DAIRY FOODS

Factors Determining Large-Strain (Fracture) Rheological Properties of Model Processed Cheese

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ABSTRACT

Torsional fracture was used to map changes in large-strain rheological properties of a model processed cheese that contained 20% protein (rennet casein), 27% anhydrous milk fat, 1.5% NaCl, and 1 to 3% Na_2HPO_4. Processing time (10, 20, or 30 min), Na_2HPO_4 (1, 2 or 3%), and pH of the Na_2HPO_4-NaCl solution (5.4, 5.6, or 5.8) were adjusted to alter the extent of casein micelle solubilization and heat-induced protein-protein interactions. A Box-Behnken experimental design was employed, and results were analyzed using response surface regression and ridge analysis techniques. Further investigation employed a single factorial design in which Na_2HPO_4 increased from 1 to 4%.

Model processed cheeses had fracture stress, fracture strain, and fracture modulus (fracture stress/fracture strain) values ranging from 25.1 to 79.7 kPa, 0.66 to 1.88 kPa, and 15.2 to 96.0 kPa, respectively. The properties of fracture modulus and fracture strain formed a master curve, independent of the processing variable. Conditions favoring increased protein solubilization and heat-induced protein-protein interactions increased fracture modulus and decreased fracture strain. This finding coincided with an increase in slope ratio (fracture modulus/modulus at 30% of the fracture strain), indicating a change in fracture mechanism from strain weakening to elastic fracture. These results suggested that large-strain (fracture) rheological properties could be used to characterize the effects of processing variables important to processed cheese quality.

(**Key words:** processed cheese, rheological analysis)

Abbreviation key: AMF = anhydrous milk fat, **DSP** = disodium phosphate, **RC** = rennet casein, $\mathbf{G_f}$ = fracture modulus.

INTRODUCTION

In production of processed cheese, water and emulsifying salts are mixed with natural cheese in the presence of heat and shear. During initial heating and mixing, lipids are emulsified, and proteins are solubilized from the natural cheese network, converting the system to a dispersion composed of emulsified fat and protein (6). Upon cooling, this dispersion forms the composite, processed cheese network (6).

Formation of a proper dispersion is essential to producing a high quality product with desirable texture and melt properties (3, 6). Several factors such as degree of proteolysis in natural cheese; pH of the cheese melt; concentration and type of emulsifying salt; processing time, temperature, and speed of mixing; and temperature after processing have been shown to alter cheese (3, 6, 18).

The impact of the large number of variables affecting cheese quality manifests itself in that it is difficult to have one formula and set processing conditions. Additionally, there is no fundamental method; reflecting protein solubilization, aggregation, and emulsification; to determine when a dispersion of desired properties has been formed. To better control production and to gain an understanding of ingredient functionality, a need exists for understanding the fundamental physical responses that reflect chemical interactions responsible for processed cheese texture and stability.

Texture of processed cheese is one physical response relating to the composite network. Large-strain (fracture) rheological properties of processed cheese reflect the forces and deformations associated with the sensory perception of texture (15). Techniques used to determine fundamental fracture properties are objective and independent of sample size and determination procedure (8, 12). Fundamental techniques restrict sample geometry so that relationships among forces and deformations can be mathematically described. Torsional fracture is a fundamental large-strain technique, which has been used on a variety of food gels (4, 10, 12, 13, 14, 15). It can be applied to processed cheese or any other food gel, if the gel can be ground or cast into a capstan shape (7, 12).

The goal of this research was to map fundamental, large-strain rheological properties associated with vari-

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Received August 24, 1998.
Accepted May 10, 1999.

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ables known to change the texture of processed cheese. Mapping rheological properties required careful control of materials and processing variables. Characteristics of natural cheese used in processed cheese production may vary considerably from batch to batch. With this consideration in mind, a model processed cheese was used that contained powdered rennet casein (**RC**), anhydrous milk fat (**AMF**), sodium chloride, emulsifying salt disodium phosphate (**DSP**), and lactic acid. Rheological properties of this system were manipulated by altering the DSP concentration, pH of the salt solution, and production time.

MATERIALS AND METHODS

Model System Production

All samples contained 20% protein from RC (New Zealand Milk Products, Santa Rosa, CA), 27% AMF (Mid-America Farms, Springfield, MO), 1.5% food-grade NaCl (Morton International, Chicago, IL), and 1 to 4% food-grade DSP (FMC Corp., Lawrence, KS). Percentage of protein in RC was determined from proximate analysis data (N × 6.36, macroKjeldahl) (1). During production, NaCl and DSP were dissolved in 90% of the total deionized water. The pH of the subsequent salt solution was adjusted using 88% lactic acid (Archer Daniels Midland Company, Decatur, IL) and then brought to final weight with deionized water. The AMF and the salt solution were warmed to 85°C while the RC was mixed in a jacketed bowl chopper (Stephan, Columbus, Ohio) for 3 min at 300 rpm. The paddle attachment was used for mixing and 80°C anti-freeze was circulated in the jacketed bowl. The AMF was added and mixed for 3 min more. Salt solution was added, and the sample was mixed at 2000 rpm for an appropriate time. A vacuum was pulled only during the last 3 min of processing in an effort to minimize moisture loss. The cheese was poured from the bowl into a 9½″ × 13″ Pyrex™ (Corning Glass, Corning, NY) dish and wrapped with Saran™ wrap (DowBrands, Indianapolis, IN) and aluminum foil. The cheese was stored overnight at 4°C. Torsional fracture and moisture analyses were conducted within the following 24-h period.

