Centrifugal Dewatering of Acid Casein Curd: Effect of Casein Manufacturing and Centrifugation Variables on Curd Compression in a Laboratory Centrifuge

P.A. Munro and H. J. Van Til
Department of Food Technology, Massey University, Palmerston North, New Zealand
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Data relevant to curd compression in a horizontal, solid bowl decanter centrifuge have been obtained by studying the dewatering of acid casein curd in a batch laboratory centrifuge. Analysis of curd compression under centrifugal force predicts a moisture content gradient in the dewatered curd from a maximum at the curd-liquid interface to a minimum at the centrifuge bowl wall. This moisture content gradient was also measured experimentally, and its practical implications are discussed. Increases in centrifugal force, centrifugation time, and centrifugation temperature all caused a marked decrease in dewatered curd moisture content, whereas increases in precipitation pH and maximum washing temperature caused a smaller decrease in dewatered curd moisture content.

INTRODUCTION

Centrifuges are widely used for the recovery of biological materials. Their use for the recovery of protein precipitates, for the dewatering of waste water sludges, and for cell recovery from fermentation broths has been reviewed recently. Most research work on centrifuge performance and design has been performed by equipment manufacturers and has been empirical in nature. Relatively little fundamental work has been performed, and that which has been done has concentrated on liquid clarification, i.e. particle recovery. However, solid deliquoring is equally important for many applications, for instance where the solid-liquid separation is followed by a washing process or a thermal drying operation. For porous, compressible biological materials, deliquored solid moisture content can be varied greatly by altering either solid preparation variables or centrifugation variables. This article explores the effect of precipitate manufacturing and centrifugation conditions on the centrifugal compression of acid casein curd.

The manufacturing process for acid casein involves isoelectric precipitation of casein from skim milk followed by acidulation. whey separation, multiple stage washing with water, mechanical dewatering and thermal drying of the resultant precipitate. The mechanical dewatering operation has been carried out with roller presses, belt presses, screw presses, and decanter centrifuges. A previous laboratory study on casein dewatering focused on pressing since this was easiest to study and presses were the predominant industrial dewatering device. A laboratory pressure cell was used to study the expression of water from washed casein curd at room temperature. The most significant finding was that at applied pressures greater than ca. 104 kPa the drainage surface of the curd became sealed (restricting water flow from the curd) and developed a translucent, plasticlike appearance. Decanter centrifuges are now widely used for dewheying and dewatering casein curd. Their advantages are good curd dewatering, good control of curd particle size, good recovery of casein fines, and relatively hygienic design. Their disadvantages are high capital costs, high repair and maintenance costs, high noise levels, and the need for good process control. Decanter centrifuges were included in a comparative study of three pilotscale casein dewatering machines. This paper describes experiments on casein curd compression which are relevant to curd dewatering in a decanter centrifuge.

CONCEPTUAL ANALYSIS OF CURD DEWATERING IN A DECANTER CENTRIFUGE

The operating principles of the horizontal, solid-bowl (decanter) centrifuge are well known. A solid-liquid slurry is fed into the central portion of the bowl and clarified liquid and dewatered solid leave from opposite ends of the bowl. Conceptually the centrifuge can be divided into three zones. At the cylindrical end of the bowl fine particles are centrifugally settled from the liquid in a “liquid clarification zone.” In the center of the bowl there is a “curd compression zone” where the entering particles are thrown to the outside of the bowl and are compressed by centrifugal force, and water is thus squeezed from the curd. At the conical end of the bowl further liquid drains from the curd as it is conveyed up the dry beach in the “curd drainage zone.” The liquid clarification zone may be
analyzed using established techniques for considering the settling of discrete particles from a dilute slurry. The curd drainage zone is difficult to study experimentally, but some analysis of scroll conveying has been attempted. This article considers the curd compression zone. Curd compression is much more important for porous, compressible particles such as casein curd than for the incompressible particles often handled in decanter centrifuges.

The principles of centrifugal compression of porous protein precipitates are similar to those for gravity thickening of sewage sludges, which have been well studied. The compressive force expressing liquid from a given layer of particles in a centrifugal field depends on the density difference between the particle matrix or solid component and the liquid, and also on the mass of particles above that layer in the centrifuge. There is thus a gradient of compressive force in the settled curd from zero at the curd—liquid interface to a maximum at the wall of the centrifuge bowl. This implies that there should be a gradient in moisture content in the settled curd from a minimum at the wall of the centrifuge bowl to a maximum at the curd—liquor interface. Such gradients in moisture content have been demonstrated experimentally for gravity thickening of sewage sludge.

