

## Rheological properties of buttermilk pellets manufactured by a new die pelleting rig of a texture analyzer

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### ABSTRACT

The shelf life of buttermilk powder can be extended by pelleting process due to decreased water absorption. Cheaper transport and storage costs are produced when pelleting products. This study shows that binders like milk powder are required to pellet buttermilk. Solubility in static water decreased when pelleting the powders, consequently the pellets must be added in a flow stream or been disintegrated for its application in processes.

### INTRODUCTION

Buttermilk is a by- product produced from the churning process of cream in butter manufacture. Buttermilk powder is obtained by removing water from liquid buttermilk by spray drying. The primary object of making a buttermilk powder is to preserve the product in a shelf-stable powder form without a need for refrigerated storage. Removing moisture from buttermilk inhibits microbial growth and delivers a shelf-life of six to nine months. This stability also makes it more preferable for applications in the field of functional technology<sup>1</sup>.

During the churning process, material derived from milk fat globule membrane (MFGM), migrates to the buttermilk fraction<sup>2</sup>. This results in a high content of phospholipids in buttermilk and buttermilk powder. Phospholipids from MFGM are

amphiphilic and contain a hydrophilic head, and two hydrocarbon tails on a glycerol backbone<sup>3</sup>. The low-molecular phospholipids are surface-active and are often added to different foods as emulsifier. Buttermilk powder also contains concentrated calcium, lactose, protein and other important nutrients.

Buttermilk is sold as powder and consequently presents all logistic disadvantages of powders, that is, it requires large storage and transport volumes and can re-absorb water. Pelleting or tableting of powders is usually done to increase bulk density and in consequence reduce transport and storage costs. Another advantage of pelleted powders compared to bulk powders is that pellets normally present an extended shelf life as the water adsorption is reduced. Pellet hardness is important to be predicted to tell how the buttermilk pellets will behave when subjected to the mechanical forces produced during packaging, transport and further handle in the trading line. Solubility is important as well to determine how easy or hard the buttermilk pellets can be dissolved, a rapid dissolution might be required for an immediate use, while in other cases a gradual release of buttermilk as ingredient might be required when for example releasing small amounts buttermilk in a flow stream.

The main goal of this research is to characterize the compactibility (i.e. the ability of a fine material to form strong compacts<sup>4</sup>) of buttermilk powder having different concentrations of milk powder acting as binder. Solubility of the compacts (pellets) is also studied.

## MATERIALS AND METHODS

### Pelleting method

Buttermilk powder was pelleted using a design variant of the capillary rheometer presented at NRC2009 by Salas-Bringas et al<sup>5</sup> and Rukke et al<sup>6</sup>. The equipment is a die pelleting rig that is attached to a Lloyd LR5K Plus texture analyzer. Using a 0.0094 m diameter rod (see Fig. 1), the equipment is able to produce compacting stresses up to 72 MPa. The die pelleting rig consists in a barrel having a cylindrical compressing channel and a blank or closed die.

In reference to Fig. 1, a small amount of the testing powder (~ 0.56 g) was added in the compressing channel. The pressing speed was set to 4 mm/min with a short retention time (1 s). After the retention time period, the pressure was released and the blank die removed. The same compressing rod and texture analyzer was used to gently move the pellet inside the channel until its discharge.

The sources of error in this type of compression tests is that at the side walls of the compressing channel, the friction reduces the stress from top to bottom<sup>7</sup>. This can result in a buttermilk pellet having a density gradient, with its lowest density close to the blank die. For this reason, it were produced short pellets having an average of 0.0076 m length and 0.0095 mm of diameter.

The pelleting temperature was room temperature (21 °C).

### Raw materials

*Buttermilk powder:* The spray dried buttermilk powder that was used was

produced by TINE, Norwegian Dairies Association, Norway. Common nutritional values for buttermilk powder are presented in Table 1. A moisture content of 3.9% (w.b.) was determined drying 0.6 g of powder milk at 105 °C overnight.