Statistical Design

A Box-Behnken experimental design was employed. This design can be thought of as a particular fraction of the 3^3 factorial with points selected to allow estimation of all first-order and two-factor interaction terms and pure quadratic effects (5). Data were analyzed by response surface regression (RSREG) and ridge analysis techniques. Models were refined using PROC REG in SAS (16).

Three factors at three levels were analyzed. Time was one factor at levels of 10, 20, and 30 min. Because of friction during processing, however, temperature was also affected by time. As a result, the time factor actually connoted a time/temperature factor. The pH of the salt solution at 5.4, 5.6, and 5.8 and DSP concentration at 1.0, 2.0, and 3.0 were also analyzed.

Based on the results of the Box-Behnken experimental design, a simple single factor design was used to consider only DSP concentration. In this experiment, pH of the salt solution and processing time were held constant at 5.4 and 10 min, respectively. Salt solution pH was adjusted with lactic acid, as described previously, and DSP concentration increased from 1.0 to 4.0% in 0.5% increments. Data were analyzed using analysis of variance (PROC REG) in SAS (16).

For both studies, data were transformed to natural logs before performing regression analysis. Ordinary least squares (OLS) is the basis of both types of analyses and assumes constant variance (16). Transforming the data to natural logs minimizes effects of possible nonconstant variance (17).

Moisture and pH Analyses

Samples were prepared for moisture analysis with a blender, using short bursts to prevent sample dehydration from heat of friction during blending. A microwave oven equipped with an internal analytical balance (CEM, Matthews, NC) was used for moisture analysis. Approximately 3.0 g of sample were evenly spread on a glass fiber sample pad (CEM, Matthews, NC). Another sample pad was placed on top. The sample was placed on the analytical balance in the microwave. Moisture analysis was conducted by operating the microwave at 80% power for 5.5 min. An average of three readings was reported.

The pH of the samples was assessed using a solid state probe (Combination Spear-tip pH Electrode; Orion, Boston, MA) at 25°C. An average of three readings was reported.

Torsional Fracture

Cylinders of cheese (19 mm in diameter) were bored by hand from the block of cheese using a cork borer. Cheese cylinders were wrapped in aluminum foil and allowed to warm to 25°C. Cheese cylinders were cut to 28.7 mm in length, and plastic disks used for mounting the sample during grinding and twisting were attached to each end with cyanoacrylate glue (Devcon Corporation, Wood Dale, IL). Samples were ground to a capstan shape with a minimum diameter of 10 mm using a grinding apparatus (Gel Consultants, Raleigh, NC) and

TABLE 1. A brief explanation of large-strain (fracture) rheological properties.

Rheological parameter	Explanation
Fracture stress	Force (Newtons) per unit area (meter^2) at fracture. Also called gel strength.
Fracture strain	Deformation at fracture. It has no units because it is the change in dimensions relative to the initial dimension.
Fracture modulus or $G_{fracture}$	Fracture stress divided by fracture strain. It is the relative rigidity or firmness at fracture.
Slope ratio	Fracture modulus divided by the modulus (stress/strain) at 30% of the fracture strain. It indicates the change in rigidity as strain is increased.

twisted to fracture at 2.5 rpm according to the procedure of Kim et al. (13). A minimum of 5 samples was tested per treatment. A brief explanation of the large-strain (fracture) rheological properties determined is found in Table 1. True shear stress (fracture stress) and true shear strain (fracture strain) at fracture were calculated from the respective torque and angular displacement as described by Diehl et al. (9). Fracture modulus [($\mathbf{G_f}$), fracture stress/fracture strain] and slope ratio ($G_f/G_{\text{at 30\% of fracture strain}}$) were also determined. Slope ratio was calculated as follows: slope ratio = (fracture stress/fracture strain)/(stress at 30% of fracture strain/strain at 30% of fracture strain).

RESULTS AND DISCUSSION

Three-Factor Study

Samples containing 1.0, 2.0, or 3.0% DSP in the Box-Behnken design were formulated to contain 43.8, 44.8, or 45.8% moisture, respectively. Actual moisture values ranged from 40.8 to 47.1%. Moisture was most significantly described by a model containing the linear terms of time and DSP, the quadratic time term, and the DSP-time interaction term (Table 2). These results were due to less water being added to the formulation as the percentage of DSP increased and due to increased moisture evaporation with increased production time.