The compressive stress useful for expressing liquid at any location in the compressed curd layer is given by:

\[ \sigma = \sigma_T - p \]  

(1)

Where \( \sigma_T \) is the compressive stress generated by the mass of solids above this layer in the centrifuge bowl (Pa) and \( p \) is the drag force exerted on the particles by the upward flow of liquid through the settled solids (Pa), and

\[ \sigma_T = (1 - \rho_L/\rho_s)w^2 \int_0^x rC \, dx \]  

(2)

where \( \rho_L \) and \( \rho_s \) are densities of liquid and solid, respectively (kg/m³); \( w \) is the centrifugal angular velocity (s⁻¹); \( r \) is the centrifugal radius (m); \( x \) is the distance below the curd—liquor interface (m); and \( C \) is the solids concentration in the sedimented curd at the radial position \( x \) (kg/m³). The first term in eq. 2 is the stress created by centrifugal force on the solid, and the second term is the buoyancy provided by the liquid in the sample. Under steady-state conditions, i.e. after long centrifugation times, \( p \) becomes very small and \( \sigma = \sigma_T \). Equation (2) may then be used to calculate the compressive stress forcing liquid from the curd, and to predict the general shape of the moisture content profile in the settled curd layer.

**EXPERIMENTAL**

**Preparation of Casein Curd**

Low-heat skim milk powder (N. Z. Cooperative Dairy Company Limited, Hamilton, New Zealand) was dispersed in warm distilled water to produce skim milk with a total solids content of 9% (w/w). Acid casein curd was prepared by heating 2 L skim milk to 53°C, stirring vigorously, and rapidly adding enough 0.3M H₂SO₄ to obtain the desired pH. The curd—whey suspension was stirred slowly and a - = o -T . Equation (2) may then be used to calculate the moisture content profile down the tube. The comparative mechanical strength of various curd layers was determined using a penetrometer device. A 3-mm-diameter spherical probe was attached to the crosshead of an Instron Universal Testing Machine (Instron Ltd, High Wycombe, Bucks., England). The probe was driven axially into a tube of centrifugally compressed casein curd, and penetration force was recorded versus penetration distance.

**Centrifugation Experiments**

Centrifugation experiments were performed in 50 mL round-bottomed tubes in a Sorvall RC5C refrigerated centrifuge (Sorvall Products, Wilmington, DE). Most experiments used an SS-34 angular rotor. Centrifugation radius at the bottom of the tubes was 107 mm. However, centrifugal force values were calculated using a radius of 94.5 mm, which corresponded approximately to the mid-point of the curd samples at the beginning of centrifugation. The required centrifugation temperature was achieved by incubating a curd—water slurry in a water bath at the required temperature. For temperatures above ambient the centrifuge was run at medium speed until frictional heat had produced the desired temperature. The centrifuge was maintained at the required temperature by the refrigeration circuit. The curd was drained on a stainless-steel mesh screen (0.39-mm apertures), and 20 g was placed in each of three centrifuge tubes. A sample of the drained curd was taken for moisture content determination. After centrifugation the surface water was poured from each centrifuge tube, and all the curd from each tube was placed in a moisture dish. Mean moisture content of the centrifuged curd sample was determined by oven drying for 18 h at 105°C.

To investigate the layering of curd under centrifugal force in a centrifuge tube, experiments were performed as above but with a HB-4 swing-out rotor. Centrifugation radius at the bottom of the tubes was 146 mm, and centrifugal force values were calculated using an average centrifugation radius of 120 mm. After centrifugation, curd layers 5 mm thick were removed from the centrifuge tube and placed in separate moisture dishes in order to measure the moisture content profile down the tube. The comparative mechanical strength of various curd layers was determined using a penetrometer device. A 3-mm-diameter spherical probe was attached to the crosshead of an Instron Universal Testing Machine (Instron Ltd, High Wycombe, Bucks., England). The probe was driven axially into a tube of centrifugally compressed casein curd, and penetration force was recorded versus penetration distance.
RESULTS AND DISCUSSION

Layering of Curd under Centrifugal Force

Results for curd moisture content versus depth in the tube (Fig. 1) show a systematic variation from drier curd at the bottom of the tube to much wetter curd near the liquid interface. Penetration force data (Fig. 2) also indicate layering in the centrifuge tube with more compact and therefore stronger curd at the bottom of the tube. It was also observed that curd plasticized sooner and more easily at the bottom of the tube. Thus, all three pieces of experimental evidence indicate drier, more compact curd at the bottom of the centrifuge tube. Moisture content results qualitatively similar to these were reported by Sokolov and Sedov for the centrifugation of cottage cheese.

