J. Dairy Sci. 88:3798–3809

Impact of Modifications in Acid Development on the Insoluble Calcium Content and Rheological Properties of Cheddar Cheese

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ABSTRACT

Cheddar cheese was made from milk concentrated by reverse osmosis (RO) to increase the lactose content or from whole milk. Manufacturing parameters (pH at coagulant addition, whey drainage, and milling) were altered to produce cheeses with different total Ca contents and low pH values (i.e., <5.0) during ripening. The concentration of insoluble (INSOL) Ca in cheese was measured by cheese juice method, buffering by acid-base titration, rheological properties by small amplitude oscillatory rheometry, and melting properties by UW-Melt Profiler. The INSOL Ca content as a percentage of total Ca in all cheeses rapidly decreased during the first week of aging but surprisingly did not decrease below approximately 41% even in cheeses with a very low pH (e.g., ~4.7). Insoluble Ca content in cheese was positively correlated (r = 0.79) with cheese pH in both RO and nonRO treatments, reflecting the key role of pH and acid development in altering the extent of solubilization of INSOL Ca. The INSOL Ca content in cheese was positively correlated with the maximum loss tangent value from the rheology test and the degree of flow from the UW-Melt Profiler. When cheeses with pH <5.0 where heated in the rheometer the loss tangent values remained low (<0.5), which coincided with limited meltability of Cheddar cheeses. We believe that this lack of meltability was due to the dominant effects of reduced electrostatic repulsion between casein particles at low pH values (<5.0).
(**Key words:** calcium, colloidal calcium phosphate, cheese functionality, casein interaction)

Abbreviation key: CCP = colloidal calcium phosphate, **DOF** = degree of flow, **G′** = storage modulus, **G″** = loss modulus, **HPHM** = high-pH method, **INSOL** = insoluble, **LPHM** = low-pH method, **LT** = loss tangent, **LT_{max}** = loss tangent maximum, **NONRO** = whole milk without reverse osmosis, **pH4.6SN** = pH 4.6 water-soluble nitrogen, **RO** = reverse osmosis, **SAOR** = small amplitude oscillatory rheology.

INTRODUCTION

It is well recognized that total Ca content, pH, and proteolysis are critical parameters that influence the textural and physical properties of cheeses (Lawrence et al., 1987; Lucey and Fox, 1993; Watkinson et al., 2001; Guinee et al., 2002; Joshi et al., 2003; Lucey et al., 2003; Pastorino et al., 2003a,b; Sheehan and Guinee, 2004). It is difficult to independently study these parameters because rate and extent of acid development, pH, and Ca contents of cheese are interrelated; Ca is lost from casein particles as the pH decreases during cheese manufacture (Lucey and Fox, 1993). The texture of Cheddar cheese at high pH (5.4) is elastic but at low pH (e.g., 4.8), cheese is brittle and crumbly (Lawrence et al., 1987; Pastorino et al., 2003b). Watkinson et al. (2001) observed that both fracture strain and fracture stress increased (i.e., cheese became longer and firmer) when the pH of model Cheddar cheeses increased from 5.2 to 6.2. However, pH itself is only one of the factors that can be responsible for differences in cheese texture (others include composition and age).

Lower total Ca levels generally result in softer cheeses and an increase in melt (Lucey and Fox, 1993). Stretch and flow of cheese when heated increase with a reduction in Ca content (Guinee et al., 2002; Joshi et al., 2003; Sheehan and Guinee, 2004). However, total Ca content alone is not the most useful predictor of the physical properties of cheese (Lawrence et al., 1987). Lucey and Fox (1993) suggested that there was a significant amount of Ca in cheese still associated with casein, which is described as insoluble (**INSOL**) Ca. The amount of INSOL Ca in cheese plays a key role in controlling cheese texture as it has a direct influence on casein–casein interactions (Lucey et al., 2003). An example in which the state of Ca in cheese could be more important than pH is in direct-acid Mozzarella cheese making, where a much higher stretching pH (~5.6) is used compared with that used in traditional cultured cheese (~5.2) to have similar stretching properties. This is probably due to different levels of total or

Received April 15, 2005.
Accepted July 15, 2005.
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INSOL Ca in these cheeses (Lucey and Fox, 1993; Lucey et al., 2003).

Recently, Hassan et al. (2004) demonstrated that the INSOL Ca content as a percentage of total Ca of Cheddar cheeses decreased from ~70% after manufacture to ~57% after 3 mo of ripening. It is therefore possible that a reduction in INSOL Ca could be partly responsible for the changes in textural and melting properties of Cheddar cheese during ripening (Lucey et al., 2005). In the study of Hassan et al. (2004), cheese pH did not vary significantly during ripening (~5.2). Pastorino et al. (2003b) demonstrated that decreasing Cheddar cheese pH (postmanufacture) from 5.3 to 5.0 by injection of a 20% glucono-δ-lactone solution increased the solubilization of Ca, which contributed to increased flow rate during melt and decreased hardness. When cheese pH was decreased below 5.0 by further injections of glucono-δ-lactone, the flow rate during melt decreased. Thus, it appears that solubilization of INSOL Ca is a critically important parameter affecting cheese texture but this impact may only be observed in a certain pH range.

