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A Response Surface Study of Extruded Corn Starch/Skim Milk Powder Blends

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A RESPONSE SURFACE STUDY OF EXTRUDED CORN STARCH-SKIM MILK POWDER BLENDS

by

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Sachin Singh

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ABSTRACT

A Response Surface Study of Extruded
Corn Starch/Skim Milk Powder Blends

by

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Skim milk was ultrafiltered to three lactose/protein ratios and spray dried. The skim milk powder was extruded with pearled corn starch at different moisture contents, protein contents, lactose/protein ratios, and feed rates (control variables). Response surface methodology and a central composite in cube experimental design were used. This design required 30 experimental runs with 16 factorial points, 8 axial points, and 6 center points for replication. The physical and functional properties evaluated were expansion ratio, product temperature, bulk density, color, shear stress, viscosity, and water absorption index (response variables). Scanning electron microscopy was done to evaluate the microstructural attributes of the extrudates.

A quadratic model was used to express the response variables in terms of the control variables. Response

surfaces were generated by assigning center point values to 2 of the 4 control variables and then solving the fitted equations as a quadratic in the remaining 2 control variables.

An increase in moisture content decreased expansion ratio, product temperature, color, and water absorption index, and increased bulk density, shear stress, and viscosity. An increase in protein content decreased product temperature, shear stress, viscosity, and water absorption index, increased color, and had no effect on expansion ratio and bulk density. An increase in lactose/protein ratio decreased product temperature, viscosity, and water absorption index, and had no effect on expansion ratio, bulk density, color, and shear stress. Feed rate did not have significant individual effect on any response variable. Expansion ratio had a negative correlation with bulk density ($r = -0.61$) and shear stress ($r = -0.62$) and a positive correlation with product temperature ($r = 0.52$). Bulk density and shear stress were positively correlated ($r = 0.69$), and product temperature and water absorption index were positively correlated too ($r = 0.81$). Expansion ratio, bulk density, color, and shear stress were chosen to determine a combined set of extrusion conditions most likely to produce an extrudate with properties suitable for a snack-type product.

(122 pages)

INTRODUCTION

Extrusion processing has found widespread application in the food industry for restructuring proteinaceous and starchy materials into a variety of fabricated, cooked, and shaped products of varying texture. Cooking extruders are used to produce breakfast cereals, noodles and pasta, biscuits, snack foods, confectionery, chewing gum, modified starches, dried soups, pet foods, crackers, bread crumbs, dried beverage powders, and texturized proteins.

Consumers are becoming increasingly aware of the nutritional importance of proteins, vitamins, and minerals, and are avoiding foods containing excess calories and saturated fats. Therefore, new products may require the addition of selected sources of proteins such as protein-rich skim milk powder. However, foods without acceptable appearance, flavor, or texture will not be successful, regardless of nutritional quality. The challenge, therefore, lies in fabricating foods that combine nutrition, desirable sensory attributes, and convenience.

The use of some dairy commodities in extruded products has been shown to affect desirable qualities such as flavor, texture, browning, and nutritional content. These dairy products include whole milk powder, casein powder, and whey proteins. However, research designed specifically to incorporate skim milk powder in extruded food products has been lacking.

LITERATURE REVIEW

Food Extruder

A food extruder can be considered as a high temperature, short-time bioreactor that transforms a variety of raw ingredients into modified intermediate and finished products (Harper, 1989). The simplest form of an extruder consists of a flighted screw rotating in a tightly fitting cylindrical barrel. At one end it has an entry port for raw ingredients, which are usually preground and blended. In some extruders, the ingredients are partially heated and hydrated in a preconditioning chamber under atmospheric or high pressure (Harper, 1981). The flights on the screw push the food mass forward, forming a continuous, viscous, plasticized mass. As the food mass is conveyed along the extruder by the action of the screw, it is subjected to large shear forces. These shear forces tend to align the molecules in the direction of flow, thereby exposing bonding sites, which leads to crosslinking and a reformed expandable structure (Harper, 1986).

Heat is generated and added to the food mass by three different methods: (1) shaft friction resulting from the shear forces acting on the food mass, (2) direct steam injection into the food mass, and (3) heat transfer from electrical heaters or steam jackets surrounding the barrel (Chilton's Food Engineering, 1977). A restriction at the discharge end of the barrel in the form of an opening called

a die causes the pressure within the barrel to increase as the food mass moves through the extruder. The high pressure prevents the boiling or flashing of water into steam as the pressure exceeds the vapor pressure of water at the extrusion temperature. As the product exits through the die, the sudden drop in pressure allows the water to vaporize, causing the product to expand (Chilton's Food Engineering, 1977). Due to moisture loss, the product is cooled and solidified, retaining its expanded shape.

Types of Extruders

There are essentially two types of extruders -- single screw and twin screw. There are various types of twin screw extruders, classified on the basis of the relative direction of rotation of the screws, counter or co-rotating and the degree of screw intermeshing. The application of single screw extruders is limited to feeds with moderate moisture content because of relatively poor mixing, kneading, and transport properties (Noguchi, 1989). Also, a single screw extruder is relatively ineffective in transferring heat from the barrel jackets because convective heat transfer is limited due to poor mixing within the screw channel. Twin screw extruders are more expensive for a given operation than comparable single screw extruders, but they offer greater advantages because of the characteristics of the screw. The greater conveying angle and self-wiping feature of the screw enable it to handle a wide variety of

ingredients (Harper, 1989). Improved processing also takes place because of the uniformity of shear rate across the channel depth and increased mixing in the screw channel. Moreover, twin screw extruders have less interaction of process variables as compared to single screw extruders, making them easier to operate and control.

Extrusion Processing

During the extrusion process the food ingredients are subjected to a number of unit operations like mixing, shearing, cooking, forming, texturizing, puffing, and drying in a single, energy efficient, rapid, and continuous process (Harper, 1981). Cooking extruders were initially developed as an economical method of gelatinizing starches. They have been modified and improved over the years to process a wide range of ingredients of plant, microbial, and animal origin (Kinsella, 1978; Harper, 1979, 1981; Lai et al., 1985a; Aguilera and Stanley, 1986; Mandigo, 1986; Lee, 1986).

The popularity of extrusion cooking stems from the flexibility it offers to produce food commodities of desired nutritional balance in an appealing form. The process is energy efficient, has low production cost, handles a variety of raw ingredients and processing conditions, offers better ingredient utilization with no or very little effluent, and is more versatile than other thermal processing methods.

Extrusion Processing of Proteins

The most important point about the protein extrusion process is that there are wide differences among materials. The central factor that determines behavior of proteins is the extent to which a continuous molten phase can be formed within the extruder. This phase is associated with material that is soluble in solvents that break disulfide bonds. For many protein systems a significant amount of material formed is not soluble in these solvents. It is not known what kind of linkages exist in this material.

Proteins are susceptible to both conformational changes and chemical reactions during extrusion. The conditions attained within an extruder are sufficient to denature proteins. This fact is used to denature enzymes which are responsible for poor storage qualities and poor taste of foods made from proteinaceous vegetable material (Chilton's, 1977). The enzymes urease and lipoxidase in soybean and lipase in rice bran are easily denatured within an extruder. Extrusion cooking also inactivates trypsin inhibitor.

The cost of extruding pure proteins is prohibitive. Most commercial extrudates contain substantial amounts of nonprotein materials. Presence of excess lipid in a protein system causes slippage under high shear conditions that normally occur during extrusion. This prevents successful processing, particularly in a single screw extruder (Harper, 1979, 1981). Lipid-protein interactions are important for

structure stabilization. The lipid moiety interacts with proteins up to a saturation limit determined by the amount of hydrophobic sites present in the protein fraction. Extrusion may increase this limit as a result of the exposure of more sites following disruption of aggregates partly maintained by intermolecular hydrophobic interactions or by the unfolding of native proteins.

Proteins extruded in carbohydrate-based systems undergo Maillard reactions in the presence of reducing sugars. This can lower the nutritional quality of the protein by reducing the lysine availability (Bjorck et al., 1984). Such interactions may also affect extrusion behavior (Ledward and Mitchell, 1989).

Modifications of the properties of milk proteins by extrusion have been attempted by a few researchers. Yagi et al. (1986) described a process for preparing milk proteins with resistance to heat, acids, and bases. Downey and Burgess (1979) described an extrusion method of production and modification of aqueous solubility of edible protein fibers composed of casein and carrageenan. The reduction of solubility enabled the use of casein fibers as meat extenders. Guiraud and Arnaud (1977) reported a method for producing fibrous protein from milk solids by extrusion into a hot aqueous solution containing $CaCl_2$.

Casein

Several studies have been reported on extrusion processing of casein. The majority of the studies has been on using an extruder as a reactor for manufacture of caseinate (Weidmann and Millauer, 1985; Tossavainen et al., 1986; Boulle, 1987; Wagner and Sillard, 1989; Borgstrom, 1990) and acid casein (Fichtali and van de Voort, 1990; Fichtali et al., 1990). The coarse product obtained by neutralization of acid casein in a twin screw extruder was reported to be less porous, more dense, and more dispersible than spray-dried caseinate (Tossavainen et al., 1986). Szpendowski et al. (1983) reported process parameters for extrusion of calcium caseinate obtained by enzymatic coagulation of pasteurized skim milk.

Thermoplastic extrusion has been utilized to develop new products from casein. Zuber et al. (1987) used a cooking extruder to continuously mix, melt, emulsify, and gel cheese mix constituents, and obtained processed cheeses or cheese analogues of varying textural attributes (spreadable or sliceable). Bisson et al. (1986, 1988) described preparation of a casein-based puff product using a cooking extruder. van de Voort et al. (1984), studying co-extrusion of casein and wheat flour, reported production of protein-fortified (casein at 10-30% of total solids) extruded products without altering the characteristic bland flavor and crisp texture of extruded wheat flour. Schroeder

(1982a, b) described extrusion processes for making protein-rich crisp and foamed proteinaceous snack products from mixtures made out of sodium caseinate or casein, soy protein, gluten, and cereal protein. The crisp product was reported to retain crispness and chewability on storage and the foamed product had a brittle structure. Addition of casein during manufacture of vegetable meat analogues by extrusion cooking has been claimed to improve consistency and color of the product (Danowski et al., 1979).

Whey Proteins

Studies have also been carried out on extrusion processing of whey proteins. Kim and Maga (1987) reported that blends of extruded whey protein concentrates and starchy flours had a lower expansion ratio and water absorption index than the starchy flours alone. A sensory panel preferred rice extruded with whey protein concentrate to rice without it. Queguiner et al. (1989) inoculated whey protein isolate with Streptococcus thermophilus and thermally processed it in a twin screw extruder at a low moisture content. They reported significant reduction in the microbial load without modification of the solubility or the gelling properties of beta-lactoglobulin or alpha-lactalbumin. Aguilera and Kosikowski (1978) used partially demineralized, delactosed whey product (containing 36% protein and 56% lactose) as a supplement during extrusion processing of a mixture of corn meal, soy flour, and soy

isolate. They observed that the whey product reduced water absorption capacity but increased water and nitrogen solubility. Martinez-Serna and Villota (1991) studied the extrusion performance of native and chemically modified whey proteins. They observed that the free amino groups of the whey proteins had a significant effect on the rheological properties of the extrudates. Queguiner et al. (1991) attempted the preparation of fat analogues by extrusion cooking of whey protein isolate (WPI) and reported that the most adequate fat analogues were obtained when WPI was extruded at pH 3.9, with barrel temperatures of 90 and 100°C. Cuddy and Zall (1982) extruded corn flour with lipid-dried acid whey, acid whey, and sweet whey and obtained products with markedly different organoleptic characteristics.

Skim Milk Powder

Skim milk powder (SMP) has also received some attention in extrusion research. Barraquio et al. (1988) studied the acid coagulation of skim milk powder in a twin screw extruder (barrel temperature T_b, 50-94°C; moisture content, 25-35%). Jones (1989) made a sweet extrudate with adequate browning and flavor using 9% skim milk powder, 13% fat, glucose syrup, icing sugar, and, in some cases, 0.3% lecithin. Tuohi et al. (1979) compared the texture of meat extenders from extruded skim milk proteins with those from

texturized vegetable protein (TVP). The extrudates (containing 58% protein and 11% moisture) displayed poorer texture characteristics after rehydration than TVP or cooked minced meat. Barraquio and van de Voort (1991) devised a method for production of sodium caseinate with the requisite properties normally associated with commercial caseinate by extrusion processing of skim milk powder. They reported that production of either acid casein or sodium caseinate by such a process would involve substantially lower costs than traditional methods. Significant research designed specifically to utilize skim milk powder in extruded food products was, however, found to be lacking.

Animal Proteins

Literature on thermoplastic extrusion of animal protein is sparse. Some studies have been conducted on extrusion of fish proteins. In an attempt to better utilize mechanically deboned minced fish, Murray and Stanley (1980) studied co-extrusion of fish and soy proteins. Coextrudate texture depended strongly upon the ratios of protein to water and of fish to vegetable protein. A product with improved texture and higher level of essential amino acids was obtained. It was concluded that coextrudate with specific textural and nutritional characteristics could be prepared by controlling composition and process variables. Maga and Reddy (1985), investigating coextrusion of carp and rice flour, observed that the extrudate could be stored for six months at room

temperature without development of off odor. An extruded snack food containing 35% carp was found to be acceptable by a sensory panel.

Nicklason and Pigott (1989) attempted to gain information for predicting the effects of heat and flow rate during extrusion processing of surimi-based products. Improvement in cooked gel strength was obtained by heating the product during flow before cooking. A texture similar to red meat was obtained by coextruding a blend of sardine and defatted soy flour (Noguchi, 1989). Bhattacharya et al. (1988, 1990) investigated extrusion of minced fish blended with wheat flour and evaluated the effect of process variables on nutritional quality and microstructure of the extrudate. Fiber formation was enhanced by an increase in temperature. Higher length/diameter (L/D) ratios and increase in fish content of the blend also resulted in the formation of organized fibrous microstructure.

Vegetable Proteins

Extrusion processing has been used to texturize plant proteins by imparting a fibrous structure to amorphous plant proteins. Globular soy protein has been successfully texturized using a food extruder. Textured soy protein is widely used as an extender of red meat products (Maurice et al., 1976). Many studies on protein texturization using an extruder have been done (Van Zuilichem et al., 1977; Stanley and de Man, 1978; Clark, 1978; Kinsella, 1978; Harper, 1981).

Cumming et al. (1972), Aguilera and Kosikowski (1976), and Maurice and Stanley (1978) studied the effect of process variables on certain textural properties of extruded products from soybean meal. Taranto et al. (1978), Aguilera et al. (1980), and Philips et al. (1984) carried out studies on peanut protein isolate, cotton seed, and cowpea meal, respectively. Holay and Harper (1982) in their study on plant protein texturization concluded that moisture content and shear stress affected the density of the product. Gujska and Khan (1991) extruded blends of high protein fractions of navy and pinto beans with corn meal. They reported that extruded blends had a lower expansion index and oil absorption capacity and higher protein density and oil emulsification capacity as compared to extruded corn. Bhattacharya et al. (1986) extruded blends of corn gluten meal and soy protein concentrate and reported models for functional properties of extrudates as a function of moisture content and shear rate. Skierkowski et al. (1990) reported instrumental and sensory evaluation of textural properties of bean extrudates made from formulations modified by addition of high protein bean fraction. Park et al. (1993) studied the effect of process variables on the physical properties of extrudates of defatted soy flour-amylose corn starch/raw beef blends. They observed that soy flour or total protein in the feed was an important determinant of extrudate characteristics.

