FILTRATION AND DRYING OF CASEIN

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ABSTRACT

The casein precipitated from the skimmed milk was filtered under constant pressure conditions followed by the cake drying in a vacuum tray dryer (VD) as well as heat pump assisted fluid bed dryer (HP-FBD).The average specific cake resistance (α_{av}) was found to be strongly dependent on the filtration pressure. Casein forms a very compressible filter cake and the compressibility index (n) was found to be 0.438 for Δp range of 0.5-2.5 bars and increases up to 0.77 at higher pressure range (1.5-2.5 bars).
The drying of casein in a vacuum tray dryer shows typical drying rate curve with small constant rate and large falling rate period. The effective moisture diffusivity computed on the basis of Fick's law varied between 3.39 x 10^{-10} to 1.51 x 10^{-9} m^2s^{-1} within the temperature range of 40^oC to 60^oC. The two step drying process was employed in order to reduce the drying time; especially in the falling rate period. It consists of decreasing the moisture content upto approximately 1 kg water kg dry solid^{-1} by VD followed by the extrusion and granulation of casein and then the drying of casein granules in a heat pump assisted fluid bed dryer (HP-FBD). Two step drying reduced the drying time drastically; especially in the falling rate period. The percentage protein retention after drying was found to be more than 90 % for the experimental drying conditions studied.

INTRODUCTION

Cake filtration, as a solid-liquid separation process, is widely used in the chemical and allied industries. It mainly involves the cake formation and growth, deliquoring, cake washing and drying operations, depending upon the requirements of a specific product. As much liquid is removed mechanically in filtration process, the solid cake can be further subjected to thermal drying in order to get the free flowing powder. The development of efficient filtration technology requires more insightful understanding and better information of various aspects of the filtration process (Tien Chi, 2002). In order to scale up and design of filtration and drying process, it is necessary to accurately determine the filtration as well as drying characteristics of cake and various scale up coefficients from the experimental data. It is also necessary to study the effect of various operating parameters on the cake properties and further on the quality of the dried product.

Casein is the phosphoprotein that accounts for nearly 80% of the protein in cow milk and cheese. It is found in the milk as a suspension of particles called casein micelles. Membrane processes are increasingly used in dairy industry for the separation of casein micelles from whey proteins by microfiltration (0.1 μm) in order to standardize the ratio casein/whey proteins. But conventional cross-flow filtration of milk has a problem of low protein transmission due to micelle cake building up (Berre and Daufin, 1996). This problem can be overcome by the use of higher recirculation velocities of retentate and permeates and also by the use of vibratory shear enhanced separation system (Al-Akoum et al., 2002). Microfiltration is also used for the separation of casein micelles from milk serum protein, mainly β-lactoglobulin and α-lactalbumin. It is desirable to remove the non-casein protein (whey) from milk before cheese or casein manufacturing, mainly because it is not a part of cheese and only the small quantity gets retained and its removal before cheese making does not alter the composition of the cheese (Nelson and Barbano, 2005). These aspects of the process and product characterization have been well studied in literature, however, the filtration behavior of the precipitated casein as well as filtration and drying characteristics of the casein cake have not been studied and published yet. Shiby and Mishra (Shiby and Mishra, 2007) studied the drying characteristics of Curd (Indian Yoghurt), a material similar to casein in a convective air dryer at different operating conditions.

The main aim of the proposed research was to study the filtration followed by drying characteristics of casein cake for the separation of precipitated casein from remaining whey, where, both the retentate and filtrate may be of great importance. It is also desirable to study the effect of different operating parameters during filtration and drying on the cake characteristics.

EXPERIMENTAL

Materials

The skimmed milk was procured from the local market in Mumbai, India. The charged modified depth filter pads having the absolute and nominal pore size rating of 5μm and 3.2μm, respectively, were supplied by the Microfilt India Pvt. Ltd., India. Bovine Serum Albumin (BSA) and Coomassie brilliant blue G dye were purchased from Hi – Media Laboratories Pvt. Ltd, Mumbai, India. Citric acid, Orthophosphoric acid, ethanol, and sodium thiosulphate were purchased from S. D. Fine Chemicals Ltd., Mumbai, India. The other chemicals and solvents were of analytical grade and were used as it is.