Model system pH was also a response variable in the analysis. The pH values of the model systems ranged from 5.48 to 5.93 (Table 3). These values were within the range of values associated with processed cheese (pH = 5.4 to 6.0) (6). Model system pH was significantly described by a model containing the linear terms of time, solution pH, and DSP; the quadratic time and DSP terms; and the DSP-time interaction term (Table 2).

Experienced producers of processed cheese report that undesirable crumbly, brittle products result from an action called over-creaming (3), which can be viewed as a network that has proceeded beyond a desired structure because of levels of ingredients or extent of pro-

TABLE 2. Models[1] describing percentage of moisture and pH for the three-factor study.

Factor variables	Response variables Moisture (%)	 Model system pH
Constant	4.46*	1.32*
ln Time	–0.49*	–0.08*
ln Solution pH	…	0.34*
ln Disodium phosphate	0.14*	–0.19*
ln Time^2	0.09*	0.01*
ln Disodium phosphate^2	…	0.05*
ln Disodium phosphate × ln time	–0.08*	0.03*
Sample size	13	13
Adjusted R^2	0.828	0.926
F	0.0008	0.0006

[1] Coefficients were determined by ordinary least squares regression technique.

*$P \leq 0.05$.

TABLE 3. A three-factor study of the mean pH of the model processed cheese.

Time	Salt solution	Disodium phosphate	Cheese pH	Cheese pH
(min)	(pH)	(%)	Average	SD
10	5.4	2.0	5.48	0.03
10	5.6	1.0	5.93	0.01
10	5.6	3.0	5.55	0.01
10	5.8	2.0	5.66	0.01
20	5.4	1.0	5.73	0.01
20	5.4	3.0	5.49	0.01
20	5.6	2.0	5.59	0.00
20	5.6	2.0	5.61	0.01
20	5.6	2.0	5.54	0.01
20	5.8	1.0	5.84	0.02
20	5.8	3.0	5.66	0.01
30	5.4	2.0	5.56	0.01
30	5.6	1.0	5.63	0.01
30	5.6	3.0	5.58	0.01
30	5.8	2.0	5.66	0.00

cessing. These textural observations suggest that an over-creamed network would be reflected in increased fracture stress, decreased fracture strain, and increased G_f (fracture stress/fracture strain) values. As a result, these fracture parameters were investigated.

In the three-factor experiment in which processing time, solution pH, and DSP concentration were altered, the sample that was processed for 10 min, with a salt solution pH of 5.6 and containing 1.0% DSP, was the only sample that did not result in a homogenous product that could be tested using torsional fracture. This finding is consistent with the concept that a minimal amount of protein must be solubilized from the casein micelle/cheese network to emulsify the fat (18, 19).

Maintaining constant amounts of protein and fat in each sample and adjustment of mixing time, solution pH, and percentage of DSP produced processed cheese samples with fracture stress (strength) ranging from 36.8 to 79.8 kPa. The fracture strain (deformability) values ranged from 0.66 to 1.88 (Figure 1), and G_f values (firmness) ranged from 20.7 to 96.0 kPa (Figure 2). Plotting fracture stress versus fracture strain produced a general grouping of all of the samples with a slight trend toward an increase in fracture stress with a decrease in fracture strain (Figure 1). Samples exhibiting fracture stress values in the relatively narrow range of 40 to 50 kPa exhibited a wide range of strain values from 0.76 to 1.82. Similarly, products exhibiting fracture strain values in the narrow range from 1.03 to 1.17 exhibited a wide range of fracture stress values from 36.8 to 79.8 kPa.

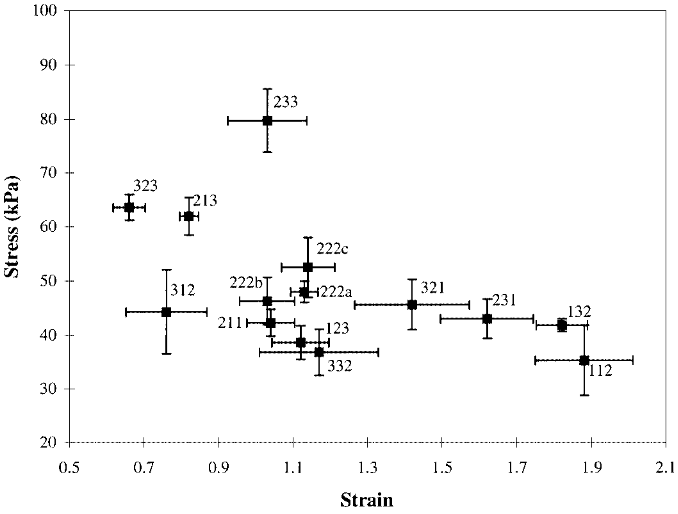

Figure 1. A three-factor study of the relationship between stress at fracture and strain at fracture of the model processed cheese. The first, second, and third digits in the number by each data point represent processing time (10, 20, or 30 min), solution pH (5.4, 5.6, or 5.8), and Na_2HPO_4 concentration (1, 2, or 3%), respectively. The numbers 1, 2, and 3 correspond to the level of the factor. Letters a, b, and c correspond to the replications for 20 min, pH 5.6, and 2% Na_2HPO_4. Error bars show standard deviations.

