the effectiveness. Calcium phosphate coagulant showed more positive effect in filtrate clarity improvement than in flux enhancement, whereas the polymeric flocculant was effective in both. Experimental flux curves could be explained by Hermia's filtration equation modified to include an asymptotic flux term. And, compressibility reduction seemed to be in a linear relationship with filtrate clarity. This type of approach can be taken to quantitatively evaluate the filter additive's characteristics and the results can be used to optimize their dosages.

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