This variation in curd moisture content with depth in the tube is predicted by eq. 2. As a first approximation, if variations in r and C with depth in the tube are neglected, eq. 2 becomes:

\[ \sigma_T = \frac{\rho_s}{\rho_L} w r C x \]

i.e. an approximately linear increase in compressive stress with depth into the curd layer. Curves for dewatered curd moisture content versus centrifugal force (Fig. 3) or versus applied pressure for press dewatering are both hyperbolic shaped with a vertical asymptote at zero pressure and a horizontal asymptote at 55–58% (w/w) moisture content. This predicts the general shape exhibited in Figure 1 for curd moisture content versus depth with a low slope at the bottom of the tube where further increases in pressure cause little decrease in moisture content. Calculation of \( \sigma_T \) for the experiment conducted at 4000g to generate Figure 1 indicates a compressive stress of approximately 131 kPa at the bottom of the centrifuge tube. This is in the range of applied pressures where increased pressure has little effect on curd moisture content. Since r and C both increase somewhat as one passes down through the curd layer, \( \sigma_T \) will increase somewhat more rapidly with x than predicted by the linear relation in eq. 3.

Particle segregation in the centrifuge tube with larger, drier particles settling first to the bottom and small ones at the top might also be used to help explain the results in Figure 1. However, this is unlikely to be important since the particles are large, mainly 1–6 mm in diameter, and all settle by gravity well before they are put into the centrifuge. The major practical implication of the variation in curd moisture content between curd layers is that centrifuges for dewatering compressible materials should turn the solid over during dewatering so that the damp upper layers of curd are also effectively dewatered. The decanter centrifuge achieves turn over of solid by using a screw to convey the curd.

In all subsequent experiments the mean moisture content of the dewatered curd was determined by taking all the curd from each centrifuge tube and drying the whole sample.

Effect of Centrifugation Variables on Moisture Content

Dewatered curd moisture content decreased with increasing centrifugal force (Fig. 3). However, centrifugal accelerations above 6000g have relatively little effect in further reducing moisture content. Similar results were reported for press dewatering with applied pressures above 100 kPa having virtually no effect on curd dewatering. The cutoff was more dramatic for press dewatering because of plasticization of curd at the drainage surface.

Dewatered curd moisture content decreased with increasing centrifugation time (Fig. 4), producing a similar curve shape to that versus centrifugal force (Fig. 3). Similar results were presented for press dewatering. For press dewatering it was possible to continuously monitor curd...
thickness and hence curd moisture content. The decrease in curd moisture content with time was still continuing after pressing at 207 kPa for $8 \times 10^3$ s. From the results in Figures 3 and 4 centrifugation conditions of 4060g for 20 min were chosen for the rest of the experimental program.

Variations in sample mass had relatively little effect on dewatered curd moisture content (Fig. 5). Presumably the effect of greater pressure on the curd with a larger mass of curd above it is balanced by the effect of the longer drainage distance for moisture to travel out of the curd. Adding an extra 15 g water to a centrifuge tube to increase the hydrostatic pressure on the curd had no effect on dewatered curd moisture content.

Increasing centrifugation temperature, which normally equals the temperature of the last washing stage in a casein plant, caused a marked decrease in curd moisture content both before and after centrifugation (Fig. 6). The upper temperature was limited to 40°C by the method of temperature control used. No curd plasticization was observed at 25°C or below, moderate plasticization at 30 or 35°C and extensive plasticization at 40°C. Increasing wash water temperature causes substantial shrinkage of casein curd particles leading to lower moisture contents after screen drainage. 

Munro and co-workers studied the behavior of three pilot-scale dewatering machines between 20 and 50°C. The results gave dependencies of moisture content (% w/w) on temperature of $-0.11^\circ C^{-1}$ for the roller press, $-0.94^\circ C^{-1}$ for the screw press, and $-0.83^\circ C^{-1}$ for the decanter centrifuge. The low temperature dependency of the roller press was attributed to the simple one-dimensional pressing in the roller press which does not expose new material to the
drainage surface, and to sealing of the drainage surface at high temperatures. Centrifugal dewatering in a batch laboratory centrifuge employs simple one-dimensional water removal, but there is no perforated drainage surface so surface sealing does not occur. An intermediate value for temperature dependency might therefore have been expected.

**Effect of Casein Manufacturing Variables on Moisture Content**

Precipitation pH had a marked effect on curd moisture content before centrifugation, and a less pronounced effect on dewatered curd moisture content (Fig. 7). The effect of precipitation pH on casein curd properties is well known with a high precipitation pH producing large, strong curd particles with a comparatively low moisture content and usually a high calcium content.\(^{10,13,14}\) These effects are attributed to electrostatic repulsion below the isoelectric point and to calcium binding above the isoelectric point.

Increasing the hot wash temperature, i.e., the maximum washing temperature encountered, caused a small but significant decrease in curd moisture content both before and after centrifugation (Fig. 8). The shrinkage of casein curd at high washing temperatures is generally regarded as reversible.\(^{11,12}\) However, the results in Figure 8 indicate some irreversible changes to the curd on exposure to high washing temperatures. Hobman and Hughes\(^{13}\) found that hot wash temperature had an important effect on casein grindability with higher hot wash temperatures making casein easier to grind. This also suggests irreversible changes to the curd on exposure to high washing temperatures.

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**References**