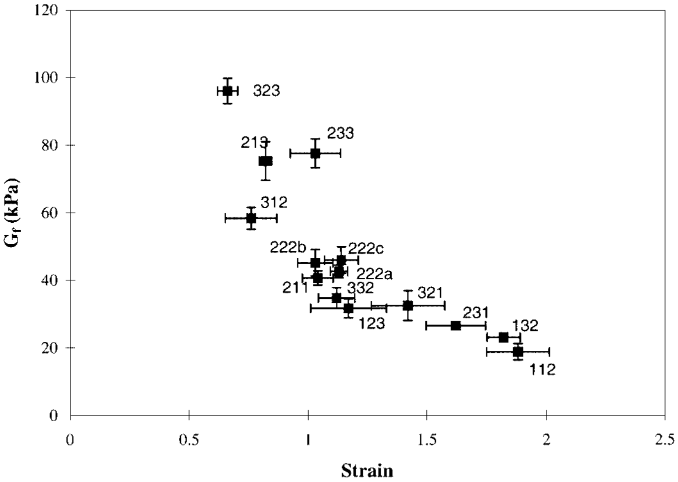

Figure 2. A three-factor study of the relationship between fracture modulus (G_f) and strain at fracture of the model processed cheese. Samples were given three-digit codes for identification purposes. The first, second, and third digits in the code represent processing time (10, 20, or 30 min), solution pH (5.4, 5.6, or 5.8), and Na_2HPO_4 concentration (1.0, 2.0, or 3.0%), respectively. The numbers 1, 2, and 3 correspond to the level of the factor. Letters a, b, and c correspond to the replicate associated with a combination of factors and levels. Error bars show standard deviations.

One possible reason for differences could be a change in fracture mode. In torsional fracture, the stresses of shear, tension, and compression act in equal magnitude, resulting in a constant shape during deformation (i.e., twisting) (12). The sample fractures along a plane at a 45° angle to the long axis if it is weakest in tension and along a plane at 90° if it is weakest in shear (12). Examination of fracture planes revealed all samples fractured at 45° angles, indicating fracture in tension. Therefore, differences were not due to changes in mode of fracture (i.e., switching from a tension fracture at 45° to a shear fracture at 90°).

The fracture modulus (G_f, fracture stress/fracture strain) relates stress to strain and gives an indication of the firmness. Plotting G_f against fracture strain resulted in a curve in which G_f values decreased as fracture strain values increased (Figure 2). Some scatter in G_f was still evident around a strain of 1.0, but a general trend was apparent. Note that the three replications of the center treatment (samples 222 a,b, and c) form a close group centered within the range of fracture strain and fracture modulus values (Figure 2). The three main outlying treatments were samples 323, 213, and 233. If these treatments are not considered, a linear relationship between G_f and strain existed. All three outlying treatments had 3% DSP and were processed for 20 to 30 min. As indicated earier, an over-creamed network was expected to result in low fracture strain and high G_f values, indicating a brittle texture. It appeared that a combination of high DSP level and processing time resulted in a brittle texture, which was different than that observed in the other treatments.

Such a systematic relationship between G_f and strain values indicated that these two fracture parameters did appear to provide a reasonable indication of the changes in network properties from a soft (low G_f), deformable (high fracture strain) network to a firm (high G_f), brittle (low fracture strain) network. Moreover, it demonstrated that various combinations of ingredient and processing variables could be used to create specific rheological properties.

Slope ratio ($G_f/G_{\text{at 30\% of fracture strain}}$) provided an indication of the overall force-deformation process and mechanism of fracture. As discussed previously, the mode of fracture (shear or tension) is determined by the angle of the fracture plane after torsional fracture. The slope ratio determines whether the material weakens, strengthens, or does not change as the material is deformed up to a point of fracture. A slope ratio of less than 1.0 indicates strain weakening where the structure yields prior to fracture. This phenomenon could be due to breaking a subgroup of weaker structural elements or breaking a fraction of one type of structural element. A slope ratio of 1.0 shows a linear relationship between stress and strain. This ratio would be expected for a pure elastic material (i.e., Hookien solid) and is seen in polyacrylamide gels (2, 11). When the slope ratio is greater than 1.0, the material becomes more firm as strain increases, which is called a strain-hardening behavior. Such changes suggest alterations in molecular organization of networks.