Preparation of Casein Suspension

The initial pH of the skimmed milk was 6.6 and brought to 4.6 by the addition of 0.2 N citric acid with gentle stirring by the glass rod. The casein starts precipitating as the pH reduces and at pH 4.6, which is the isoelectric point of casein, it precipitates out completely. This casein suspension or slurry was further used for all filtration experiments. The particle size of the casein suspension was determined by LS 230, Beckman Coulter Inc. particle size analyzer.

Filtration of Casein suspension

The filtration assembly comprises of a filter media casing, feed hold up tank, pressure regulator, and compressor. The filter casing, when fitted with filter media provides the effective filtration area of 0.00385 m^2. The desired pressure of compressed air was adjusted and maintained at constant value with the help of a pressure regulator.
A fresh filter media was used for each experiment. It was first wetted with the distilled water so as to fill the pores with water and then fitted onto the perforated steel support plate provided in the casing. A fixed amount of casein slurry was poured onto the filter medium and the system was pressurized as soon as possible at the desired pressure. The bottom valve was kept open completely and immediately started the measurement of filtrate volume with respect to time. After the completion of filtration process, the cake thickness was noted down. The filter cake was then removed from filter media, weighed and its moisture content determined to get the dry mass of cake. The constant pressure filtration experiments were carried out at different operating pressures ranging from 0.5 bars to 2.5 bars.

Analysis of filtration data

The general filtration equation used for constant pressure filtration was,

$$\frac{t}{V} = \frac{\alpha_{av}\mu c}{2A^2\Delta p}V + \frac{\mu R}{A\Delta p} \qquad (1)$$

The graph between t/V and V gives a straight line. The average specific cake resistance, α_{av} (mkg^{-1}) and the filter medium resistance, R (m^{-1}) can be determined from the slope and intercept of the graph, respectively.
The study of variation of average specific cake resistance (α_{av}) and average cake porosity (ε_{av}) or average cake solids fraction (C_{av}) with respect to operating pressure is of great importance and the following well known correlations were used to evaluate these properties.

$$\alpha_{av} = \alpha_0 (1-n) \Delta p^n \qquad (2)$$

$$C_{av} = C_0 \Delta p^{\beta} \qquad (3)$$

Where, n is the compressibility index i.e. indication of the extent to which the filter cake can compress when subjected to compressive forces and C_{av} is the average cake solids fraction. The α_o and C_o are the specific cake resistance and cake solids fraction at unit applied pressure respectively. The index β gives a measure of the potential for deliquoring the cake by compression, which is an important factor for mechanical dewatering. Equations (2) and (3) were linearised in order to calculate the values of different coefficients (Tarleton and Wakeman, 2007).

Cake washing

The washing of casein cake was performed by reslurrying as well as by displacement washing methods at a pressure of 1 bar. The distilled water was used as wash liquor and the instantaneous solute concentration (ϕ) was monitored and plotted against the wash ratio (W). The wash ratio is the ratio of volume of wash liquor used to the volume of liquor present in cake at the start of washing. The solute (soluble whey proteins) concentration was determined by the Bradford method of protein analysis (Bradford, 1976).

Vacuum Tray Drying

The drying of casein cake was carried out by using vacuum tray dryer (VD) supplied by the Salvis Lab, Switzerland. The casein cake was spread on to the tray to form a slab of 12.5 cm × 13 cm × 1 cm. The drying experiments were carried out within the temperature range of 40^{o}C to 60^{o}C and at a constant vacuum of 50 mbar. The weight loss over the period of time was monitored and the effect of temperature on the drying rate and protein retention in the dried powder was studied.