Large decreases in soy protein solubility have been reported following extrusion (Stanley, 1989). A major factor thought to contribute to the decline in protein solubility is aggregation due to enhanced hydrophobic bonding between neighboring subunits following thermal denaturation. Rearrangements in disulfide crosslinking patterns may also be important. Neumann et al. (1984), studying the extrusion of blends of corn gluten meal and soy flour, suggested the formation of intermolecular disulfide bonds. However, Cumming et al. (1973) and Burgess and Stanley (1976) in their studies showed that disulfide bonds had no role in extrusion texturization. Burgess and Stanley (1976) suggested intermolecular amide bonds as being responsible for the texture formation. Hager (1984) extruded defatted soy meal at 135°C and found evidence of intermolecular disulfide bonding but found no evidence of intermolecular peptide bond formation.

Proteins from sources other than soy are thought to behave similarly. Racicot et al. (1981) showed that the solubility of cornmeal protein in ethanol and alkali significantly decreased after extrusion. Wen et al. (1990) observed a small decline in the solubility of cornmeal protein in dimethyl sulfoxide following extrusion.

Extrusion Processing of Starch

The major effects on starch during extrusion processing

are the disruption of the crystalline regions in the granule followed by a loss of granule integrity and, in the case of cereal starches, the formation of amylose-lipid complexes. It is well established that starch fractions of wheat (Davidson et al., 1984) and corn (Chinnaswamy and Hanna, 1990) undergo a certain degree of fragmentation during extrusion processing. The effects of extrusion on starch structure have been examined by a number of researchers, using both direct and indirect methods. Indirect measurements consistent with starch fragmentation include decreased viscosity (Owusu-Ansah et al., 1983; Diosady et al., 1985), decreased water absorption index (Mercier and Feillet, 1975; Owusu-Ansah et al., 1983), and increased degree of gelatinization (Gomez and Aguilera, 1984). Direct measurements documenting starch fragmentation in wheat and cornstarch by gel filtration chromatography have been reported by a number of researchers (Colonna et al., 1984; Davidson et al., 1984; Chinnaswamy and Hanna, 1990).

Food extruders were initially used as an economical method of gelatinizing starches. Starch granules are gelatinized by means of water absorption inside the extruder. The cooking conditions can be controlled to obtain a specific degree of starch gelatinization or dextrinization. In general, high moisture/low temperature conditions result in mild gelatinization while low moisture/high temperature conditions result in a high degree

of gelatinization and some dextrinization (Chilton's, 1977).

Starch, gelatinized in the presence of fat, forms a loose complex with fat. The starch-fat complex is easily separated by acid hydrolysis, and the fat is still nutritionally available. The formation of starch-fat complex is desirable because it makes the fat less noticeable in the product. Proteins extruded in the presence of starch and certain sugars can undergo Maillard reactions (Chilton's, 1977). For browning to occur, extrusion should be carried out under low moisture/high temperature conditions. Strong water-binding agents like salts and sugars bind water in competition with starch (Fennema, 1985). The presence of such substances reduces the extent of starch gelatinization during extrusion cooking.

Extruders are an efficient means of preparation of pregelatinized starches (Millauer and Weidmann, 1984). Extrudates with markedly different paste qualities have been produced by extrusion of starch under varying conditions (Anderson et al., 1969; Lawton et al., 1972; Meuser et al., 1984). Starch extrudates have a honeycomb cellular structure like popcorn (Faubion and Hoseney, 1982a, b; Hoseney et al., 1983; Lai et al., 1985a, b). Both size and number of cells in starch extrudates are affected by (1) moisture content, (2) presence of proteins, (3) lipids, and (4) yeast cellular components (Faubion and Hoseney, 1982a,

b; Lai et al., 1985a, b). Extruder conditions and presence of additives will not alter the number of starch hilum. The changes in the ultrastructure of the extrudate, therefore, tend to suggest that the starch hilum may not be the nucleation site in extrusion.

Expansion of an extruded product is dependent essentially on its starch content. Expansion of starchy materials during extrusion cooking is related to the original native form of the starch and to the extent of starch damage. Untangling, aligning, and/or partially cleaving the starch molecule diminishes its ability to store energy during deformation and flow through the die. Mercier and Feillet (1975), Chinnaswamy and Hanna (1988), and Fornal et al. (1987) reported that starch type seemed to be a significant determinator of extrudate expansion. Chinnaswamy and Hanna (1988) observed that sectional expansion differed from amylose and amylopectin and that maximum sectional expansion of each starch type occurred at different temperatures. It was also observed that maximum expansion occurred at approximately the same moisture content for different starch types, thereby indicating that moisture did not affect expansion as much as temperature and starch type. Fornal et al. (1987) suggested that expansion is also dependent on protein content and not only on starch content. Faubion et al. (1982) and Peri et al. (1983) also observed that expansion decreased with increasing protein

content.

Effect Of Process Variables on

Protein Extrudate Characteristics

The physical, chemical, and functional characteristics of the extrudate are determined by the operating variables of the process. Several studies have been carried out to determine the effects of process variables on protein extrudate characteristics. These include effects of temperature, pH, feed composition (especially moisture and protein content), screw speed, and extruder length/diameter (L/D) ratio on texture, bulk density, expansion, solubility, water absorption capacity, color, rehydration, and microstructure of extrudates.

Feed Composition

Feed composition has a striking effect on extrudate characteristics as has been discussed extensively in the literature review section. The extrudates from different protein sources are expected to have significantly different textural attributes because of differences in protein structure and composition. The texture is affected by the presence of lipids, starches, and reducing sugars. The presence of lipids results in a softer texture while the effect of starches and reducing sugars depends upon gelatinizing characteristics and interactions with protein (Kinsella, 1978).

Product characteristics are affected by moisture and protein concentration in the feed mix. The influence of moisture content is largely governed by the prevailing temperature and pressure during the actual extrusion process (Kinsella, 1978). Holay and Harper (1982) observed reduced density with increasing moisture content for soy protein. But with wheat gluten and a bran-like fraction, density was found to increase with increasing moisture content (Lawton et al., 1985). Bhattacharya et al. (1986) reported that higher product moisture content resulted in a denser product with decreased water-holding capacity and increased shear strength. Pham and Del Rosario (1984) reported reduced solubility and better texture with higher protein content. Rhee et al. (1981) observed smoother surface morphology, lower bulk density, and higher water retention and holding capacity with increasing protein content. Badrie and Mellowes (1992) reported lower expansion and water solubility and higher bulk density and water absorption with increased protein content.

Temperature

Temperature strongly influences the extrudate microstructure. Maurice et al. (1976) reported an increase in aligned fibers and development of spongy texture of soy protein extrudates at higher process temperatures. A sharp decrease in density of wet extruded soy bean meal and an increase in water absorption capacity with increasing

extrusion temperature were observed (Cumming et al., 1972; Aguilera and Kosikowski, 1976; Lawton et al., 1985). Similar results were reported by Pham and Del Rosario (1984) for defatted soybean and air classified mung bean.

Temperature, moisture content, and retention time proved to be significant parameters for full fat soy flour (Mustakes et al., 1970). Li Sui Fong (1980) studied effects of extrusion cooking on the availability of lysine and concluded no loss of protein nutritional value as a result of extrusion. The loss of lysine was a function of extrusion temperature. Aguilera and Kosikowski (1976) used response surface analysis to optimize process temperature, feed moisture content, and screw speed for production of soybean-extruded product. Superior rehydration characteristics were observed at high processing temperature (above 140°C) and moisture content below 30%. Temperature and protein content were found to be the most important parameters controlling the texture of extruded soy protein (Maurice and Stanley, 1978). Lower sample stress and a higher sensory score for crispness with increasing process temperature were observed for bean extrudates produced from formulations modified by the addition of high protein fraction (Skierkowski et al., 1990).

Screw Speed

Many extruders are provided with a variable speed

drive, which enables the screw speed to be varied easily and independently. Screw speed has been reported to affect product characteristics significantly. Pham and Del Rosario (1984) reported higher water-absorption capacity with increasing screw speed and process temperature for defatted soybean and air classified mung bean. Aguilera and Kosikowski (1976) observed screw speed to be an adjustable parameter for obtaining desired product characteristics at constant temperature and moisture content. Bhattacharya et al. (1986) used combinations of different screw speeds with different die diameters to generate different shear environments and observed significantly different extrudate characteristics. Badrie and Mellowes (1991) texturized cassava flour by single-screw extrusion processing and reported that the effect of screw speed was significant on all textural parameters except springiness and energy for the first bite. Screw speed, along with process temperature and feed moisture content, was found to significantly affect the physicochemical changes in extrusion processing of corn starch (Owusu-Ansah et al., 1983). Hsieh et al. (1990) reported that higher screw speed during extrusion processing of corn meal increased axial expansion, breaking strength, and specific energy but decreased radial expansion, bulk density, die pressure, and percentage torque.

pH

The pH of the mix, especially protein mix, is an

important process variable. The physicochemical state of the protein, particularly conformation and protein-water interactions as affected by pH, influences the dough fluidity in the extruder and the product characteristics such as shape, rehydration, density, and texture.

Acidic pH (5-6.5) results in dense and chewy products that shape easily, harden slowly, and show poor rehydration properties. Products with opposite characteristics are observed with alkaline pH (Kinsella, 1978). Simonsky and Stanley (1982) showed good texture of soybean extrudate at pH 8. Significant decrease in extrudate texture and loss of structural integrity and increased solubility were observed at the extremes of the pH range. Extrusion cooking of sunflower protein resulted in good texturized derivative at its natural pH 7.0 (Rossi and Peri, 1986). Deviation from pH 7.0, denaturing the protein with heat treatment, and addition of lecithin resulted in lower quality extrudate.

Pham and Del Rosario (1984) reported higher water-absorption capacity with increasing pH for defatted soybean and air classified mung bean. Rhee et al. (1981) studied extrusion of soy flour and showed a decrease in bulk density and an increase in water retention and holding capacities of the extrudates with increasing pH values. Raw ingredients with lower pH values had stronger texture. Alkaline pH of flour resulted in an increased surface smoothness and extrudate diameter. Dahl and Villota (1991) texturized soy

flour, modified by addition of acid (HCl) or alkali (NaOH). They observed that acid-modified, texturized protein had little expansion, increased peak force, increased work of shearing, and a nonoriented fiber arrangement. Slightly alkaline extrudates had poor texture but increased rehydration, whereas strongly alkaline extrudate had poor rehydration but high peak force measurements and higher density.

Length/Diameter (L/D) Ratio

The extruder L/D ratio is the distance from the rear edge of the feed opening to the discharge end of the barrel bore divided by the bore diameter to express a ratio where the diameter is reduced to 1 (Harper, 1981). The L/D ratio affects the flow characteristics of the product in the barrel and the shear to which it is subjected, thereby affecting extrudate characteristics. Bhattacharya et al. (1988, 1990), studying the extrusion of minced fish blended with wheat flour, observed that higher L/D ratios and increased fish content resulted in the formation of organized fibrous structure. In most of the literature reviewed, however, L/D ratio was found to be constant.

Ultrafiltration

Ultrafiltration (UF) can be defined as a method for simultaneously purifying, concentrating, and fractionating macromolecules or colloidal substances in process streams

(Cheryan, 1986). UF is used in a variety of industries like food, pharmaceutical, biological, and chemical processing industries.

UF involves passing a liquid mixture over a membrane under pressure. The pressure gradient across the membrane forces the solvent and smaller species through the pores of the membrane, while the larger molecules are retained. The size of the particles retained can be controlled by using membranes with the desired pore size. The retentate becomes enriched in the retained macromolecules while the permeate is depleted of the macromolecules. Depending on rejection of the permeable solute by the membrane, some of it may be present in the retentate in the same or higher concentration as compared to the permeate. Since the retentate has a much smaller volume as compared to the feed, there is an increase in the concentration of the retained macromolecules.

The four major factors affecting the permeation rate in UF are (1) pressure, (2) temperature, (3) feed concentration, and (4) flow rate (Cheryan, 1986). Pressure is the driving force in UF. An increase in pressure increases the permeation rate, up to a limiting pressure value. Since UF deals with the separation of large molecules, like proteins, the osmotic pressures involved are fairly low. Thus, low pressures (0.1 to 1.0 MPa) are used during operation, which results in lower operating costs. UF is mostly done at ambient temperatures (Cheryan, 1986).

Table 1. Effect of Operating Temperature on Ultrafiltration of Milk (Cheryan, 1986)

Temperature (°C)	Effect
2-6°C	--increased viscosity --low flux --little microbial growth
15-45°C	--little protein denaturation --significant microbial growth --better flux
50-60°C	--good flux --lowered viscosity --low microbial growth --some whey protein denaturation

Thus, there are no problems of thermal or oxidative degradation. An increase in operating temperature increases the permeate rate. Table 1 lists the effects of temperature on UF of milk. An increase in the feed concentration causes an exponential decrease in the permeation rate as there is a huge increase in the viscosity of the feed (Cheryan, 1986). Flow rate also has a significant effect on the permeation rate. Turbulence in the flow sweeps away the solute accumulated on the membrane surface, thereby increasing permeation rate.

UF is used extensively in the dairy industry. The two largest applications are the fractionation of cheese whey and pre-concentration of milk for cheese manufacture. When milk is passed over a UF membrane, water, salts, lactose, non-protein nitrogen, free amino acids, and small peptides pass through the membrane while fat and most of the protein are retained. Not all calcium and phosphorous are removed because some of the calcium and phosphorous is bound to casein micelles as colloidal calcium phosphate.

Product quality considerations favor the concentration of milk by the use of membrane separation techniques like UF, because extensive heating during evaporative concentration often causes product degradation. Also, since UF does not involve a change in the phase of the solvent, it results in considerable savings in energy.

Spray Drying

Spray drying is used extensively in the dairy industry for the production of a variety of products like skim milk powder, cheese powder, cream powder, and whey powder. As defined by Masters (1985), spray drying consists of the following stages: (1) atomization of the feed into a spray, (2) spray-air contact, (3) drying of spray, and (4) separation of dried product from the air.

Atomization transforms the feed into a spray of minute droplets with a very large combined surface area from which evaporation can take place. The surface area of a droplet

being inversely proportional to its mass, smaller droplets give more effective evaporation. Two types of atomizers are commonly used in milk powder manufacture, namely, rotary atomizers and pressure nozzle atomizers. Pressure nozzle atomizers can give powders of a higher bulk density and better flowability. Also, powder manufactured by nozzle atomization has a lower level of occluded air, as well as a lower level of free fat. Rotary atomizers, on the other hand, can handle high viscosity materials more easily and have a much greater production capacity (Early, 1991).