There were two general relationships between slope ratio and G_f. One group of model cheeses had G_f values ranging from 20.7 to 32.5 kPa and slope ratios of 0.80 to 0.91 (Figure 3). Model cheeses with these properties had either a short processing time (samples 112, 132, and 123) or the lowest level of DSP (samples 231 and 321) (Figure 3). At G_f values greater than approximately 34.0 kPa, slope ratios approached 1.0 and remained constant (Figure 3). These results indicated that the fracture mechanism, as described by slope ratio, reached a critical level and then remained constant while cheese firmness (G_f values) increased. Thus, a change in molecular organization, or intermolecular forces, of the gel network occurred between G_f values of 20.7 and 32.4 kPa. This change resulted in a shift in the fracture mechanism from strain weakening to that approaching pure elastic behavior.

Simple graphs of G_f versus strain and G_f versus slope ratio illustrate the combined effects of variables on fracture parameters (Figures 2 and 3). However, determining how individual factor variables (processing time, solution pH, and percentage of DSP) affected fracture parameters required statistical analysis. Regression analysis techniques did not determine a model describing the fracture stress response. However, fracture strain was described by the linear components of all three factor variables (Table 4). The only response vari-

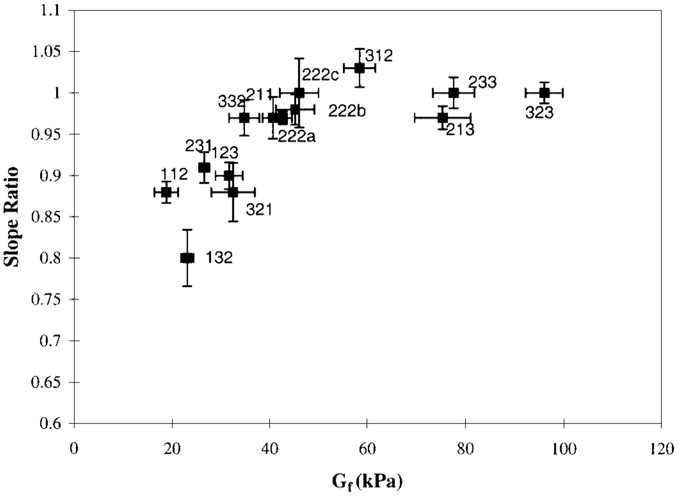

Figure 3. A three-factor study of the relationship between slope ratio and fracture modulus (G_f) of model processed cheese. Samples were given three-digit codes for identification purposes. The first, second, and third digit in the code represent processing time (10, 20, or 30 min), solution pH (5.4, 5.6, or 5.8), and Na_2HPO_4 concentration (1.0, 2.0, or 3.0%), respectively. The numbers 1, 2, and 3 correspond to the level of the factor. Letters a, b, and c correspond to the replicate associated with a combination of factors and levels. Error bars show standard deviations.

TABLE 4. Models[1] describing fracture variables strain, G_f[2], and slope ratio from a three-factor study.

Factor variables	Response variables		
	Strain	G_f	Slope ratio
Constant	–4.48	–166.00	–2.21*
ln Time	–0.55*	17.55*	1.34*
ln Solution pH	3.80*	174.54*	–0.60
ln Disodium phosphate	–0.46*	–14.56*	0.09*
ln Time^2	…	–0.69*	–0.21*
ln Solution pH^2	…	–46.34*	…
ln Disodium phsophate^2	…	0.90*	…
ln Solution pH × ln time	…	–7.77*	…
ln Disodium phosphate × ln time	…	0.64*	…
ln Disodium phosphate × ln solution pH	…	7.23*	…
Sample size	14	14	14
Adjusted R^2	0.796	0.979	0.780
F	0.0002	0.0005	0.001

[1] Coefficients were determined by ordinary least squares regression technique.

[2] G_f = fracture modulus (fracture stress/fracture strain).

*$P \leq 0.05$.

able described by the full model was G_f, which included all linear, quadratic, and secondary interaction terms. Slope ratio was described by all of the linear terms and the quadratic time term. All of the *t* statistics associated with the factors in each model were significant at a $P \leq 0.05$, except for the *t* statistic associated with each intercept and with solution pH in the slope ratio model. Each of the models was highly significant, as described by the *F* significance values. The model describing G_f was highly predictive ($R^2 = 0.979$), and the models describing strain ($R^2 = 0.796$) and slope ratio ($R^2 = 0.780$) were less predictive.

Although mathematical models describe relationships of response variables with factor variables, the complex association is difficult to visualize. Response surface graphs are often used to help conceptualize such relationships. Yet, it is impossible to depict three-factor variables in dynamic interaction with each other on a two-dimensional surface. However, the shape of such a complex response surface can be described mathematically using canonical analysis. Canonical analysis indicated that the predicted response surface was shaped like a saddle. Interpretation of the analysis indicated that solution pH had less effect on the response surface than did DSP concentration or time. This result most probably was related to the limited range of pH investigated (model cheese pH range of 5.48 to 5.93, Table 3) and did not indicate that pH is not important to the texture of processed cheese.