Two Step Drying of Casein

The two step drying process was employed in order to reduce the drying time. It involves the primary drying by vacuum tray dryer to reduce the moisture content of casein cake to approximately 50% by weight. In the second stage, this partially dried casein cake was subjected to the extrusion and spheronization process to form 1 mm diameter casein granules. The drying of casein granules was then carried out in the heat pump assisted fluid bed dryer (HP-FBD). Fluid bed dryer (M/s S. B. Panchal and Company, Mumbai, India) utilized the dehumidified air (10% RH) from heat pump system as a drying medium. The casein granules were dried at temperature from 35-55^{o}C and at a constant air velocity of 3 m/s.

Analysis of drying data

The moisture contents of the each sample were determined according to the vacuum oven method (i.e. drying at 70^{o}C and 25 mbar for 12 h) (AOAC, 1995). Moisture ratio (MR) of the sample was determined by the following equation:

$$MR = \frac{M - M_e}{M_0 - M_e} \qquad (4)$$

Where, M is the moisture content of a sample at any time of drying, M_o and M_e stands for initial and equilibrium moisture content, respectively. Also the protein content of the initial and dried samples was determined by the Bradford method of protein analysis (Bradford, 1976).
The effective moisture diffusivity (D_{eff}) during the drying process was calculated by using Fick's second law of diffusion, given by the following equations (Senadeera et al., 2003):

$$MR = \frac{8}{\pi^2} \exp\left[-\frac{\pi^2 D_{eff} t}{L^2} \right] \quad (5)$$

$$MR = \frac{6}{\pi^2} \exp\left[-\frac{\pi^2 D_{eff} t}{r_s^2} \right] \quad (6)$$

Where, D_{eff} is the effective moisture diffusivity or diffusion coefficient (m^2s^{-1}), t is drying time, and MR is the dimensionless moisture ratio. Equation (5) is used in case of slab drying, where L is slab thickness (m) and Equation (6) is for the sphere having radius r_s (m).

RESULTS AND DISCUSSION

Feed suspension properties

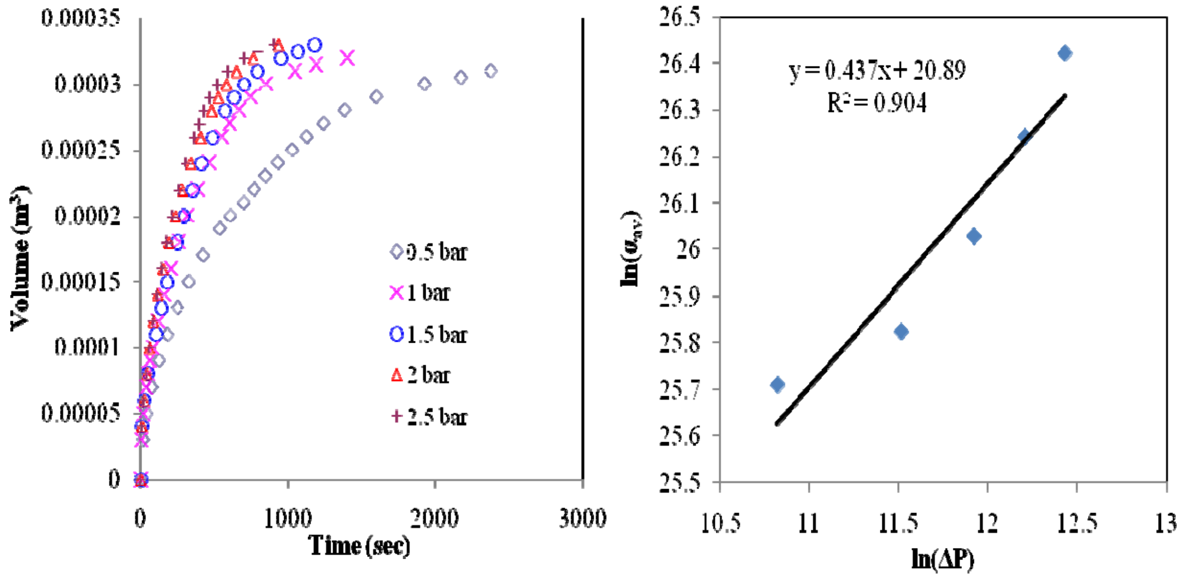
The solid in the feed suspension is primarily the precipitated Casein and the liquid (filtrate) mainly contains the soluble lactose and whey proteins. The solid and liquid densities were almost same at 1005.8 kgm^{-3} and 1009.3 kgm^{-3}, respectively. The filtrate has a viscosity of 0.0025 Nsm^{-2}. The particle size of the suspension was measured by the laser diffraction particle size analyzer (LS 230, Beckman Coulter Inc.) and the mean particle size was found to be 181.3 µm. Table 1 shows the particle size distribution of the feed suspension.