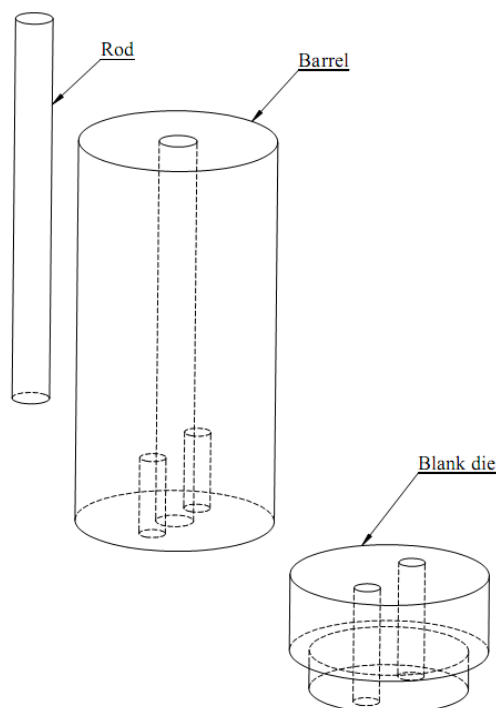


Figure 1. Exploded view of the barrel-blank die setup.

Table 1. Nutritional values of buttermilk, low fat per 100 g.

Energy	1660 kJ
Carbohydrates	5.1 g
Fat	0.7 g
Protein	3.1 g
Calcium	105 mg

*Milk powder:* The milk powder that was used was Småfolk 1, the nutritional values are presented in Table 2. A moisture content of 2.66 % (w.b.) was determined drying 0.6 g of powder milk at 105 °C overnight.

Table 2. Nutritional values of Småfolk 1 milk powder per 100 g according to producer.

Energy	2150 kJ
Carbohydrates	57 g
Fat	27 g
Protein	10 g
Calcium	320 mg

#### Compression of buttermilk and milk powder

A first preliminary pelleting at 70 MPa of 100% buttermilk powder resulted in a poorly bounded pellet, that disintegrates when touch, reason why it was not possible to measure its physical strength. These preliminary results indicated the need of using binders. It was decided to use milk powder because of its good binding properties and relatively similar nature. In consequence, it was produced pellets having 50, 60, 70, 80 and 90% (w/w) of buttermilk in milk powder. It was also produced pellets made of 100% milk powder to characterize its rheological properties as binder.

All buttermilk pellets were produced using 40 MPa of normal stress. It was not used higher compacting stresses as they produced jumps in the rod displacements because of the presence of high friction at the walls of the compressing channel. Consequently, a poor powder flow occurred at high consolidating stresses.

#### Physical strength of buttermilk and milk pellets

The stress analysis of the pellets showed that they were brittle and presented a tensile failure when pressed diametrically (ref. Fig. 3 and Fig. 4) and therefore it was possible to determine the maximum tensile stress based on an equation for cylindrical specimens as follows<sup>8,9</sup>:

$$\sigma \approx \frac{F}{\pi r L} \quad (1)$$

were  $F$  is the first peak force in Newtons during a diametrical compression (normal

force) in a Lloyd LR5K Plus texture analyzer.  $L$  is the pellet length and  $r$  the pellet radius. All stress analysis were repeated three times.

#### Solubility tests

Solubility was determined by leaving each of the samples of buttermilk powder, milk powder and pellets in 100 ml of distilled water at room temperature for 20 minutes in the containers shown in Fig. 2.

The experimental set up used for the solubility test consist in a graduated container having an open top and bottom, at the bottom of the container a pre-dried and weighed paper filter of 45 $\mu$ m is added to collect the non-dissolved particles. The container and paper filter are connected to a piping system where vacuum is applied through a pump. A relatively quick drainage occurred after the 20 minutes test having the sample in water.