Canonical analysis showed how the factor variables affected the response variable G_f. The saddle shape described by the analysis indicated that a unique combination of factor variables was not necessarily responsible for unique G_f values. This finding coincided with the operational knowledge that various production parameters may be adjusted to obtain similar texture (3).

When a unique optimum within the range of experimental factor variables is not identified, ridge analysis indicates the direction in which further research should be conducted to locate the optimum response (16). Consideration of the estimated ridge of maximum response indicated that further research should focus on lower salt solution pH, greater DSP level, and processing time. The combination of increased processing time and DSP level should have resulted in more protein dispersion and, therefore, increased the amount of protein that could form a gel network. This coincided with known relationships between protein concentration and rheological properties of whey protein gels, where G_f values increased with an increase in protein concentration (10, 14). The trend toward low predicted pH values might also have been due to an increased amount of dispersed protein at lower pH. Alternatively, it could have been due to a change in gel network structure. At a constant protein concentration, G_f values of whey protein gels will vary with the type of gel network structure formed (i.e., a stranded or a particulate structure) (4).

An additional study was conducted to investigate the specific effect of DSP on rheological properties. This study necessitated choosing a solution pH and processing time in which a wide range of rheological properties could be achieved by altering DSP concentration. The lowest G_f value and highest fracture strain values were exhibited by the model processed cheese sample in the three-factor study that had a solution pH of 5.4 and had been processed for 10 min. Ridge analysis indicated that a large range of G_f values could be generated using a low processing time, low pH, and a range of DSP concentrations. As a result a constant processing time of 10 min and solution pH of 5.4, and a range of 1.0 to 4.0% DSP, increasing in 0.5% increments, were chosen.

Single-Factor Study

In this study, 1.0% DSP was the only concentration that did not result in a homogenous product that could be tested by torsion. This finding coincided with the 1% DSP, pH 5.6, 10-min processing treatment from the first study, which did not form a homogeneous, stable gel. All model cheese moisture values, except for the sample containing 1.0% DSP, were 0.5 to 4.0% lower than formulated values. Lower values were most likely due to evaporation during processing. Because all treatments underwent the same time-temperature processing, the greater amount of evaporation might have been associated with the level of phosphate; however, this observation requires further investigation.

The pH of the model processed cheese in the single-factor study ranged from 5.87 to 5.50. The pH decreased from 5.87 to 5.43 as percentage of DSP increased from 1.0 to 2.0% (Figure 4). The pH remained constant at approximately 5.5 for all processed cheese samples containing between 2.0 and 4.0% DSP, except for the sample containing 3.5% DSP that exhibited a slight dip in pH to 5.43.

Analogs containing calcium caseinate also exhibit the most significant change in pH between emulsifying salt concentrations of 0.0 to 1.0% (7). However, pH increased from 6.1 to 6.4, 6.8, 6.9, or 7.0 for analogs containing DSP, sodium citrate, sodium tripolyphosphate, or tetrasodium pyrophosphate, respectively. The pH decreased from 6.1 to 5.9 for analogs containing sodium polyphosphate. Differences in pH trends of analogs made from calcium caseinate (7) and the model processed cheese containing RC in the present study are

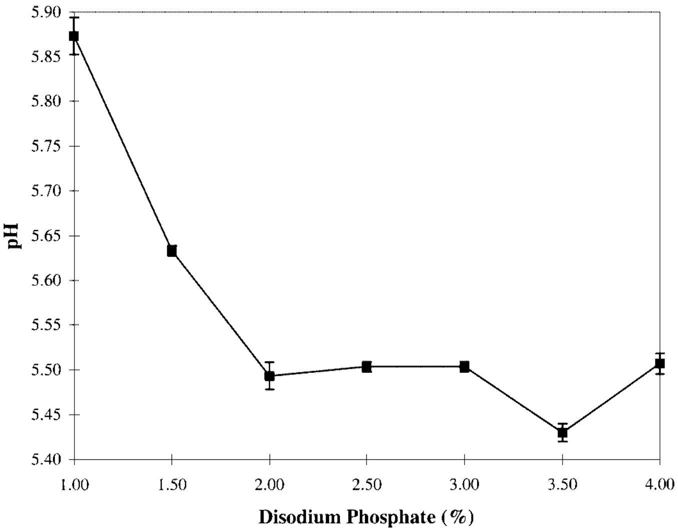

Figure 4. A single-factor study of the effect of disodium phosphate concentration on the model processed cheese pH. Error bars show standard deviations.