Table 1. Particle size distribution.

Size, µm	19.84	54.12	147.1	272.7	403.5
% < Size	10	25	50	75	90

Filtration of casein suspension

To study the effect of operating pressure on cake properties, the casein suspension was filtered in the pressure range between 0.5 to 2.5 bars. The effect of pressure on rate of filtration and average specific cake resistance is shown in Figure 1 (a) and (b), respectively. It shows that, with an increase in pressure from 0.5 bar to 1 bar, the rate of filtration increases drastically. Further increase in pressure marginally increases the filtration rate. This may be due to corresponding increase in the specific cake resistance at higher pressures.
A cake solids fraction (C_{av}) also changes with pressure and a plot similar to Figure 2 (b) can be plotted for C_{av} as a function of pressure. As can be seen from Figure 1 (b), the relationship between α_{av} and Δp is not linear in the region of lower pressure and the slope becomes steeper at higher pressure. This behavior suggests the significance of higher pressure influence on the average specific cake resistance. It is therefore desirable to consider different ranges of pressure to evaluate the constants of Equations (2) and (3). These are tabulated in Table 2.



(a) (b)

Figure 1. Effect of pressure on: (a) rate of filtration (b) Average Specific cake resistance

Table 2. Filtration parameters

Sr. No.	Pressure range (bar)	n	α_o	β	C_o
1	0.5 to 2.5	0.438	2.10×10^9	0.063	0.132
2	1 to 2.5	0.651	2.56×10^8	0.089	0.096
3	1.5 to 2.5	0.770	9.04×10^7	0.115	0.070

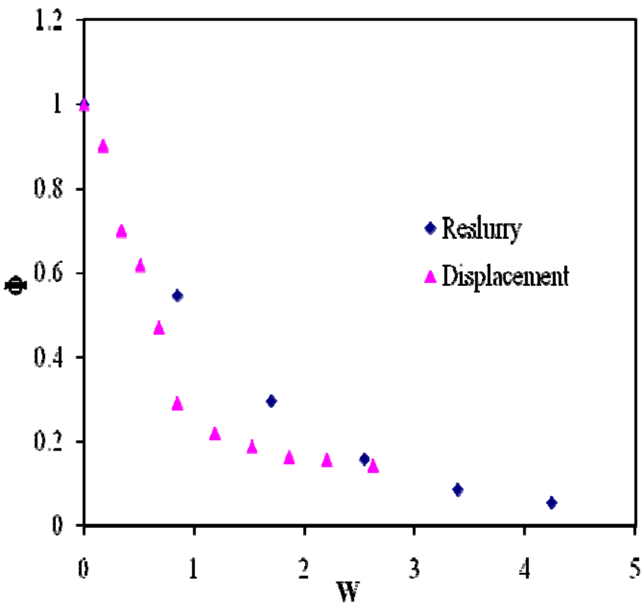


Figure 2. Washing curve.

Table 2 shows that, the compressibility index (n) increases with an increase in pressure i.e. the casein cake show more compressible nature at a pressure above 1.5 bars. Hence, with increasing pressure above 1.5 bars, the filtration rate does not increased significantly as can be seen from Figure 1 (a). Also, the coefficient β increases with pressure, showing greater tendency for compression deliquoring.

Cake washing

The casein cake washing was carried out by reslurrying as well as by displacement method. The plot of dimensionless solute concentration in washings (ϕ) versus wash ratio (W) is shown in Figure 2. It shows that, in displacement washing, ϕ decreases almost linearly up to the wash ratio of 1.2, but after that no significant change in ϕ was observed. This could be mainly due to the channeling of the wash liquor through the cake. The reslurry washing is like the continuous dilution of the retained liquid in cake by wash liquor and ϕ decreases exponentially with W.