Following atomization, the droplets are brought into contact with hot air. In milk powder manufacture, co-current flow configuration for mixing the hot air with the atomized spray is used. As the hottest air is in contact with the wettest droplet, the droplet temperature remains at the wet bulb temperature and heat damage due to denaturation is minimized (Anonymous, 1987).

Initially, as the moisture evaporates from the surface of the droplet, it is replaced by diffusion from within. This is the constant rate period of drying. However, as more moisture is lost, the diffusion rate falls and surface moisture saturation cannot be maintained. As a result, the surface of the droplet forms a dry shell. The period through which further drying occurs is known as the falling rate drying period. If the droplet is too large, and a case-hardened shell forms around it too quickly, steam and

air inside the particle can rapidly expand to fracture the surface. This can result in an increase in the number of particle fragments in the final product.

After drying, the powder is separated from the moisture-laden air. Most of the powder falls from suspension in the air and is discharged through the powder outlet while the air is removed through the air outlet. Some of the powder, especially the fines, also gets removed with the air. This air is made to flow in a cyclone so that a vortex is created in which the powder particles move towards the periphery while the air passes up along the axis of the vortex, and the fines get separated from the air.

Response Surface Methodology

Response surface methodology (RSM) is a collection of mathematical and statistical techniques useful for analyzing problems where several independent control variables influence a dependent response variable, and the goal is to optimize the response variable (Montgomery, 1976). The control variables are assumed to be continuous variables while the response variable is assumed to be a random variable. RSM has been used extensively in various extrusion studies (Aguilera and Kosikowski, 1976; Olkku and Vainionpaa, 1980; El-Dash et al., 1983; Meuser et al., 1984; Linko et al., 1985; Reinikainen et al., 1986; Park et al., 1993).

If y is a response, and x_1, x_2,...., x_n are a set of control variables, then one can suppose a functional relationship between the two, as

$$y = f(x_1, x_2, \ldots, x_n) + e$$

where e is a random error component. If the response is expressed as E(y) = n, then the response surface is represented by

$$n = f(x_1, x_2, \ldots, x_n)$$

The first step in RSM is to find a suitable approximation of f, the function expressing the relationship between the response variable and the independent control variables. Usually, a low-order polynomial in some region of the independent variables is used. The polynomial to be used depends on the accuracy needed and on the contribution of the extra terms to the overall fitting. A second-degree polynomial of the form

$$y = b_0 + \sum_{i=1}^{n} b_i x_i + \sum_{i=1}^{n} \sum_{j=i+1}^{m} b_{ij} x_i x_j + \sum_{i=1}^{n} b_{ii} x_i^2$$

is usually adequate to determine the changes in an extrusion process (Eerikainen and Linko, 1989). The parameters in the approximating polynomial are estimated by the method of

least squares. The RSM analysis is then done in terms of the fitted surface. The analysis of the fitted surface will be approximately equivalent to the analysis of the actual system if the fitted surface is an adequate approximation of f. The coefficient of determination, R^2, gives the fit of the model to the experimental data (Adams, 1985). R^2 can be determined by the following relationship :

$$R^2 = 1 - \frac{\sum_{i=1}^{n} (Y_i - Y_{ip})^2}{\sum_{i=1}^{n} (Y_i - Y_{ip})^2 + \sum_{i=1}^{n} (Y_i - Y_{av})^2}$$

where

Y_i : real value of Y from experiment i

Y_{ip} : predicted value for Y for experiment i

Y_{av} : average value for Y in all experiments

The closer the R^2 value is to 1, the better predictor the model is of the actual response surface.

Response Surface Designs

A careful experimental design is necessary for successful response surface analysis. Designs for fitting response surfaces are called response surface designs.

An experimental design for fitting a second-order model must have at least three levels of each factor in order for

the model parameters to be estimated. Rotatable designs are the most preferred second-order response surface designs. If the variance of the predicted response y at some point x is a function of only the distance of the point from the design center and not a function of the direction, the design is known as a rotatable design (Montgomery, 1976). The variance contours of the response are thus concentric circles, and rotating the design about the center does not change the variance of the response. The most widely used rotatable design for fitting a second-order model is the central composite design. The composite design has three parts: 2^k factorial or fractional factorial points, an extra point at the center of the entire design, and 2k (where k = number of factors) axial (extra) points, one at either extremes of each factor and at the center of all other factors (Anderson and McLean, 1974). Hence, the total number of experimental combinations in a composite design with a complete factorial in it is $2^k + 2k + 1$.

The advantage of a composite design over the fractional or complete three-levelled factorial is that it reduces the number of treatment combinations required to estimate the squared terms in a second-order model, as is evident from Table 2.

Table 2. Number of Treatment Combinations (Anderson and McLean, 1974).

Number of factors, k	Three-levelled factorial, 3^k	Composite 2^k+2k+1
2	9	9
3	27	15
4	81	25
5	243	43

OBJECTIVES

The objectives of this study were

1. To study the effects of moisture content, protein content, lactose/protein ratio, and feed rate on the physical, functional, and microstructural attributes of extruded corn starch-skim milk powder blends.
2. To determine extrusion conditions most likely to produce extrudates with properties suitable for a snack type product.

MATERIALS AND METHODS

Milk

Pasteurized skim milk was obtained from the Utah State University Gary H. Richardson Dairy Products Laboratory.

Ultrafiltration

The skim milk was heated to 44°C and ultrafiltered to three lactose/protein ratios (Table 3). Ultrafiltration was accomplished using a polysulfone, spiral-wound membrane unit having a molecular weight cutoff of 20,000 daltons (Osmonics Inc., Minnetonka, MN). Inlet and outlet pressures were 80 psi and 40 psi. Total protein (percent nitrogen x 6.38) was determined by Association of Official Analytical Chemists method (AOAC, 1984). Lactose content was measured using an enzymic assay developed by Cleyn and Trout (1984). A kit based on this method was used for the assays (Catalog #176303, Boehringer Mannheim, Indianapolis, IN).

Spray Drying

The ultrafiltered skim milk was spray dried using a Niro Mobile Minor laboratory spray drier (Niro Atomizers Inc., Columbia, MD), modifying the feed rate such that an electrically generated inlet temperature of 220°C resulted in an outlet temperature of 80°C. The powder obtained was collected and stored in polyurethane containers at 1°C.

Table 3. Composition of Ultrafiltered Skim Milk

Total Solids %	Protein Content %	Lactose Content %	Lactose/ Protein
13.67	7.60	4.66	0.61
17.07	12.60	4.35	0.34
19.88	15.69	4.14	0.26

Corn Starch

Pearled corn starch was used. It was obtained as a gift from the Staley Company (A.E. Staley Manufacturing Company, Decatur, IL). The corn starch granules were separated according to size by use of two U.S. standard testing sieves. Granules used were such that they passed through a #8 sieve but did not pass through a #16 sieve.

Feed Preparation

Pearled corn starch was mixed with skim milk powder to make up the feed. The bulk of the material in each feed mixture was corn starch, and skim milk powder was added in the proper proportion to give the appropriate protein percentage and lactose/protein ratio. Table 4 gives the feed formulation for each experimental run.

Experimental Design

Response surface methodology was used for the study.

Table 4. Feed Formulation

Run Number	Trial Number	Corn Starch (gm)	Milk Powder (gm)	Water (gm)
1	1	445.94	104.06	183.33
2	4	517.14	32.86	183.33
3	10	323.89	226.10	235.71
4	1	445.94	104.06	183.33
5	3	438.51	111.49	235.71
6	20	507.60	42.39	235.71
7	5	374.74	175.25	183.33
8	1	445.94	104.06	183.33
9	21	512.90	37.09	137.50
10	17	523.86	26.13	137.50
11	1	445.94	104.06	183.33
12	2	452.44	97.55	137.50
13	8	445.94	104.06	183.33
14	18	323.89	226.10	235.71
15	22	390.71	159.29	235.71
16	11	352.16	197.84	137.50
17	1	445.94	104.06	183.33
18	15	410.62	139.38	137.50
19	12	507.60	42.39	235.71
20	23	410.62	139.38	137.50
21	7	424.70	125.30	183.33
22	25	523.86	26.13	137.50
23	6	461.72	88.27	183.33
24	1	445.94	104.06	183.33
25	19	352.16	197.84	137.50
26	13	512.90	37.09	137.50
27	24	520.13	29.87	235.71
28	16	520.13	29.87	235.71
29	9	445.94	104.06	183.33
30	14	390.71	159.29	235.71

Four variables (moisture content, protein content, lactose/protein ratio, and feed rate) were chosen as the control variables. Based on preliminary trial runs, the ranges for these variables were determined. Seven response variables were evaluated: expansion ratio, product temperature, bulk density, color, shear stress, viscosity, and water absorption index. Scanning electron microscopy was done to find out the relationship between the microstructural and rheological properties of the extrudates.

A central composite in cube experimental design was generated using a statistical software called ECHIP (Echip Inc., Hockessin, DE). The design consisted of 16 (2^4) factorial points, 8 (2x4) axial points, and 6 center points for replication, giving a total of 30 experimental runs (Table 5). A quadratic model was used to express the response variables in terms of the control variables. The effect of each control variable on each response variable and the relationship, if any, among the different response variables were determined. Response surfaces and contour plots were generated. Optimum extrusion conditions for each response variable were determined.

Extrusion

All extrusion was done on a Wenger X-5 single-screw cooker extruder equipped with eight barrel sections attached

Table 5. Experimental Design

Run Number	Trial Number	Moisture Content %	Protein Content %	Lactose/ Protein	Feed Rate gm/min
1	1	25.00	9.50	0.435	77.50
2	4	25.00	3.00	0.435	77.50
3	10	30.00	16.00	0.610	100.00
4	1	25.00	9.50	0.435	77.50
5	3	30.00	9.50	0.435	77.50
6	20	30.00	3.00	0.610	55.00
7	5	25.00	16.00	0.435	77.50
8	1	25.00	9.50	0.435	77.50
9	21	20.00	3.00	0.610	55.00
10	17	20.00	3.00	0.260	100.00
11	1	25.00	9.50	0.435	77.50
12	2	20.00	9.50	0.435	77.50
13	8	25.00	9.50	0.435	55.00
14	18	30.00	16.00	0.610	55.00
15	22	30.00	16.00	0.260	55.00
16	11	20.00	16.00	0.610	100.00
17	1	25.00	9.50	0.435	77.50
18	15	20.00	16.00	0.260	100.00
19	12	30.00	3.00	0.610	100.00
20	23	20.00	16.00	0.260	55.00
21	7	25.00	9.50	0.610	77.50
22	25	20.00	3.00	0.260	55.00
23	6	25.00	9.50	0.260	77.50
24	1	25.00	9.50	0.435	77.50
25	19	20.00	16.00	0.610	55.00
26	13	20.00	3.00	0.610	100.00
27	24	30.00	3.00	0.260	55.00
28	16	30.00	3.00	0.260	100.00
29	9	25.00	9.50	0.435	100.00
30	14	30.00	16.00	0.260	100.00

to cooling water/compressed air lines (Wenger Manufacturing, Sabetha, Kansas) (Figure 1); screw length: 432 mm, diameter: 25 mm, die diameter: 5 mm. The extruder was equipped with a variable speed screw feeder. Water addition was controlled by use of a rotameter (L-03219-25, Cole-Parmer Instrument Company, Niles, IL). Screw speed was constant at 500 rpm. The first six barrel sections were cooled with water while the last two were filled with water, and then valves were closed.

A specially built barrel spacer was placed between the end of the screw and the die plate. It was equipped with a rigid thermocouple which extended into the product flow, thereby giving true product temperature (Figure 2). According to Mulvaney (personal communication), such a configuration gives a better indication of the product temperature. The thermocouple was connected to a PC-based data acquisition unit (WB-ASC Interface Card, Omega Engineering, Inc., Stamford, CT). Product temperature was recorded every 5 seconds. Extrudate samples were collected when a time versus temperature graph generated by the data acquisition software indicated that the product temperature had stabilized.

Sample Storage

After the samples were collected, they were dried in a vacuum oven at 70°C under 23 inches Hg for 2 hours. The