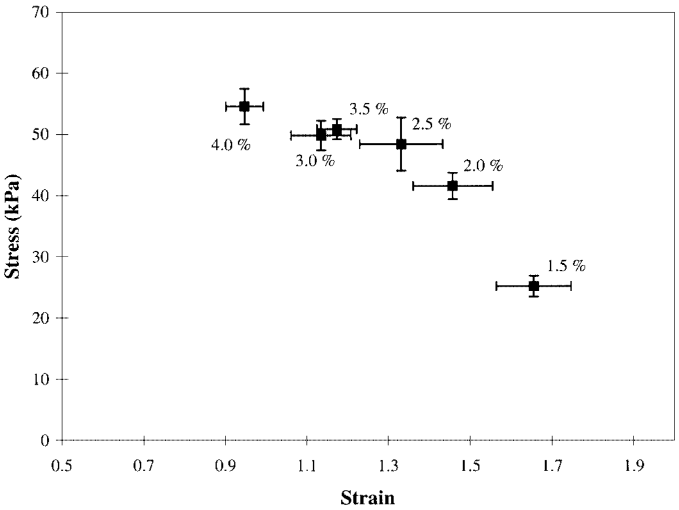

Figure 5. A single-factor study of the relationship between stress at fracture and strain at fracture of the model processed cheese containing 1.5 to 4.0% Na_2HPO_4. Error bars show standard deviations.

most probably due to differences between calcium caseinate and RC.

The relationship among each response variable, including stress, and the factor variables were significantly described by mathematical models (Table 5). Each of the models, except for the one describing strain, depicted a quadratic relationship between the response variables and DSP concentration. Similar to the model describing strain from the three-factor study, the strain association with DSP concentration was linear. The *F* significance values indicated that all of the models were significant.

The model significance and high adjusted coefficient of determination values associated with stress at fracture did not correspond with results from the three-factor experiment. In the three-factor study, stress did not correlate with any of the factor variables, including DSP. This inconsistency between the three-factor and one-factor studies implied that although stress was correlated with respect to percentage of DSP, when other variables are involved, it cannot be described by a simple model. As a result, although stress provided an indication of reactions associated with DSP concentration, it was not the best indicator in the more complex system. Conversely, G_f and fracture strain exhibited significant, predictable relationships in the three-factor and the single-factor studies. The significance and highly predictive nature of the model describing response of G_f in conjunction with its relationship with fracture strain strengthened the validity of using G_f and fracture strain to describe the large-strain rheological properties of processed cheese.

As in the three-factor study, rheological properties varied greatly. Increased DSP level caused fracture strain to decrease from 1.66 to 0.95, whereas fracture stress increased from 25.1 to 54.5 kPa (Figure 5). The overall trend of an increase in fracture stress with a decrease in fracture strain was consistent with changes in whey protein gels when protein concentration is increased (10) or when the gel network structure is changed (4). The amount of protein forming the gel network would be increased by the degree of protein solubilization from RC and the moisture loss, which would explain the results. It is also possible that the type of network structure is changing, also contributing to the rheological changes. Maximum fracture stress was achieved at 2.5% DSP and additional DSP caused a decrease in fracture strain (Figure 5). Model processed cheese with 3 and 4% DSP contained 43 and 40% mois-

TABLE 5. Models[1] describing fracture variables stress, strain, G_f[2], and slope ratio from a single-factor study.

Factor variables	Response Variables Stress	Strain	G_f	Slope ratio
Constant	2.37*	0.73*	1.77*	–0.10*
ln Disodium phosphate	2.59*	–0.52*	2.78*	–0.16*
ln Disodium phosphate^2	–1.05*	…	–0.86*	0.16*
Sample size	6	6	6	6
Adjusted R^2	0.9505	0.9163	0.9491	0.9075
F	0.0051	0.0017	0.0053	0.0131

[1] Coefficients were determined by ordinary least squares regression technique.

[2] G_f = fracture modulus (fracture stress/fracture strain).

*$P \le 0.05$.

ture, respectively. The difference in fracture stress, 5 kPa or 10%, was most likely due to factors other than protein concentration, because fracture stress is proportional to protein concentration to the second or third power (fracture stress ∝ $[\text{protein}]^{2 \text{ to } 3}$) (10).

Fracture strain exhibited a linear relationship with G_f such that, as G_f increased from 15.2 to 57.7 kPa, strain decreased from 1.66 to 0.95 (Figure 6). Increasing G_f, and concomitant decreasing strain values corresponded to the previous combined effects of DSP concentration and processing time. The continuity of the trends in fracture values between the three-factor and single-factor studies indicated that increasing DSP concentration incrementally changed network properties from a weak (low fracture stress), deformable (high fracture strain) texture to a firm (high G_f), brittle texture. This result could have been due to an increase in solubilized protein, an increase in moisture loss, a change in gel network structure, or a combination of all of these.

As discussed previously, plotting slope ratio against G_f provided an indication of how the fracture mechanism changes with gel firmness. The model cheeses containing 1.5 to 2.0% DSP had slope ratios in the range of 0.85 to 0.89, indicating a strain weakening type of fracture (Figure 7). While slope ratio did not change, the G_f values increased from 15.2 to 28.5 kPa when DSP concentration was increased from 1.5 to 2.0% (Figure 7). A lack of change in slope ratio with an increase in G_f indicated that, although the overall firmness increased, the basic molecular interactions, network structure responsible for fracture, or both did not change. For samples containing 2.0 to 4.0% DSP, slope ratio increased linearly from 0.87 to 0.99 as G_f increased from 28.5 to 57.7 kPa.