Vacuum Tray Drying

Casein cake was dried in vacuum tray dryer at a temperature 40, 50 and 60°C. The moisture profile and the corresponding drying rate curve for casein drying is shown in Figure 3. The moisture ratio (MR) decreased exponentially with the drying time. Vacuum tray drying of casein shows typical drying rate curve including initial constant drying rate period followed by the falling rate period. The drying rates were found to increase with the drying temperature as attributed to the increase in the moisture diffusivity with temperature as can be seen from Table 3.

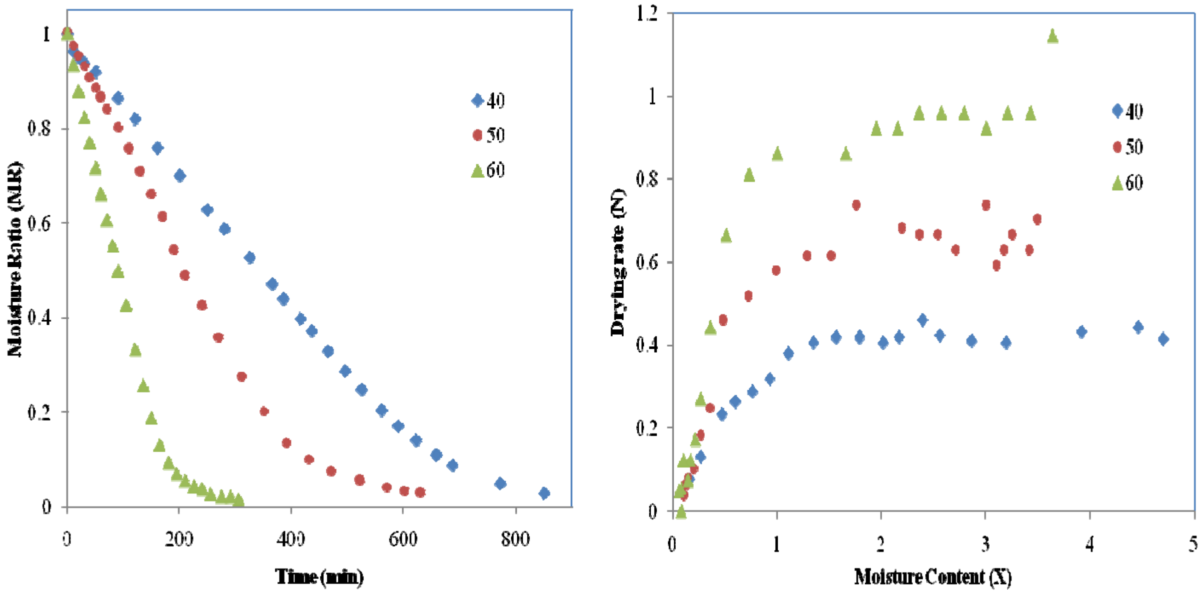


Figure 3. Moisture profile and drying rate curve for vacuum tray dryer (40, 50, and 60°C).

Table 3. Different drying characteristics for casein drying.

Dryer Type	**Temperature (°C)**	**Moisture Content (kg water kg dry solid^{-1})**	**Protein Content (mg gm dry solid^{-1})**	**% Protein retention**	**Diffusivity, D_{eff} (m^2s^{-1})**
Vacuum Dryer	40	0.163	891	95.81	$3.39x10^{-10}$
	50	0.113	875	94.09	$5.39x10^{-10}$
	60	0.066	848	91.18	$1.51x10^{-9}$
HP-FBD (Secondary Drying)	35	0.099	885	95.16	$5.04x10^{-11}$
	45	0.093	871	93.66	$7.42x10^{-11}$
	55	0.0943	855	91.94	$1.11x10^{-10}$