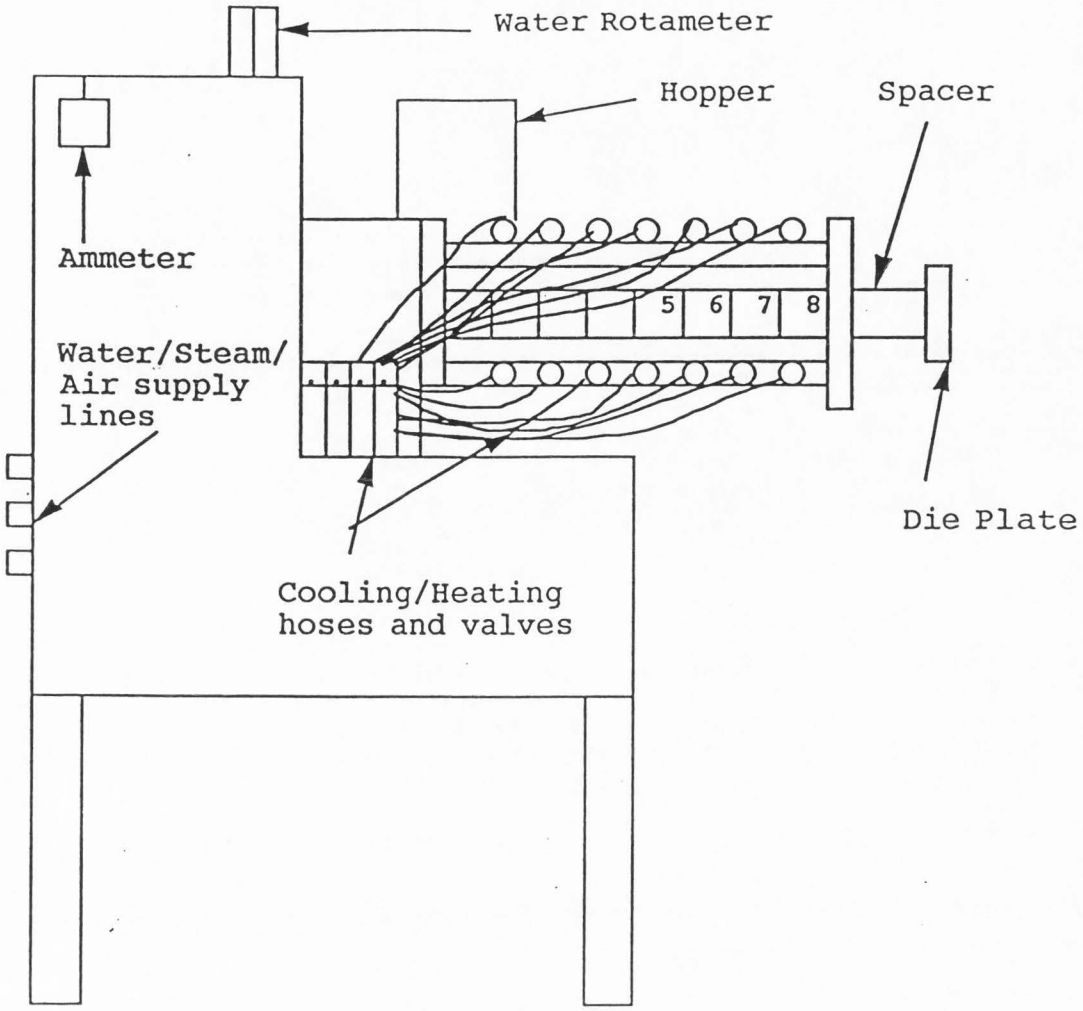


Figure 1. Wenger X-5 single-screw extruder

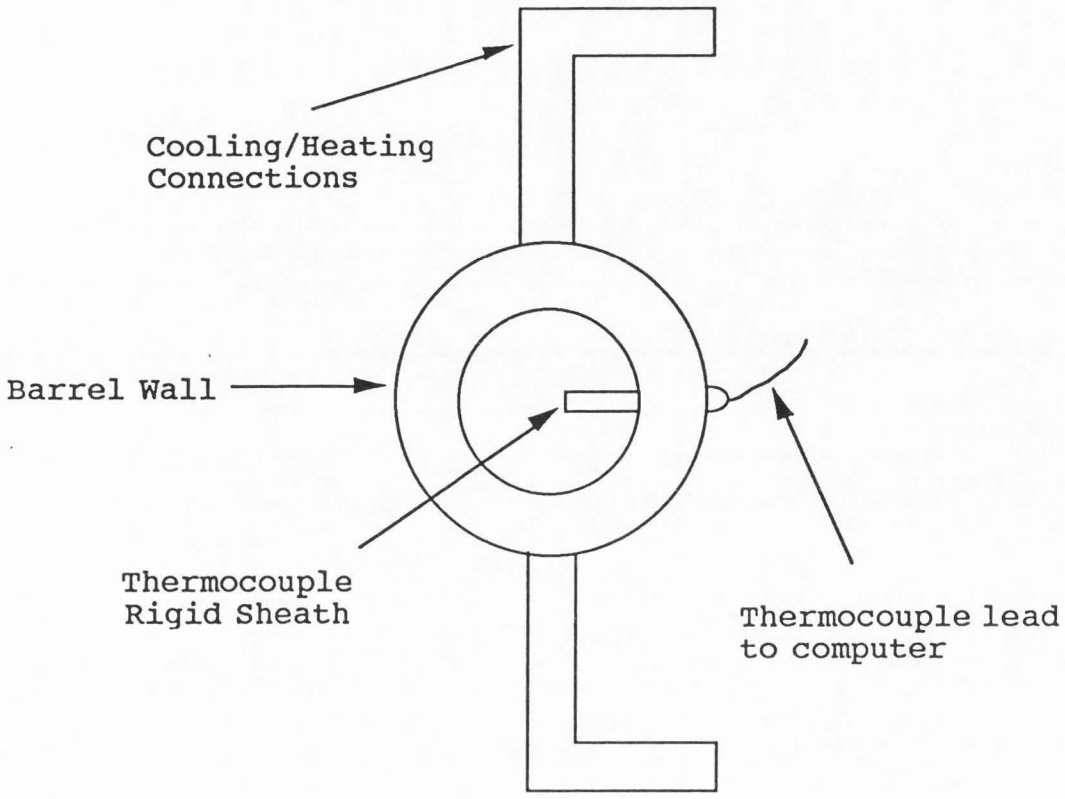


Figure 2. Barrel spacer equipped with thermocouple.

dried samples were placed in Ziploc® gallon-size freezer bags and stored at 4°C until analysis.

Expansion Ratio

Expansion ratio was calculated as the ratio of the diameter of the extrudate to the diameter of the die. The average diameter of the extrudate was obtained by taking measurements with a Vernier caliper on 20 randomly selected segments of each run.

Product Temperature

Product temperature for each run was obtained as the average of the temperatures over the time period during which the samples were collected. The samples were collected when the temperature had stabilized and did not vary over more than a 5°C range.

Bulk Density

Randomly selected extrudates were chopped so that they passed through a U.S. #4 sieve but were retained on a U.S. #6 sieve. A 100-ml graduated cylinder was filled with the chopped extrudates, while repeatedly gently tapping the cylinder bottom until there was no further volume reduction. Bulk density was calculated as weight of the sample divided by the volume occupied by the sample. Each run bulk density was determined in triplicate.

Color

Chopped extrudates (passing through U.S. #4 sieve but retained on U.S. #6 sieve) were evaluated for color using a reflectance colorimeter (Omnispec® 4000, Wescor Inc., Logan, UT). L^* values (0 = black, 100 = white) and b^* values (positive values indicating yellowness and negative values blueness) were determined. b^* values were used in the analysis as they showed a higher coefficient of determination (R^2) than L^* values. Each value reported is an average of 24 readings.

Shear Stress

A Warner-Bratzler shear was used to determine the shear stress. Maurice et al. (1976) reported a strong correlation of Warner-Bratzler values with sensory evaluation panel analyses for texturized soy protein. The sample was inserted into a triangular opening in the blade between two rectangular bars. The maximum shear force was divided by the average cross-sectional area of the extrudate (determined by a Vernier caliper) to give the shear stress. Each run shear stress reported is an average of 5 readings.

Viscosity

Cooked product viscosity was determined using the procedure of Maga and Lorenz (1978). Twenty-three grams of ground product (< 40 mesh) was added to 175 ml of boiling water in a 250-ml beaker and stirred vigorously with a fork.

Boiling was continued for 2 minutes. The slurry was removed from the heat source and held in a 30°C water bath for 10 minutes. The beaker was placed on a scale and water was added to a total slurry weight of 200-gms to compensate for water loss due to evaporation. The slurry was held at 30°C for 1 hour before making Brookfield measurement. Each value reported is an average of 3 readings.

Water Absorption Index

Water absorption index (WAI) was determined using the procedure of Anderson et al. (1969). A 2.5-gm sample of ground product (< 60 mesh) was suspended in 30-ml of water at 30°C in a 50-ml tared centrifuge tube, stirred intermittently over a 30-minute period, and centrifuged at 3000xg for 10 minutes. The supernatant liquid was poured out. The remaining gel was weighed and WAI was calculated as the weight of gel obtained per gram of dry sample. Each value reported is an average of three readings.

Scanning Electron Microscopy

Sample extrudates were frozen with liquid nitrogen and fractured. They were mounted on aluminum stubs and coated with a 2.5-nm layer of iridium using an IBS/TM 200S ion beam sputterer (VCR Group, San Francisco, CA). Due to the porous nature of the samples, they were coated with silver-conducting paint on the outside. The samples were then viewed in an S4000 field emission scanning electron

microscope (Hitachi, Japan) at 90° tilt and 5.0 kV. The micrographs were obtained at 500X magnification on cross sections of extrudates cut perpendicular to the path of extrusion.

RESULTS AND DISCUSSIONS

Physical and Functional Properties

The experimental data for the various physical and functional properties evaluated and their analysis of variance tables are listed in Appendices 1 and 2, respectively.

Expansion Ratio

When the cooked product exits the extruder die, expansion occurs for two reasons: (1) vaporization of water until temperature reaches 100°C (Williams et al., 1977) and (2) pressure differential between the interior and exterior of the extruder.

Table 6 lists the effects of the various control variables on expansion ratio (ER). Moisture content had a significant linear and quadratic effect on ER and also had a significant interaction effect with feed rate.

ER decreased with increase in moisture content until about 26%-27% moisture content and then increased slightly at very high moisture content (Figure 3). An increase in moisture content makes the food mass less viscous, thereby decreasing the pressure differential which results in less expansion. However, the vaporization of water is the principal cause of expansion of the product. At very high moisture contents it is possible that there is more free water available for vaporization and this makes up for the

Table 6. Effect Table for Expansion Ratio

Term	Effect
moisture content	***
protein content	.
lactose/protein	.
feed rate	.
moisture content x protein content	.
moisture content x lactose/protein	.
moisture content x feed rate	*
protein content x lactose/protein	.
protein content x feed rate	.
lactose/protein x feed rate	.
$(\text{moisture content})^2$	***
$(\text{protein content})^2$	.
$(\text{lactose/protein})^2$	.
$(\text{feed rate})^2$	.

*** : $p < 0.001$

* : $p < 0.05$

. : $p > 0.05$

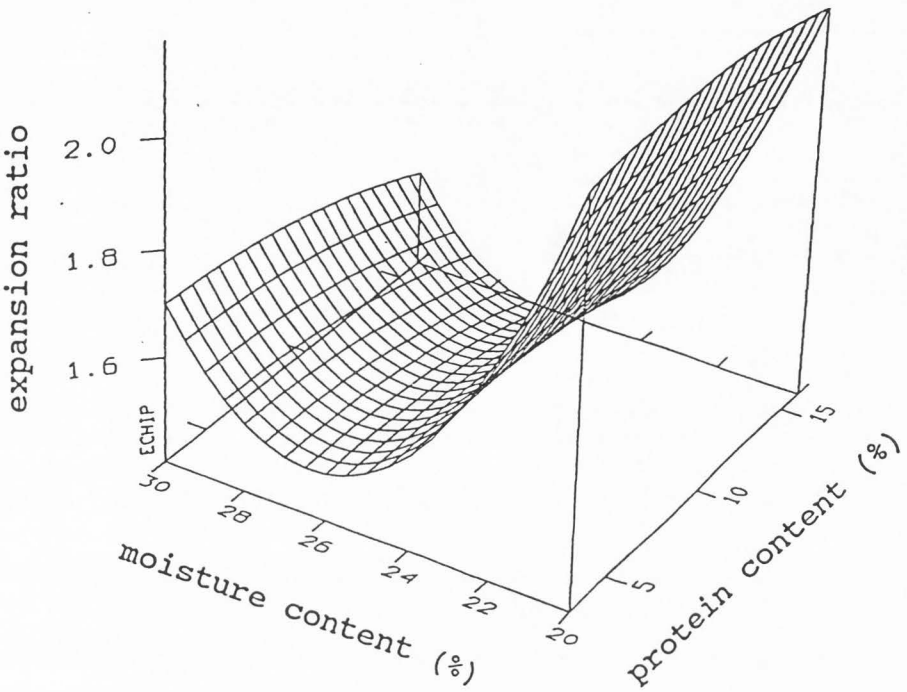


Figure 3. Response surface for expansion ratio as plotted for moisture content and protein content with lactose/protein ratio and feed rate fixed at mid-point values.

lower pressure differential, thereby causing a slight increase in the ER. At low moisture content, ER decreased slightly at higher feed rates, but at very high moisture content, the trend was reversed (Figure 4).

Product Temperature

Product temperature (PT) was influenced by all control variables except feed rate (Table 7). PT decreased with an increase in moisture content and protein content (Figure 5), and with an increase in lactose/protein ratio (Figure 6). There were also significant interactions between moisture content and protein content, and between lactose/protein ratio and feed rate. At the same moisture content, PT decreased with increasing protein content (Figure 5). PT was maximum at low lactose/protein ratios and low feed rates (Figure 6).

Heat is generated in an extruder by shear and friction developed by a rapidly rotating shaft working against the material to be extruded (Chilton's, 1977). Frictional heat generated is directly proportional to the viscosity of the food mass (Tadmor and Klein, 1970; Harper et al., 1971). An increase in moisture content made the food mass less viscous, which resulted in less shear and friction and consequently less heat was generated. An increase in protein content decreased the starch content, resulting in less gelatinized starch, which reduced the viscosity of the food mass. Strong water-binding agents like sugars bind

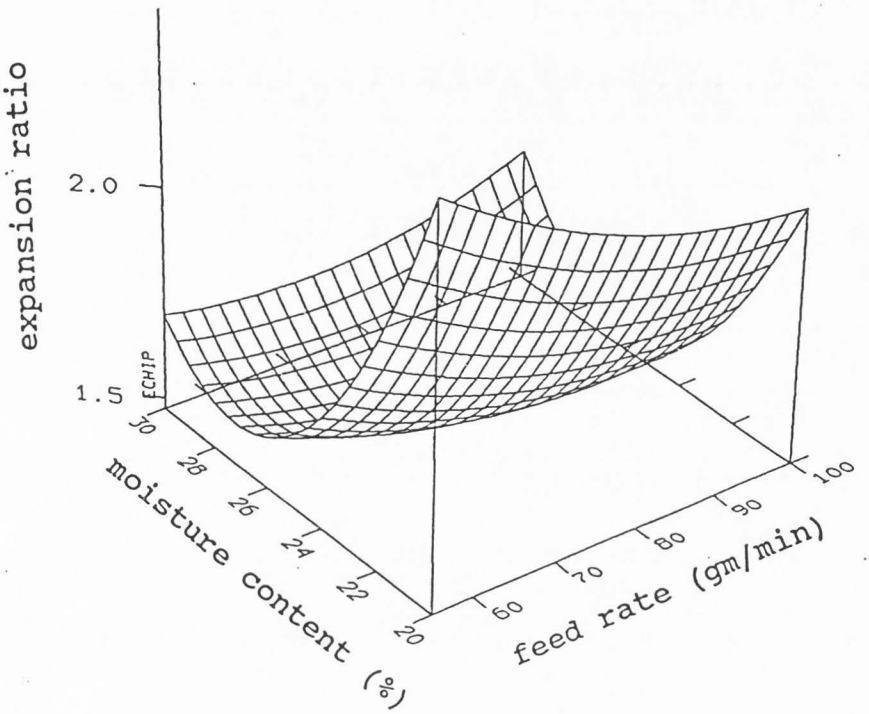


Figure 4. Response surface for expansion ratio as plotted for moisture content and feed rate with protein content and lactose/protein ratio fixed at mid-point values.

Table 7. Effect Table for Product Temperature

Term	Effect
moisture content	***
protein content	***
lactose/protein	**
feed rate	.
moisture content x protein content	*
moisture content x lactose/protein	.
moisture content x feed rate	.
protein content x lactose/protein	.
protein content x feed rate	.
lactose/protein x feed rate	**
$(\text{moisture content})^2$	*
$(\text{protein content})^2$	.
$(\text{lactose/protein})^2$	.
$(\text{feed rate})^2$	.

