Summary and Conclusions

In the Irish dairy industry, casein/caseinates have shown variable behaviour in their functionality. While such variation cannot be explained by gross compositional analysis of the ingredient, little is known about factors that affect functionality, such as 1) the level of individual casein components in the ingredient; 2) the interaction/aggregation behaviour of these components; 3) the influence of such interactions on the functional behaviour of casein/caseinate, and 4) the effect of chemical and thermal treatments used in the manufacture of casein/caseinate on the casein interactions and functionality.

Hence, the overall objective of this project was to investigate the effects of key processing steps in the industrial production of acid casein on the characteristics and functionality of sodium caseinate with particular emphasis on analytical/functionality testing and seasonal/lactational effects on the original milk.

The main conclusions were as follows:

- The most significant result indicates that drying and concentration after washing of the acid casein curd are responsible for alterations in the structure of casein, which result in sodium caseinates with different properties. This was confirmed in the case of two acid casein plants investigated which showed similar results even though using different washing and drying technologies. This difference due to the drying step may be further amplified depending upon whether commercial sodium caseinate is manufactured from acid casein in the dried or wet curd state.

- The analytical and functional testing methodology adapted in our laboratory proved effective in predicting the effects of processing steps on the functionality of sodium caseinate. In particular, the ability to detect the presence of aggregate formation was particularly important. The database generated subsequently helped an acid casein
manufacturer in modifying its process(es) to manufacture experimental sodium caseinate for specific food end-uses.

- Progress was greatly facilitated by the collaboration of individual manufacturers in the sourcing of problem samples from previously manufactured codes, and facilitating access to process plant during production.
- In a commercial application of the database, confidential work was undertaken on behalf of a client. Experimentally-produced sodium caseinate ingredients were evaluated using our adapted functionality testing methods and based on the results, the company was able to modify its process(es) to produce sodium caseinates with functionality for specific food end-users.
- It was concluded that while processing parameters in the production of acid casein can have a significant effect on the functional behaviour of the resultant sodium caseinate, the ability to assess this change in functional behaviour, through relevant functional testing, was equally important.

**Research and Results**

In late 1995, acid casein curd was sampled at three stages (after acidification/dewheying, after washing/dewatering, and after drying) of its manufacture at two Irish industrial plants which use different processes. This study was repeated, again with acid caseins made from late-lactation milk (1996), and with mid-lactation milk (1997) from one of the two original plants.

Since acid casein is insoluble as a result of precipitation at its isoelectric pH, conversion to a solubilised form is necessary in order to evaluate its functionality. The most common form of solubilisation is by neutralisation using sodium hydroxide to produce the sodium-form of the protein. Sodium caseinates were prepared in the laboratory from these acid casein samples by a standardised procedure using mild processing treatments in order to minimise damage to the protein. Freeze drying was selected in preference to the hot air drying approaches used industrially. A reference sodium caseinate was produced in the laboratory from the original silo milk (see Fig. 1). Adverse effects on casein during processing were reflected by the tendency of the protein to form aggregates - an association/interaction of individual casein fractions. The extent of aggregation could be monitored using size exclusion HPLC which provides a graphical representation of the protein according to molecular weight map. Increased aggregation is indicated by a larger peak area which is eluted from chromatographic column at the start of a run.
Functional tests which determined stability to alcohol, sensitivity to calcium, viscosity, and behaviour in a fat-free cream liqueur analogue system, were adapted to characterise the casein samples in their solubilised, sodium caseinate form.

The sodium caseinates from the acid caseins of late-lactation milk showed little differences in their gross compositions, both within and between the two casein plants. However, functionality tests indicated that the processing stages in the conversion of skim milk to acid casein, particularly the drying step, affected the aggregation behaviour, stability to alcohol, sensitivity to precipitation by calcium and, viscosity of cream liqueur analogues when compared to reference caseinates.

**Alcohol Stability**

There was a major difference in stability to alcohol between sodium caseinates derived from the commercially dried acid casein and the reference caseinate.

The alcohol stability was higher in the reference caseinate than in the caseinates made from commercially dried caseins (Fig. 2). This difference was observed at all sampling periods.
Calcium Sensitivity

The effect of moderate concentrations of calcium ions on the aggregation behaviour of the various caseinate samples was compared. The reference caseinate was more sensitive to calcium under the conditions used than the caseinate from commercially dried acid casein over all sampling periods. (fig 3.) This characteristic could be used to advantage in different food applications depending on whether or not extra stability in the presence of calcium is desirable. Roller-dried caseinates are known to be highly aggregated due to the severity of that drying process, and are particularly insensitive to calcium.

Thus, it is possible to achieve a similar specification to roller-dried casein by combining the effects of different processing steps with spray drying.

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Fig. 2. Alcohol stabilities of sodium caseinate derivatives of caseins from various stages of production

Fig. 3. Calcium sensitivities of sodium derivatives of caseins obtained from various stages of production
Cream Liqueur Analogue

The functionality of the sodium caseinates was evaluated in a fat-free cream liqueur analogue system. The laboratory fat-free cream liqueur analogue system was validated by using commercial 'good' and 'sub-standard' sodium caseinates as judged by their performance in commercial cream liqueurs by two cream liqueur manufacturers. The development of viscosity was measured during storage of the analogues at 45°C for 60-90 days. Commercial 'good' sodium caseinates showed lower viscosity increases during storage in cream liqueur analogues than 'sub-standard' sodium caseinates.

Sodium caseinates prepared from commercially dried acid caseins showed higher final viscosities than the reference sodium caseinates in cream liqueur analogues.

However, (fig 4.) HPLC analyses of the 'sub-standard' sodium caseinates, supplied by the cream liqueur manufacturer, did not show a consistent trend in relation to aggregate formation, and suggests that other factors may be at play.

![Fig 4. Viscosity increase of fat-free cream liqueur analogues containing sodium derivatives of caseins obtained from various stages of production.](image)

Seasonal Variation In Functionality Of Commercial Acid Casein

Sodium caseinates showed variations in sensitivity to precipitation by calcium and stability to precipitation by alcohol, but not in chemical composition and aggregation behaviour. *No obvious trend was observed with the stage of lactation.*

Confidential Industrial Work

Confidential work was undertaken on behalf of an Irish acid casein producer to test its experimental sodium caseinate ingredients using our adapted functionality testing methods. The company made modifications to their processes to produce sodium caseinates with functionalities comparable to the competitors, and with specific food end-uses in mind.
Publications


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