The objectives of this study were to determine the effects of altering pH values during cheese manufacture and acid levels in cheese on the INSOL Ca content and the physical properties of Cheddar cheese during ripening. Strategies were used to obtain cheese with low pH values (<5.0) during ripening, as studies on cheeses with higher pH values have been previously reported (Hassan et al., 2004; Lucey et al., 2005).

MATERIALS AND METHODS

Reverse Osmosis of Milk

Whole milk was concentrated to ~14% total solids content using the reverse osmosis (**RO**) unit in the pilot plant of the University of Wisconsin-Madison. This RO unit was fitted with 2 spiral-wound elements that were arranged in parallel and composed of thin film composites. Each element had a membrane area of 7.4 m^2 and a typical NaCl rejection of 99.5% (PTI Advanced Filtration, Oxford, CA). The unit was operated at 4°C and at ~1655 kPa outlet pressure. Reverse osmosis milk concentrate was pasteurized at 73°C for 15 s, and cooled to 4°C. Pasteurized milk at 74°C for 18 s that was not concentrated (**NONRO**) was used to make control Cheddar cheeses.

Cheese Manufacturing

Licensed Wisconsin cheese makers manufactured 2 types of full-fat, milled curd Cheddar cheese, designated high pH method (**HPHM**) and low pH method (**LPHM**) at the University of Wisconsin-Madison Dairy Plant. The HPHM cheese had a higher rennet, drain, and mill pH, whereas LPHM cheese had lower rennet, drain, and mill pH (Table 1). It should be noted the HPHM cheese had lower drain and mill pH values compared with many commercial US Cheddar cheeses. Three separate cheese-making trials were performed using RO milk, and 3 separate trials using NONRO milk were conducted over a period of 18 mo. An outline of the cheese manufacturing conditions used is given in Table 1. A mixed-strain starter culture containing *Lactococcus lactis* ssp. *cremoris* and *Lactococcus lactis* ssp. *lactis* was inoculated into the milk at the rate of 1490 g per vat (226 kg) of milk. Double-strength chymosin (Chymostar; Rhodia, Madison, WI) was added at the rate of 17 mL per 226 kg of milk at 32°C. The coagula were cut at similar firmness, as subjectively determined by cheese makers, using 0.63-cm knives, and the curd was given a 5-min healing time, followed by 10 to 15 min of gentle agitation before heating. The temperature of the curd-whey mixture was raised from 32 to 39°C and curd was continuously stirred at 39°C until the curd reached pH ~6.1 and ~5.8 for HPHM and LPHM cheeses, respectively. Curd slabs were cheddared and milled at pH 5.2 to 5.3 and pH 5.0 to 5.1 for HPHM and LPHM cheeses, respectively. Curd was salted at the rate of 0.72 kg per 226 kg of milk. Curd was packed in 9-kg Wilson-style hoops, pressed for 4 h, and then stored overnight at ambient temperature. Cheeses were packaged and stored at 10°C for 1 wk and then 5°C for the rest of ripening. Two 9-kg blocks of cheese were produced from each vat of cheese.

Compositional Analysis

Milk was analyzed for fat, protein, and casein (Marshall, 1992), total Ca (IDF, 2003) and buffering by the acid-base titration method (Lucey et al., 1993; Hassan et al., 2004). Lactose and lactic acid (both D- and L-lactate) were determined by enzymatic methods (AOAC, 2000; Boehringer Mannheim Biochemicals, Mannheim, Germany). Rennet whey was made from milk on the same cheese-making day and was analyzed for Ca content (IDF, 2003). Compositional analysis on cheese was done after 1 mo for moisture, fat, protein (Marshall, 1992), total Ca (IDF, 2003), and salt by Corning Salt Analyzer (Marshall, 1992). Cheese pH (Marshall, 1992), pH 4.6 soluble nitrogen (**pH4.6SN**; Kuchroo and Fox, 1982), and the INSOL Ca contents of cheese were determined by the cheese juice method as described by Hassan et al. (2004) after 1 d, 1 wk, 2 wk, 3 wk, 1 mo, and 3 mo. Buffering of cheese was determined by acid-base titration (Hassan et al., 2004) and buffering capacity reported as volume of 0.5 *N* HCl required to decrease the pH of cheese dispersions (8 g