*** : $p < 0.001$

** : $p < 0.01$

* : $p < 0.05$

. : $p > 0.05$

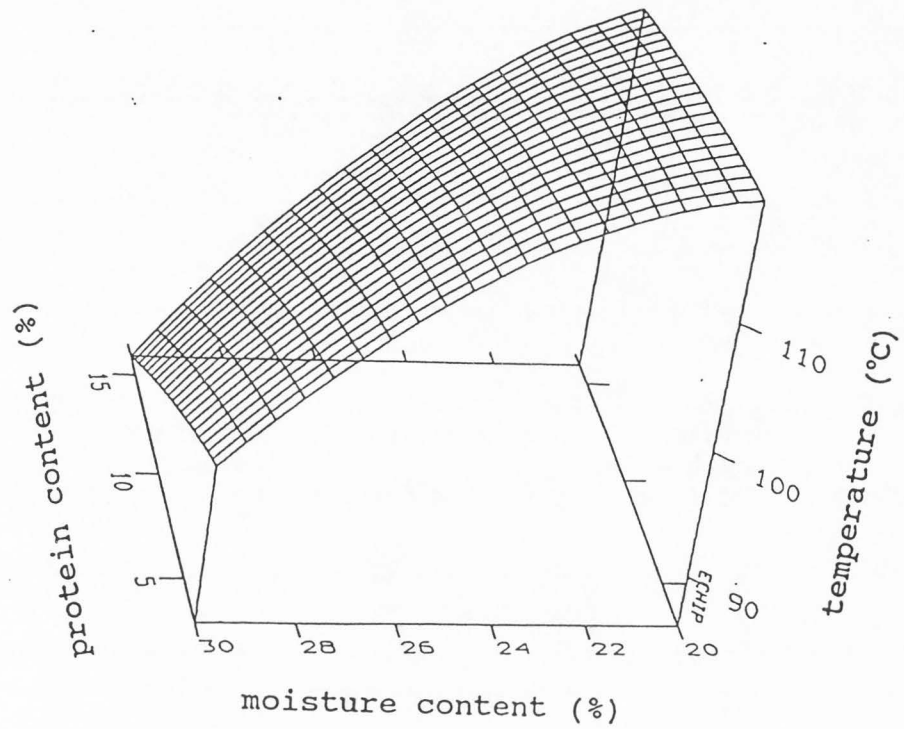


Figure 5. Response surface for product temperature as plotted for moisture content and protein content with lactose/protein ratio and feed rate fixed at mid-point values.

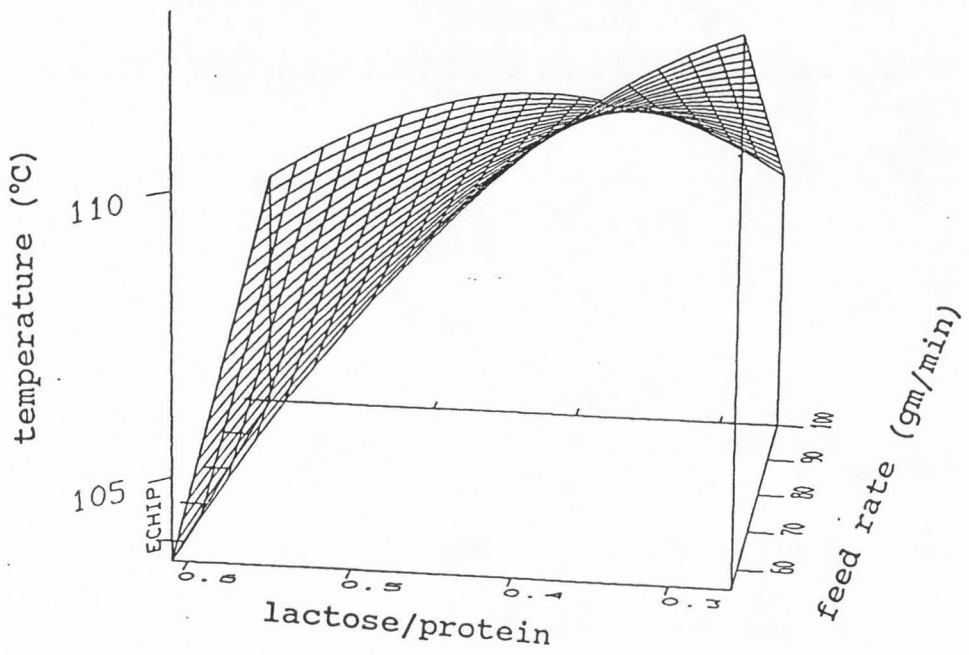


Figure 6. Response surface for product temperature as plotted for lactose/protein ratio and feed rate with moisture content and protein content fixed at mid-point values.

water in competition with starch and so reduce starch gelatinization, thereby making the food mass less viscous. As such, an increase in the lactose/protein ratio also decreased PT.

Bulk Density

Moisture content was the main and only control variable affecting bulk density (BD), although protein content had a significant second-order term (Table 8).

BD increased with increasing moisture content (Figure 7). This agrees with the results of Bhattacharya et al. (1986), who studied extrusion of blends of corn gluten meal and soy protein concentrate. An increase in moisture content decreases expansion ratio and hence results in a denser product. Also, higher initial moisture content means higher moisture in the final product, which could also account for the higher BD.

Color

Table 9 lists the effects of the various control variables on color. Moisture content had the dominating effect, having significant linear and quadratic effects and an interaction effect with feed rate. Protein content also had a significant linear effect.

Color decreased with an increase in moisture content until about 26%-27% moisture content and then increased slightly at very high moisture content (Figure 8).

Table 8. Effect Table for Bulk Density

Term	Effect
moisture content	***
protein content	.
lactose/protein	.
feed rate	.
moisture content x protein content	.
moisture content x lactose/protein	.
moisture content x feed rate	.
protein content x lactose/protein	.
protein content x feed rate	.
lactose/protein x feed rate	.
$(\text{moisture content})^2$	*
$(\text{protein content})^2$	.
$(\text{lactose/protein})^2$	.
$(\text{feed rate})^2$	.

*** : $p < 0.001$

* : $p < 0.05$

. : $p > 0.05$

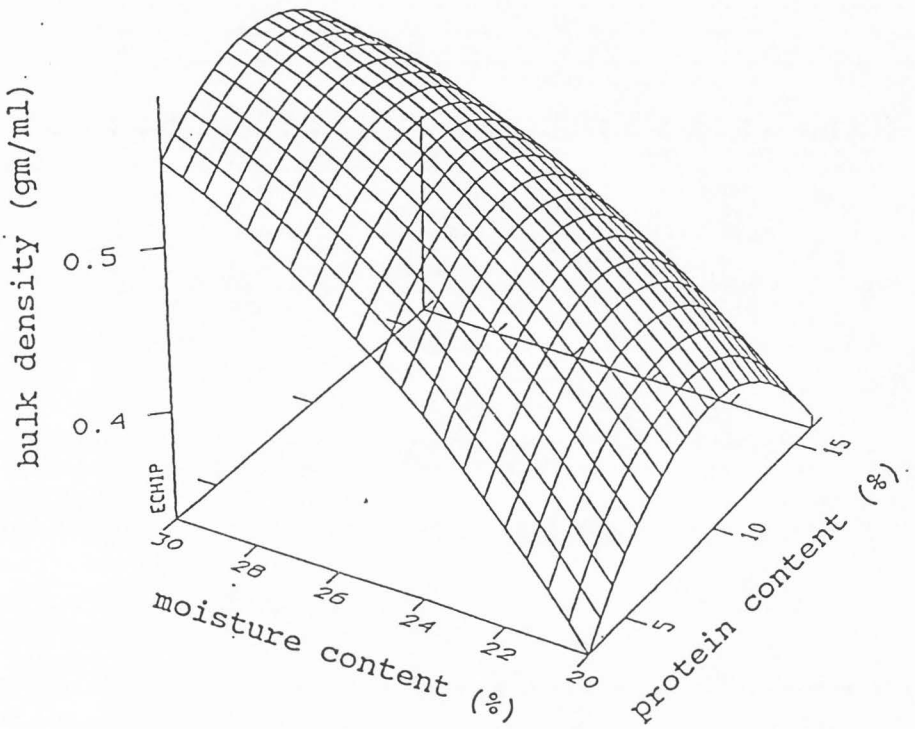


Figure 7. Response surface for bulk density as plotted for moisture content and protein content with lactose/protein ratio and feed rate fixed at mid-point values.

Table 9. Effect Table for Color (b^*)

Term	Effect
moisture content	***
protein content	***
lactose/protein	.
feed rate	.
moisture content x protein content	.
moisture content x lactose/protein	.
moisture content x feed rate	**
protein content x lactose/protein	.
protein content x feed rate	.
lactose/protein x feed rate	.
$(\text{moisture content})^2$	**
$(\text{protein content})^2$	.
$(\text{lactose/protein})^2$	.
$(\text{feed rate})^2$	.

*** : $p < 0.001$

** : $p < 0.01$

. : $p > 0.05$

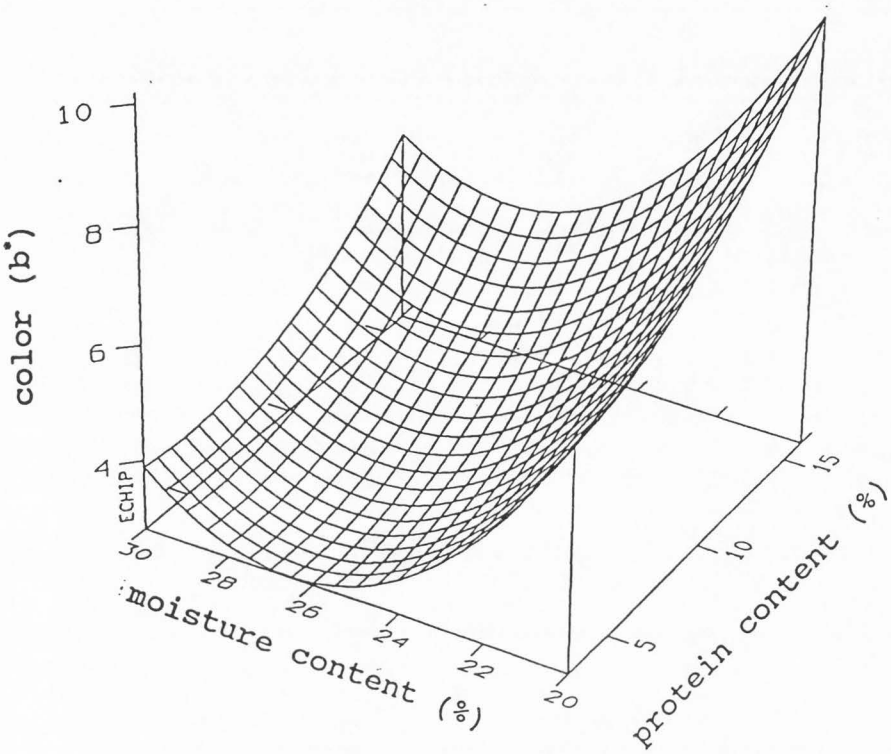


Figure 8. Response surface for color as plotted for moisture content and protein content with lactose/protein ratio and feed rate fixed at mid-point values.

Extrusion under low moisture/high temperature conditions results in more browning (Chilton's, 1977). An increase in moisture content decreased the product temperature, resulting in less browning. Color increased with an increase in protein content (Figure 8). Proteins extruded in carbohydrate-based systems can undergo Maillard reactions in the presence of reducing sugars (Bjorck et al., 1984). An increase in protein content also resulted in an increase in the lactose content due to an increase in the skim milk powder content, which accounted for the increase in browning. Color was maximum at low moisture content and low feed rate (Figure 9).

Shear Stress

Moisture content and protein content were the influencing variables for shear stress, both having significant linear effects (Table 10).

Shear stress increased with an increase in moisture content (Figure 10). When the food product is extruded at high moisture content, it loses some moisture when water flashes into steam, but the resulting product due to its high moisture content is still soft enough to collapse. This forms a tough skin around the surface and reduces the size of the pores within the product. After drying, this type of product becomes very hard (Chilton's, 1977). Also, higher moisture content resulted in a denser product, which could cause more work to be done to shear the product,

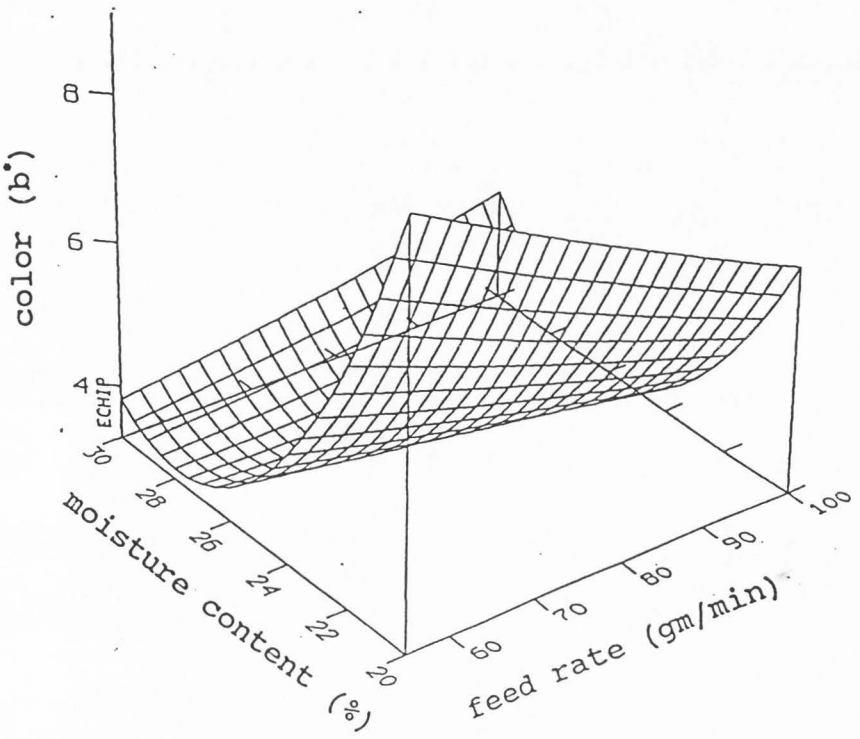


Figure 9. Response surface for color as plotted for moisture content and feed rate with protein content and lactose/protein ratio fixed at mid-point values.

Table 10. Effect Table for Shear Stress

Term	Effect
moisture content	***
protein content	**
lactose/protein	.
feed rate	.
moisture content x protein content	.
moisture content x lactose/protein	.
moisture content x feed rate	.
protein content x lactose/protein	.
protein content x feed rate	.
lactose/protein x feed rate	.
$(\text{moisture content})^2$	.
$(\text{protein content})^2$	.
$(\text{lactose/protein})^2$	.
$(\text{feed rate})^2$	.