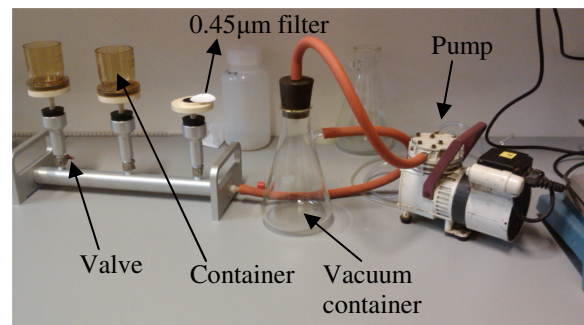


Figure 2. Arrangement used for solubility tests.

After draining the water from the containers, the filters were placed in a pre-weighed metallic plate and moved to an oven at 105 °C overnight. The amount of dry matter (% w.b.) over the paper was calculated.

#### RESULTS AND DISCUSSIONS

All pellets produced at 40 MPa and at different concentrations of buttermilk and powder milk had a brittle structure. An example of the brittleness is shown in Fig. 3

for two pellets made at high concentrations of buttermilk and using pure powder milk. Additionally a picture of a pellet during diametrical compression is presented in Fig. 4. According to the description given by Sinka et al<sup>4</sup>, and the picture taken from one of the buttermilk pellets, it can be concluded that a tensile failure is present in the pellets when pressed diametrically.

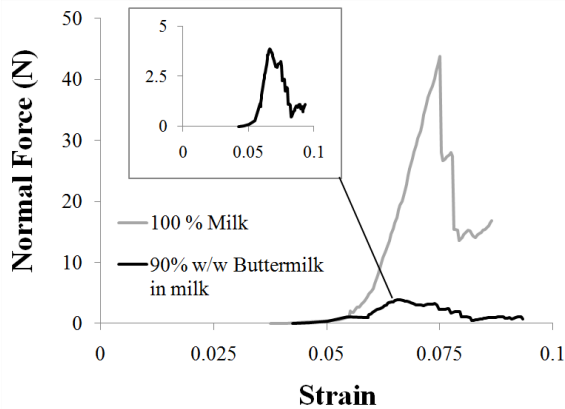


Figure 3. Texture analysis of two pellets. One produced with high concentrations of buttermilk in powder milk (90% w/w) and the other of pure powder milk. The plot shows the brittle nature of both pellets.

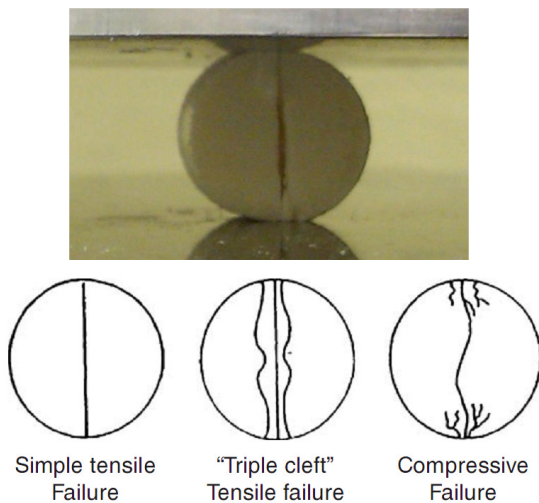


Figure 4. At the top, tensile failure of a buttermilk in powder milk pellet during a diametrical compression. At the bottom, failure modes in diametrical compression test<sup>4, 10</sup>.

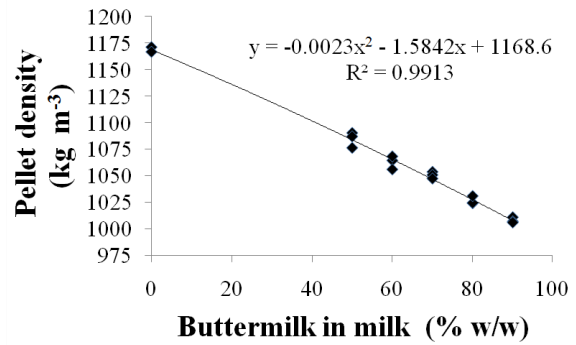


Figure 5. Densities of pellets at different concentrations of buttermilk in milk. The plot also shows the density of a pellet made of pure powder milk. Each test was repeated three times.

The pellets made of pure powder milk present the highest densities. Pellet density decreases at increased additions of buttermilk powder, see Fig. 5.