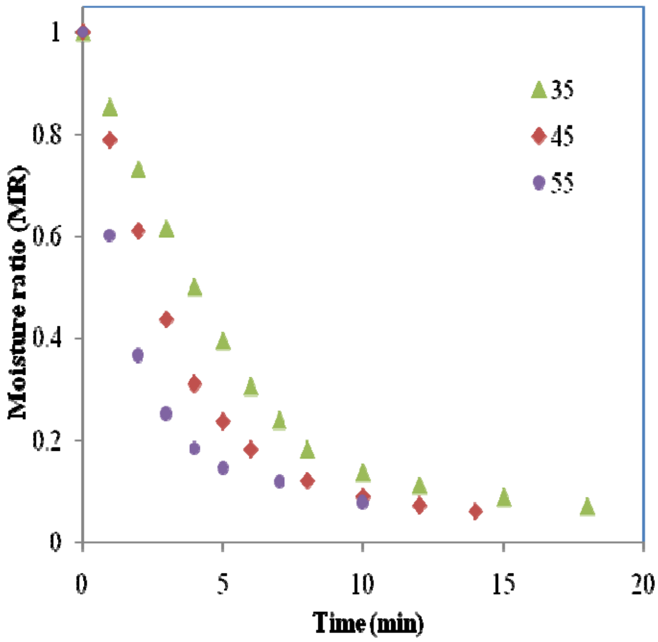


Figure 4. Moisture profile in HP-FBD (35, 45 and 55°C)

Two Step Drying

As can be seen from Figure 3, the falling rate period starts at moisture content of approximately 1.5 kg water/kg dry solid and thereafter the drying rate reduces drastically. In two step drying, the primary drying was carried out by vacuum tray dryer upto moisture content of approximately 1 kg water/kg dry solid and then extrusion and spheronization process was employed to form casein granules. The secondary drying of the casein granules was carried out with HP-FBD at temperature of 35, 45 and 55°C. The moisture profile for the drying of casein granules is shown in Figure 4. The drying time in HP-FBD was drastically reduces due to the increased drying rate by increase in the surface area for drying.

Table 3 shows different drying characteristics for the casein drying under the experimental conditions studied. The dried casein powder/granules show good protein retention of more than 90% even at higher temperature. As expected, the effective moisture diffusivity was found to increase with the drying temperature and it varied between 5.04×10^{-11} m^2s^{-1} and 1.51×10^{-9} m^2s^{-1}.

CONCLUSION

The casein forms a compressible cake and the average specific cake resistance and hence the compressibility index increases with pressure, therefore no significant increase in filtration rate was observed at higher pressure. Casein cake also shows the higher tendency for compression deliquoring as indicated by the higher value of coefficient β. The displacement cake washing mainly depends on the structure of cake formed during filtration, if the channeling of the wash liquor through cake occurs, then displacement washing has lower efficiency. In such cases, the combination of displacement and reslurry washing may serves the purpose.

Casein drying in vacuum tray dryer shows a typical drying rate curve with small constant rate period followed by the large falling rate period. The drying rate and moisture diffusivity increases substantially with increase in the drying temperature. The two step drying process employed for casein drying was found to be effective in reducing the drying time; especially in the falling rate period.

LIST OF SYMBOLS

α_{av}	Average specific cake resistance (mkg^{-1})
R	Filter medium resistance (m^{-1})
V	Filtrate volume (m^3)
A	Filter area (m^2)

Δp	Applied pressure difference (bar)
c	effective feed concentration (kgm^{-3})
μ	Filtrate viscosity (NSm^{-2})
ε_{av}	average cake porosity (-)
n	compressibility index (-)
α_o	Specific cake resistance at unit pressure (mkg^{-1})
C_o	Cake solids fraction at unit pressure (-)
β	Filtration constant (-)
ϕ	Dimensionless solute concentration in washings (-)
W	Wash ratio (-)
MR	Dimensionless moisture ratio (-)
M	Moisture content (kg water kg dry solids^{-1})
t	Drying time (hr)
N	Drying rate (kg m^{-2} hr^{-1})
D_{eff}	Effective moisture diffusivity (m^2s^{-1})
L	Slab thickness (m)
r_s	Radius of sphere (m)

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