*** : $p < 0.001$

** : $p < 0.01$

. : $p > 0.05$

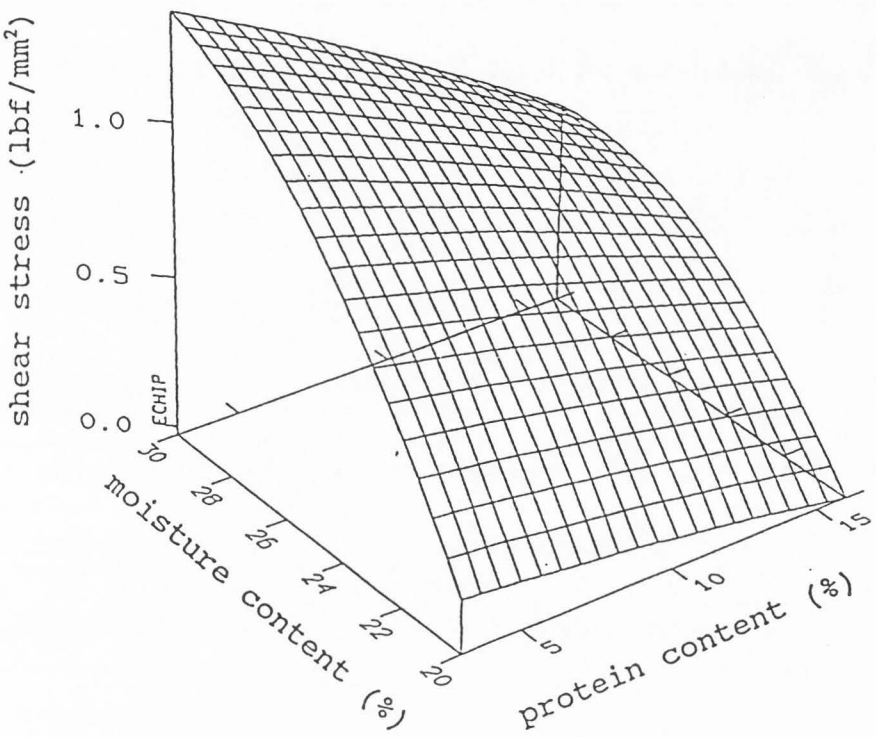


Figure 10. Response surface for shear stress as plotted for moisture content and protein content with lactose/protein ratio and feed rate fixed at mid-point values.

thereby increasing the shear stress.

Shear stress decreased with increase in protein content (Figure 10). As explained by Singh et al. (1991), interactions between the protein and the carbohydrate components control the breaking strength of the extrudate. However, the primary function of the protein fraction within the 20% level of incorporation is to reinforce the cell walls of the expanded extrudates. In our study the maximum protein content was 16% and, as such, an increase in protein content caused a decrease in the shear stress.

Viscosity

Table 11 lists the effects of the various control variables on the cooked product viscosity.

Viscosity increased with an increase in moisture content up to around 28% moisture content and then decreased at very high moisture content (Figure 11). Moisture content had significant interaction terms with protein content and lactose/protein ratio. Viscosity was maximum at around 26%-28% moisture content and low protein content (Figure 11) and at 26%-28% moisture content and low lactose/protein ratio (Figure 12). Viscosity decreased with an increase in protein content and with an increase in lactose/protein ratio (Figure 13). There was also a significant interaction effect between protein content and lactose/protein ratio. Viscosity was maximum at low protein content and low lactose/protein ratio (Figure 13). Overall, cooked product

Table 11. Effect Table for Viscosity

Term	Effect
moisture content	*
protein content	***
lactose/protein	*
feed rate	.
moisture content x protein content	**
moisture content x lactose/protein	*
moisture content x feed rate	.
protein content x lactose/protein	*
protein content x feed rate	.
lactose/protein x feed rate	.
$(\text{moisture content})^2$	***
$(\text{protein content})^2$	.
$(\text{lactose/protein})^2$	.
$(\text{feed rate})^2$	.

*** : $p < 0.001$

** : $p < 0.01$

* : $p < 0.05$

. : $p > 0.05$

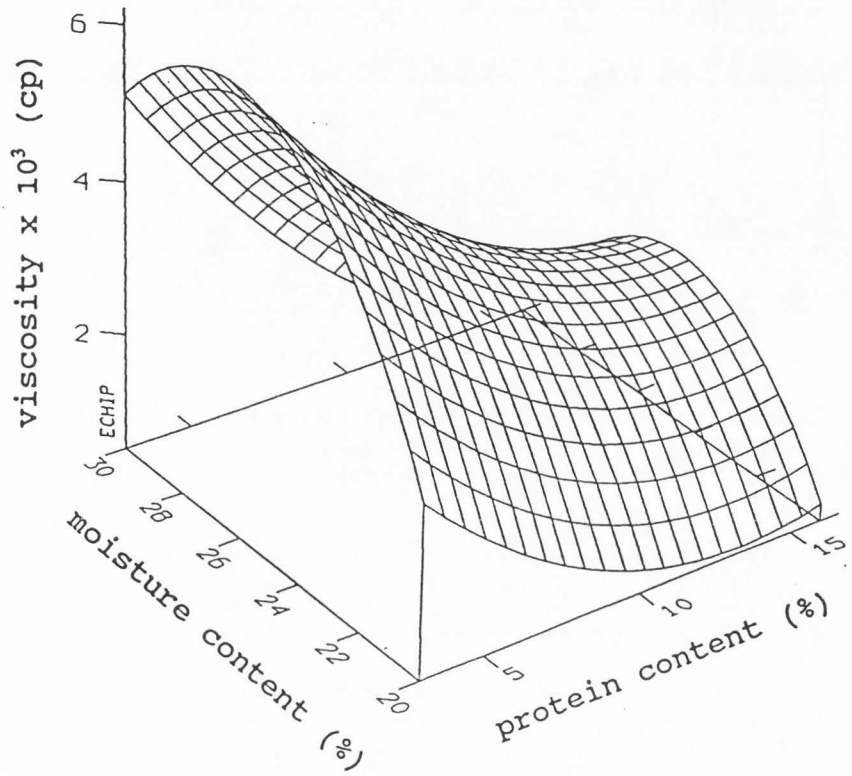


Figure 11. Response surface for viscosity as plotted for moisture content and protein content with lactose/protein ratio and feed rate fixed at mid-point values.

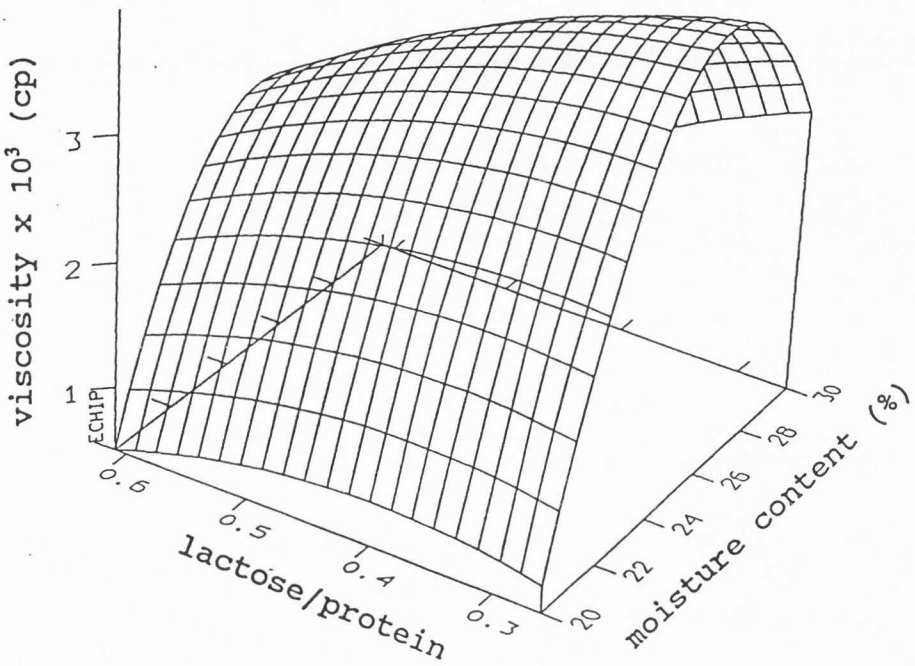


Figure 12. Response surface for viscosity as plotted for moisture content and lactose/protein ratio with protein content and feed rate fixed at mid-point values.

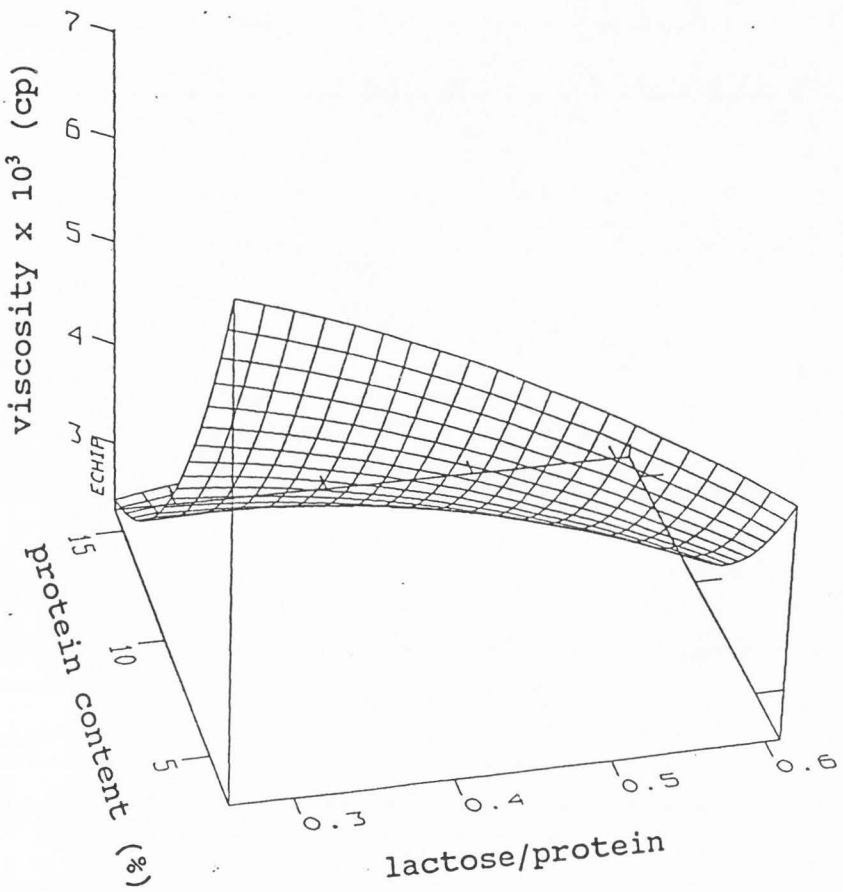


Figure 13. Response surface for viscosity as plotted for protein content and lactose/protein ratio with moisture content and feed rate fixed at mid-point values.

viscosity was maximum at 26%-28% moisture content and low protein content and low lactose/protein ratio.

Very high moisture content resulted in a low product temperature and resulted in a high moisture/low temperature cook, which caused mild gelatinization of starch, thereby resulting in a decrease in viscosity. However, maximum viscosity around 26%-28% moisture content implied that maximum starch gelatinization occurred under those conditions. An increase in protein content resulted in a decrease in starch content, causing a decrease in the gelatinized starch structure. Strong water-binding agents like lactose retard starch gelatinization by binding water in competition with starch. As a result, an increase in lactose/protein ratio decreased viscosity.

Water Absorption Index

The effects of the various control variables on water absorption index (WAI) are listed in Table 12.

WAI increased with an increase in moisture content until about 26%-28% moisture content and then decreased at very high moisture content (Figure 14). WAI decreased with an increase in protein content (Figure 15). WAI also decreased with an increase in lactose/protein ratio and the effect was pronounced at high moisture content (Figure 16). There were also significant interaction effects between moisture content and feed rate and between moisture content and lactose/protein ratio. WAI was maximum at around 26%-

Table 12. Effect Table for Water Absorption Index

Term	Effect
moisture content	***
protein content	***
lactose/protein	**
feed rate	.
moisture content x protein content	.
moisture content x lactose/protein	*
moisture content x feed rate	***
protein content x lactose/protein	.
protein content x feed rate	.
lactose/protein x feed rate	.
$(\text{moisture content})^2$	**
$(\text{protein content})^2$	.
$(\text{lactose/protein})^2$	.
$(\text{feed rate})^2$	.

*** : $p < 0.001$

** : $p < 0.01$

* : $p < 0.05$

. : $p > 0.05$

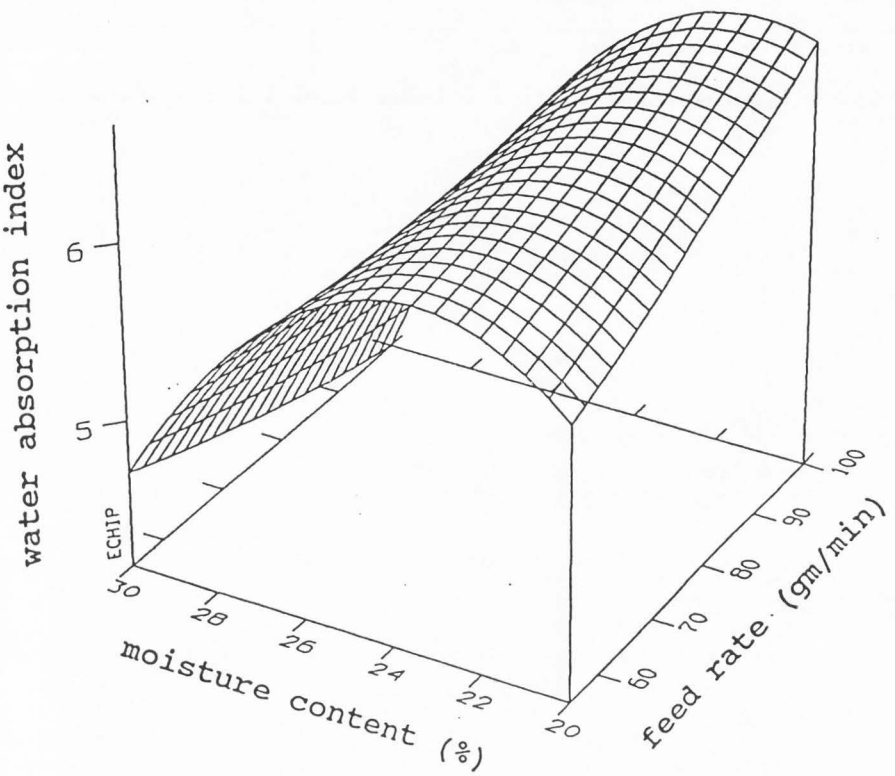


Figure 14. Response surface for water absorption index as plotted for moisture content and feed rate with protein content and lactose/protein ratio fixed at mid-point values.