The contribution of powder milk as a binder is shown in Fig. 6. Additions of powder milk increased the strength in an exponential way. It was not possible to test a pellet having 100% buttermilk because it disintegrates when manually handle.

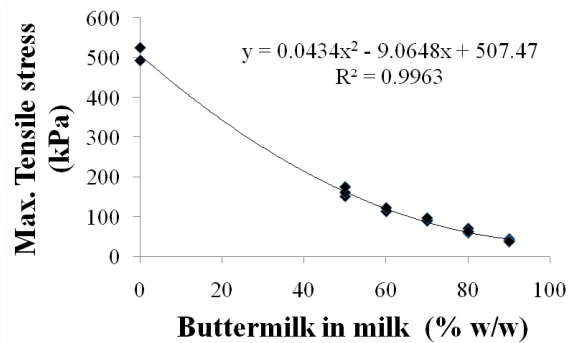


Figure 6. Maximum tensile stresses for pellets made at different concentrations of buttermilk in milk. The plot also shows the stress values for pure milk pellet. The tests were repeated three times.

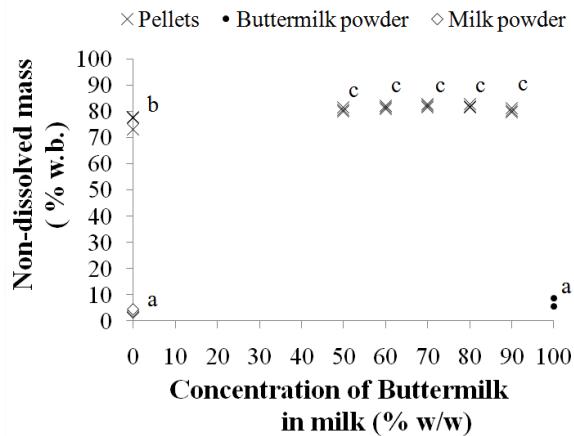


Figure 7. Solubility of buttermilk in milk. The plot uses the non-dissolved mass (% w.b.) as a measurement of solubility in water. Different letters indicate significant differences ( $p < 0.05$ ). The number of samples,  $n$ , is three.

The solubility results that are presented in Fig. 7 show high values of solubility for both powders after 30 minutes in water. It were no significant differences between them ( $p > 0.05$ ) using one-way ANOVA,  $F$ -test. Solubility decreases considerably when pelleting pure milk and mixtures of buttermilk in milk. The pellets made with 100% milk powder presented slightly higher solubility (enough to be significantly different,  $p < 0.05$ ) compared to the pellets made from the mixture buttermilk in milk (50 – 90% w/w). No significant changes in solubility were found ( $p > 0.05$ ) among the pellets made from the mixture buttermilk – milk (50 – 90% w/w).

The test showed a large reduction in solubility when the powders were pelleted. The tests were conducted using un-disturbed water, however it is of future interest to discover how much the solubility can increase by having the pellets in a continuous flow stream, so the pellet solubility could increase by the shear stresses given by the dissolving medium. Another possibility of use for these pellets is to disintegrate them into powder again

before use since only small forces are needed (ref. Fig. 3 and Fig. 6).

## CONSLUSIONS

Buttermilk powder presents poor consolidating properties and binders are needed to produce stronger compacts.

Powder milk as binder increases the density and strength of buttermilk pellets. Only 10% w/w of powder milk in buttermilk was necessary to produce a pellet strong enough to resists a manual handling.

Buttermilk and milk in powder form presented no significant differences ( $p > 0.05$ ) in solubility.

Small, but statistically significant differences ( $p < 0.05$ ) in solubility were found between pellets made with pure milk and the ones made with mixtures of buttermilk in milk (50 – 90% w/w). The pellets having mixtures of buttermilk-milk had the lowest solubility.

Undisturbed water at room temperature is not a good medium to dissolve these pellets. Further research considering a flow stream of a solution (e.g. water) over the pellets is of interest to determine the best way of using buttermilk pellets in processes.

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