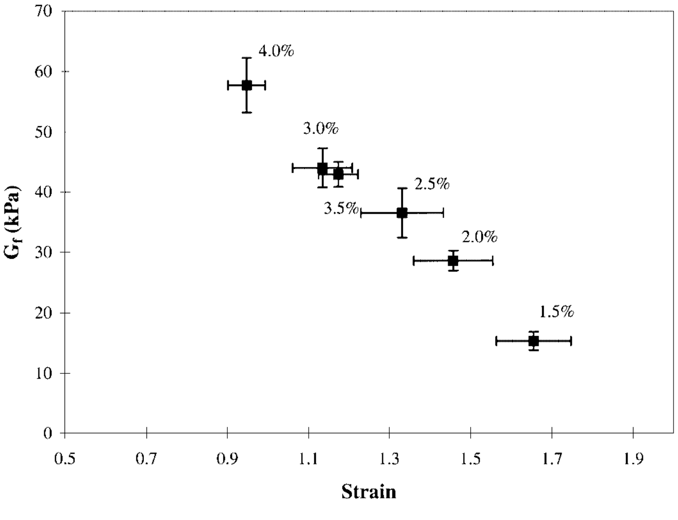

Figure 6. A single-factor study of the relationship between fracture modulus (G_f) and strain at fracture of the model processed cheese containing 1.5 to 4.0% Na_2HPO_4. Error bars show standard deviations.

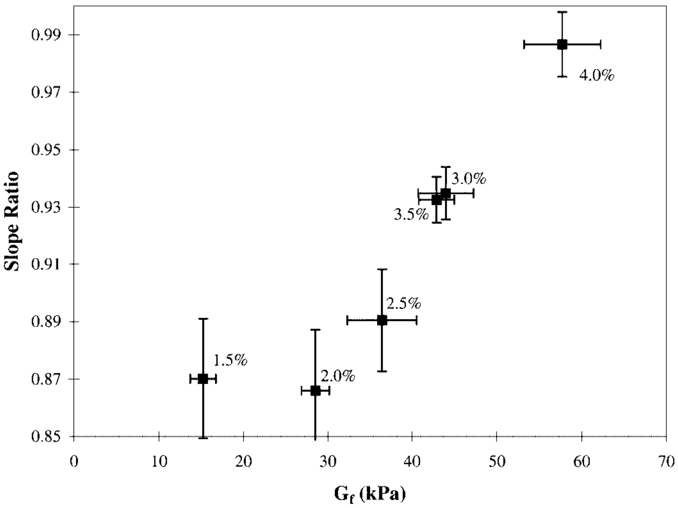

Figure 7. A single-factor study of the relationship between slope ratio and fracture modulus (G_f) of the model processed cheese containing 1.5 to 4.0% Na_2HPO_4. Error bars show standard deviations.

A distinct shift in slope ratio in samples containing between 2.0 and 4.0% DSP indicated a change in the mechanism of fracture of the samples from strain weakening to almost pure elastic fracture. Thus, the inference may be made that a shift in molecular interactions, network structure responsible for fracture, or both occurred in samples containing 2.0 to 4.0% DSP. The model cheese made with 1.5% DSP was different than that of the others in having the lowest fracture stress (Figure 5) and not following the linear trend in slope ratio versus G_f (Figure 7). Model cheeses made with 1 to 1.5% DSP had rheological properties that were distinctively different from the rest, suggesting that a critical amount of DSP lay between 2.0 and 2.5% for this formulation and process. This value would vary with formulation and processing conditions.

CONCLUSION

Results from large-strain (fracture) rheological analysis of the three- and single- factor studies showed that fracture parameters provided indicators of textural changes associated with ingredient and processing variables. Moreover, the relationship between G_f and fracture strain was shown to be the best indicator. Increased G_f and decreased fracture strain values indicated the transition from a soft, deformable texture to a firm, brittle one. In addition, the slope ratio indicated that the fracture mechanism progressed from strain weakening to almost pure elastic as the texture became firm and brittle.

The G_f and strain responses were significantly described by mathematical models containing factor variables. These models indicated that generally decreasing salt solution pH and increasing DSP concentration and processing time moved the texture more toward firm and brittle.

ACKNOWLEDGMENTS

Paper Number 98-32 of the Journal Series of the North Carolina Agricultural Service, Raleigh 27695-7643. The authors would like to acknowledge New Zealand Milk Products, Inc. (Santa Rosa, CA) for contributing the rennet casein used in the model system. This project was supported in part by the Southeast Dairy Foods Research Center (Raleigh, NC).

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