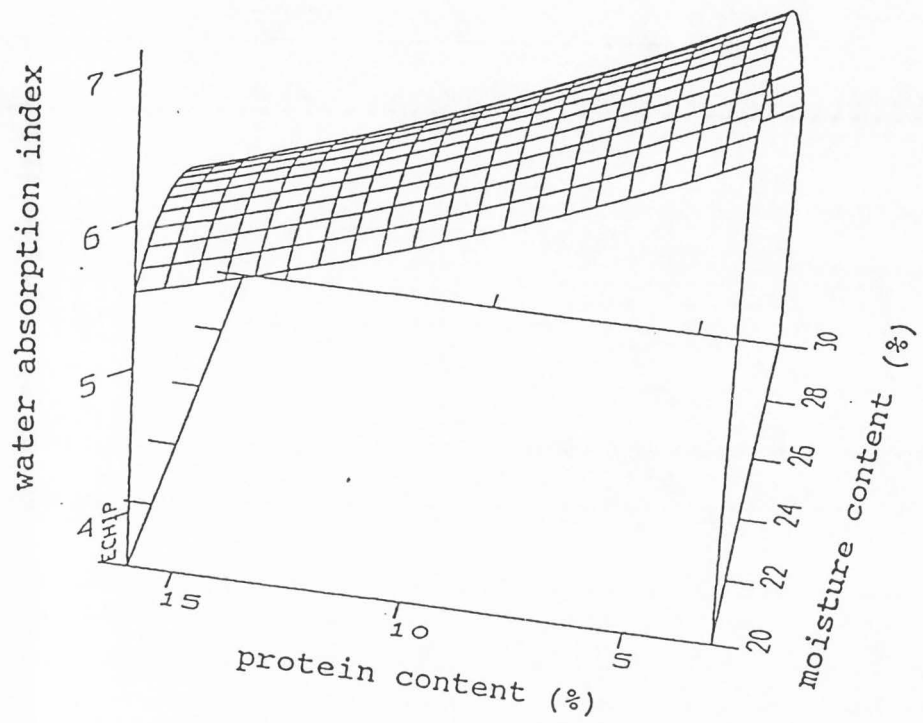


Figure 15. Response surface for water absorption index as plotted for moisture content and protein content with lactose/protein ratio and feed rate fixed at mid-point values.

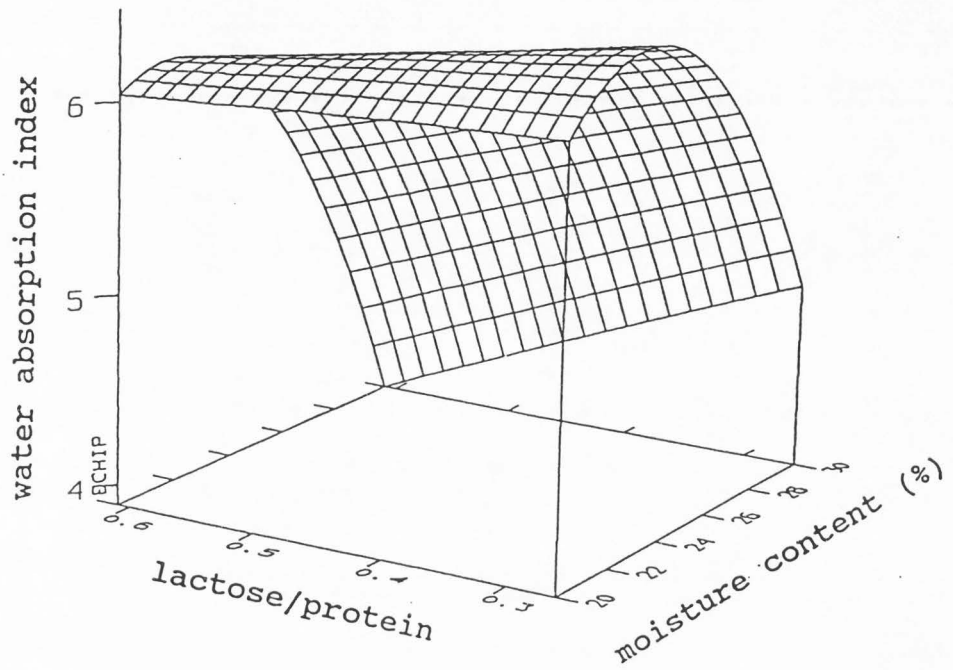


Figure 16. Response surface for water absorption index as plotted for moisture content and lactose/protein ratio with protein content and feed rate fixed at mid-point values.

28% moisture content and high feed rate (Figure 14) and at around 26%-28% moisture content and low lactose/protein ratio (Figure 16).

Starch granules are gelatinized by means of water absorption inside the extruder, and thus starch gelatinization is the primary reason for water absorption. As mentioned above, conditions for maximum starch gelatinization occurred at around 26%-28% moisture content. WAI increased until about 26%-28% moisture content and then decreased at high moisture content. An increase in protein content decreased the starch content and thus the extent of gelatinization. An increased lactose/protein ratio decreased the water activity, thereby reducing the water available for starch gelatinization and so reduced WAI. At low moisture content, temperature had the dominating effect. The effect of lactose/protein ratio was more pronounced at high moisture content.

Model Adequacy

A quadratic model of the form given below was used to express the response variables in terms of the control variables.

$$y = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_4x_4 + b_{12}x_1x_2 + b_{13}x_1x_3 + b_{14}x_1x_4 + b_{23}x_2x_3 + b_{24}x_2x_4 + b_{34}x_3x_4 + b_{11}x_1^2 + b_{22}x_2^2 + b_{33}x_3^2 + b_{44}x_4^2$$

where

y denotes response variable,

x_1 denotes moisture content,

x_2 denotes protein content,

x_3 denotes lactose/protein ratio,

x_4 denotes feed rate, and

b_i and b_{ii} denote regression coefficients.

Appendix 3 lists the regression coefficients for the various response variables.

The adequacy of the model for each response variable was tested by the lack of fit and coefficient of determination (R^2). As can be seen from Table 13, a lack of fit was observed for the response variables product temperature, color, and shear stress. The fit of the model is measured by the ratio of the residual standard deviation to the replicate standard deviation. For a good fit of the model, the residual and replicate standard deviations should be about the same (Echip Inc., Hockessin, DE). The presence of outliers in the data can enlarge the residual standard deviation as compared to the replicate standard deviation, causing the lack of fit. Appendix 4 shows the plots for the residuals versus fitted values for the response variables product temperature, color, and shear stress. As can be seen, outliers are present in each of the plots, which explains the observed lack of fit.

Table 13. Model Adequacy Test for Response Variables

Response Variable	R^2	Lack Of Fit
Expansion Ratio	0.85 (p=0.0007)	No
Product Temperature	0.96 (p=0.0000)	Yes
Bulk Density	0.84 (p=0.0009)	No
Color	0.92 (p=0.0000)	Yes
Shear Stress	0.82 (p=0.0022)	Yes
Viscosity	0.90 (p=0.0000)	No
Water Absorption Index	0.95 (p=0.0000)	No

Unlike the lack of fit, the coefficient of determination (R^2) values (Table 13) indicated that the model was adequate ($p < 0.05$) for all the response variables.

Relationship Between Response Variables

The relationship between the various response variables was determined by the coefficient of correlation (r). ER indicates the extent of puffing of the extrudate. Greater expansion results in a less dense and more fragile product. In our study ER was observed to have a negative correlation

with both BD (r = -0.61) and shear stress (r = -0.62). BD and shear stress were observed to be positively correlated (r = 0.69). Product temperature was observed to be positively correlated to ER (r = 0.52) and WAI (r = 0.81). Higher temperature meant more vaporization of water which caused more expansion. Higher temperature also resulted in greater starch gelatinization, thereby increasing WAI. It was expected that WAI and BD might be related. However, in our study they did not have a significant correlation (r = -0.21). Rather than the water absorbed, BD was more influenced by the feed moisture content. Overall, higher product temperature caused more expansion which resulted in a less dense and more fragile product.

Extrusion Conditions

Extrusion conditions (coordinates of control variables) suitable for a snack-type product were determined (Table 14). Based on this fact, the response variables were either maximized or minimized. As can be seen from Table 14, the extrusion conditions for each response variable were different. As mentioned above, ER, BD, and shear stress were observed to be correlated to each other. As such, these properties along with color, were chosen to determine conditions for a snack-type product. A combined set of conditions was determined for these properties. Table 15 lists the properties and the combined extrusion conditions.

Table 14. Extrusion Conditions

Response Variable	Maximum/ Minimum	Moisture Content %	Protein Content %	Lactose/ Protein	Feed Rate gm/ min
Expansion Ratio	Maximum	20.00	8.01	0.395	55.0
Product Tempe-rature	Maximum	20.00	5.13	0.260	55.0
Bulk Density	Minimum	20.00	16.00	0.347	100.0
Color	Maximum	20.00	16.00	0.326	55.0
Shear Stress	Minimum	20.00	15.93	0.370	75.4
Viscosity	Maximum	27.50	3.00	0.260	55.0
Water Absorption Index	Maximum	22.50	3.00	0.260	100.0

Table 15. Combined Extrusion Conditions

Expansion Ratio	Bulk Density	Color	Shear Stress
Maximum	Minimum	Maximum	Minimum
Moisture Content %	Protein Content %	Lactose/ Protein	Feed Rate gm/min
20.00	16.00	0.347	55.00

Scanning Electron Microscopy

Extrudates with maximum or minimum value for one control variable and center point values for the remaining three control variables; extrudates with maximum or minimum value for all four control variables; and an extrudate with center point value for all four control variables were evaluated by scanning electron microscopy (SEM) for microstructure.

Figure 17 shows the micrograph for the extrudate produced with maximum moisture content (run 5, Table 4). As expected, the starch was not fully gelatinized due to the high moisture/low temperature cook. Also, due to the high moisture content the product had collapsed upon cooling and as such air cells were absent. At the lowest moisture level (run 12, Table 4; micrograph not shown) the air cells were more developed and a continuous matrix was observed,

Figure 17. Scanning electron micrograph of the extrudate produced with maximum moisture content (run 5, Table 4). The micron bar has length equal to 30 μm.

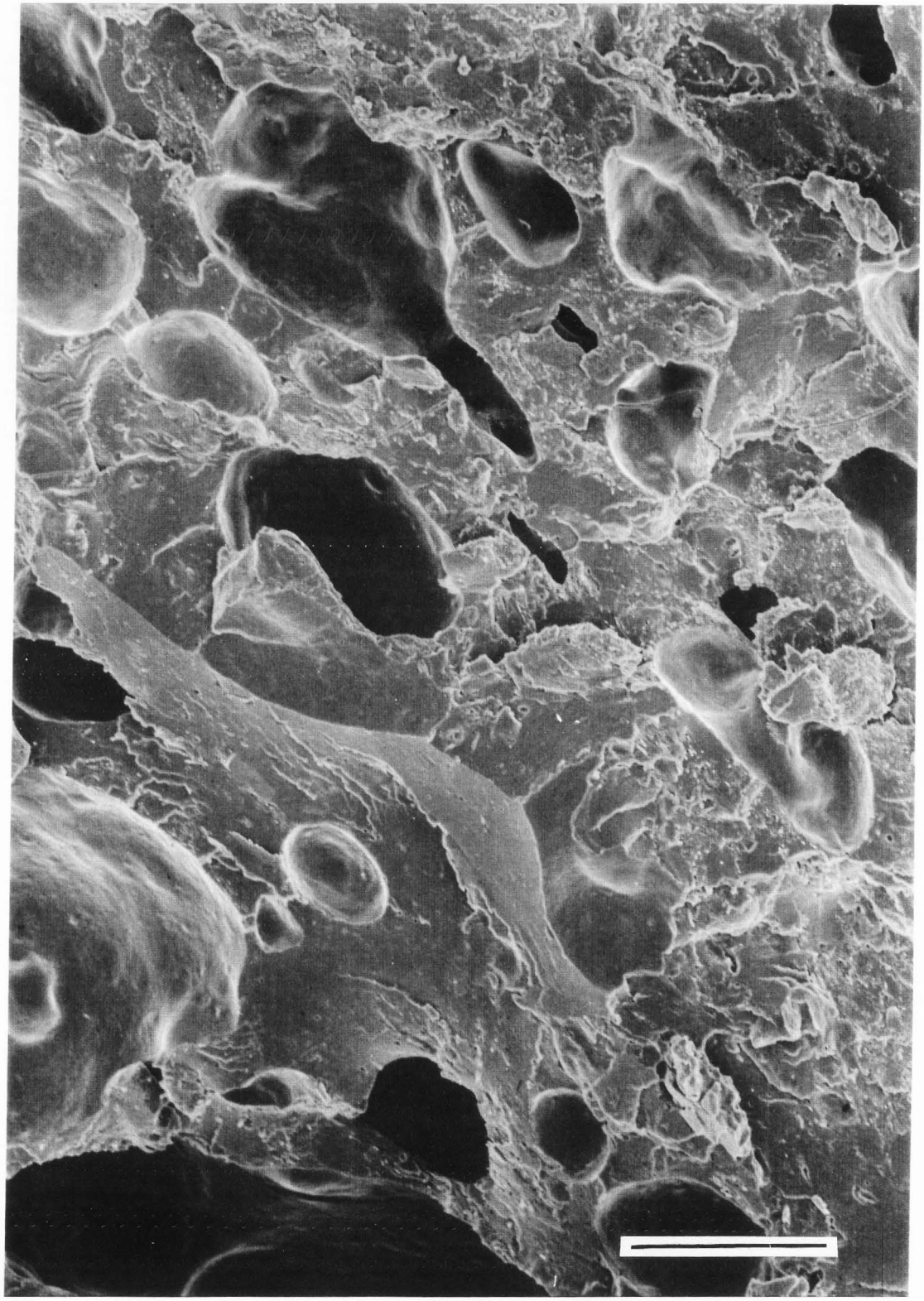
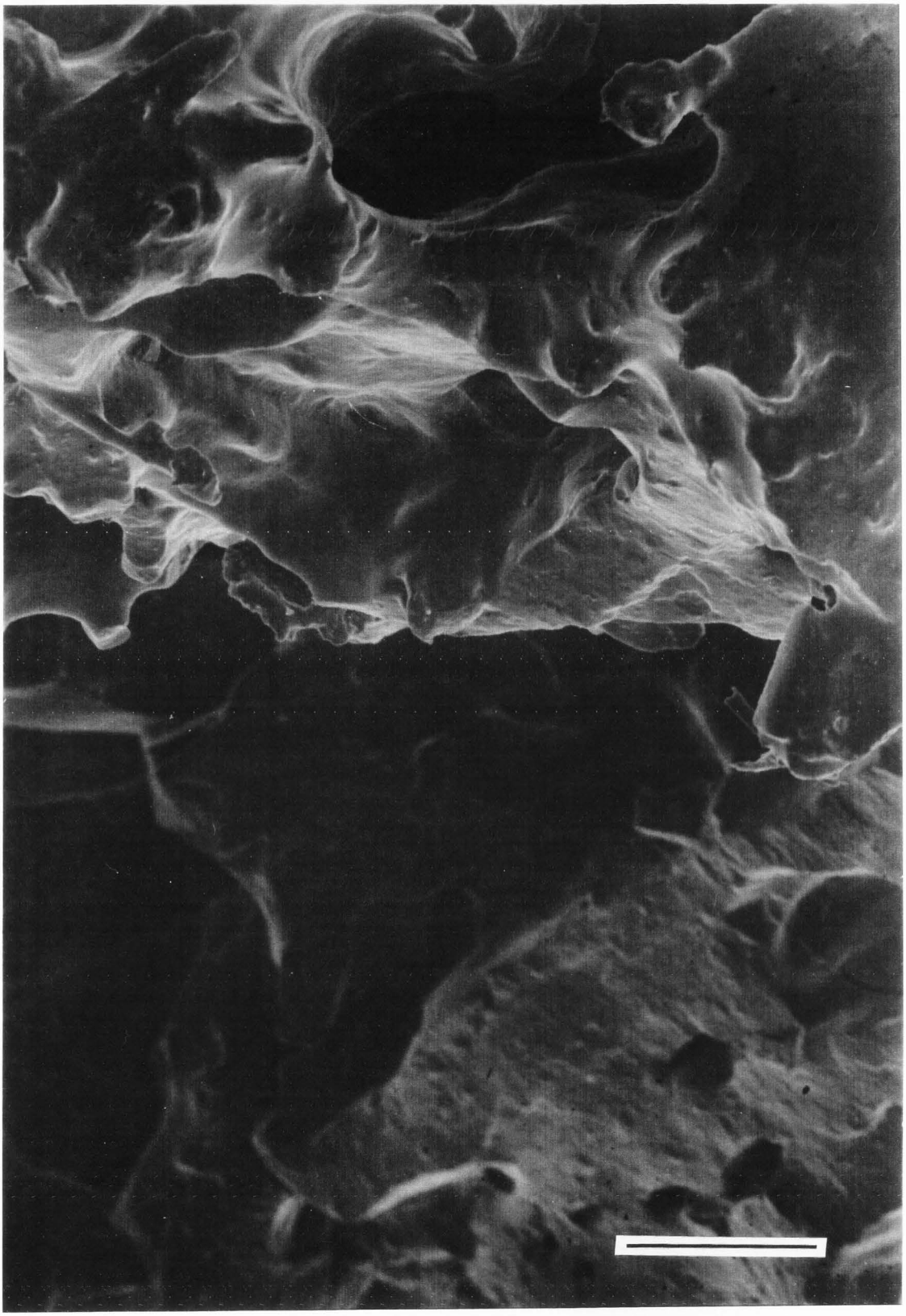


Figure 18. Scanning electron micrograph of the extrudate produced with minimum moisture content (run 5, Table 4). The micron bar has length equal to 30 μm.



indicating better starch gelatinization.

At the lowest protein level (Figure 18; run 2, Table 4) gelatinized starch was observed as compared to the extrudate with maximum protein content (run 7, Table 4; micrograph not shown). Both extrudates were compact dense structures, lacking any air cells. Similar observations were made regarding lactose/protein ratio. Figure 19 (run 23, Table 4) shows the micrograph for the extrudate with minimum lactose/protein ratio. At the highest feed rate (Figure 20; run 29, Table 4) starch was not fully gelatinized and air cells were absent. In contrast, at the lowest feed rate (run 13, Table 4; micrograph not shown) starch was more gelatinized and air cells were developed.

Extrudates produced with maximum values for all four control variables (Figure 21; run 3, Table 4) showed a discontinuous matrix. Complete starch gelatinization did not occur due to the high moisture content, protein content, and lactose/protein ratio. Also, the structure had collapsed upon cooling due to high elasticity because of high moisture content, resulting in a compact dense structure. At the lowest value for all four control variables (run 22, Table 4; micrograph not shown) a continuous and smooth starch matrix with air cells was observed.

Figure 22 shows the micrograph of the extrudate with center point values for all four control variables (run 1,

Figure 19. Scanning electron micrograph of the extrudate produced with minimum lactose/protein ratio (run 23, Table 4). The micron bar has length equal to 30 μm.

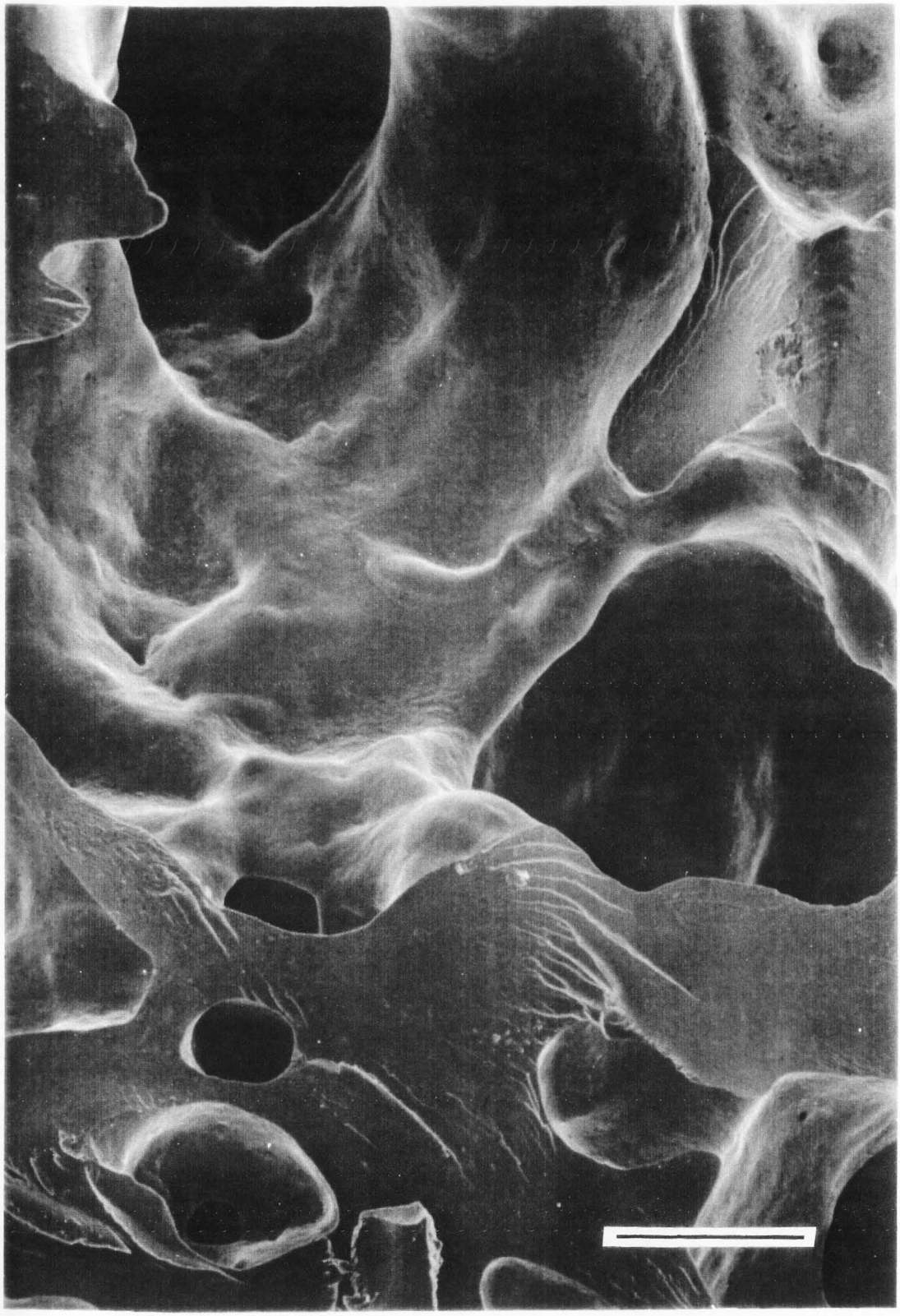


Figure 20. Scanning electron micrograph of the extrudate produced with maximum feed rate (run 29, Table 4). The micron bar has length equal to 30 μm.

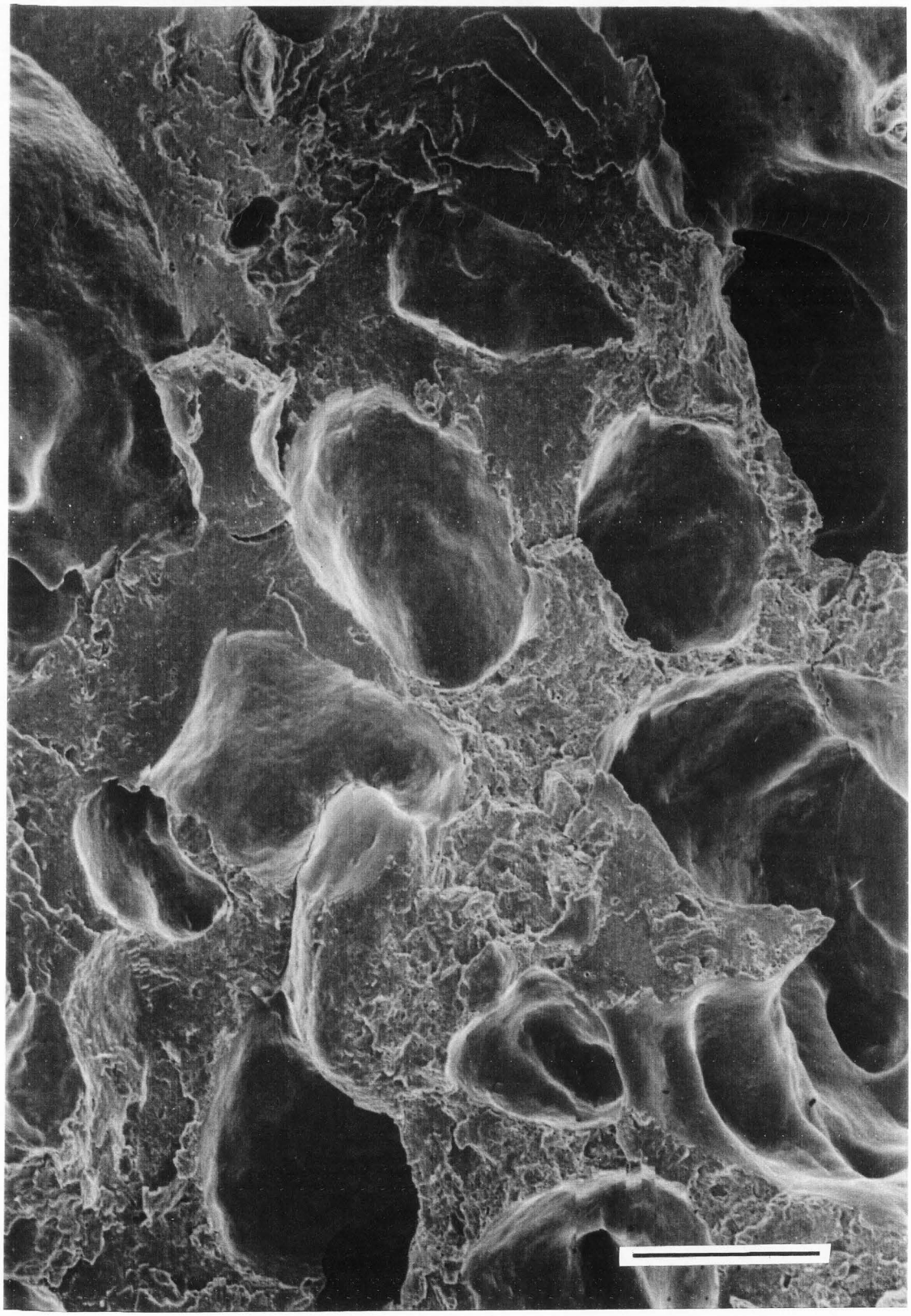


Figure 21. Scanning electron micrograph of the extrudate produced with maximum values for all four control variables (run 3, Table 4). The micron bar has length equal to 30 μm.

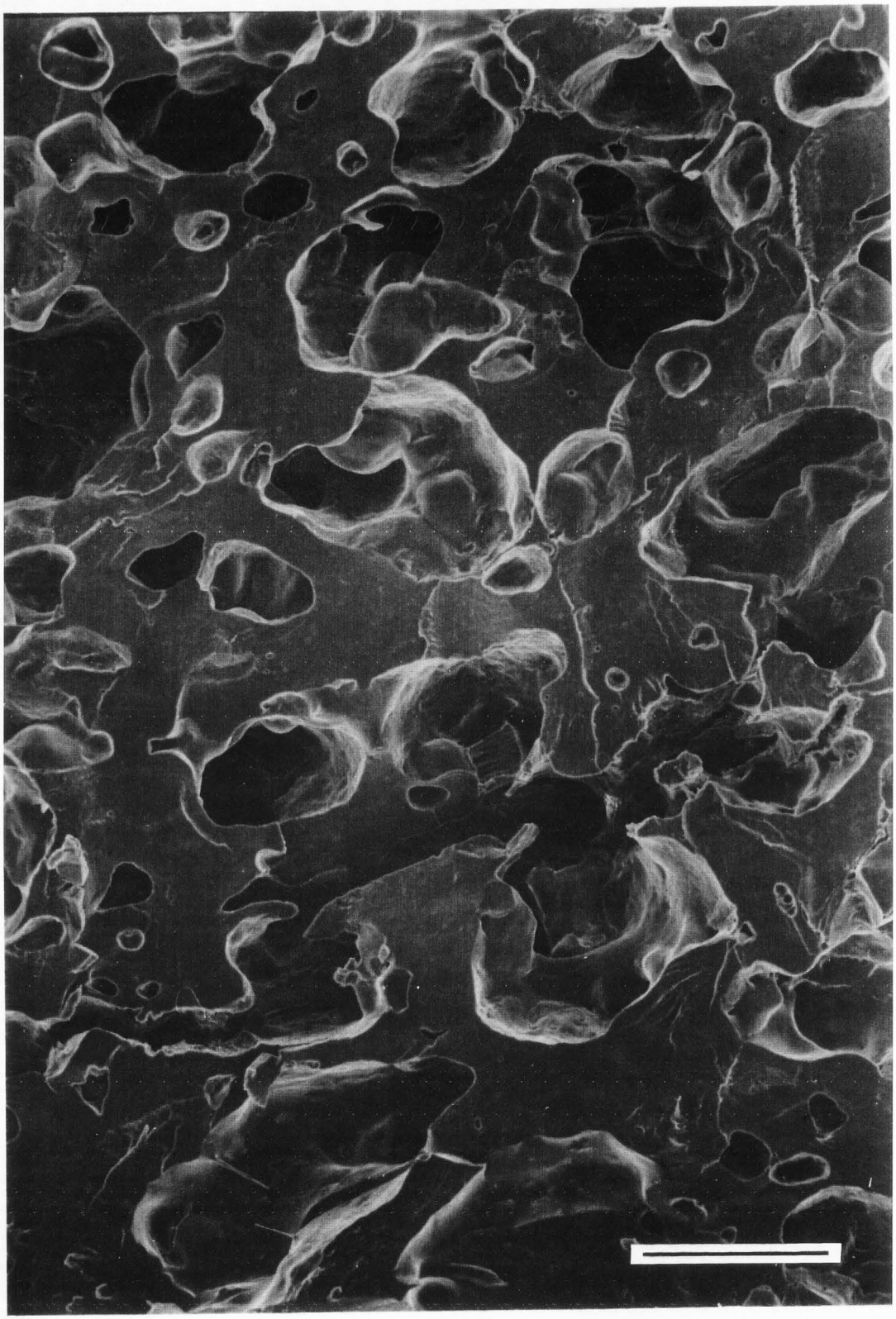


Figure 22. Scanning electron micrograph of the extrudate produced with center point values for all four control variables (run 1, Table 4). The micron bar has length equal to 30 μm.

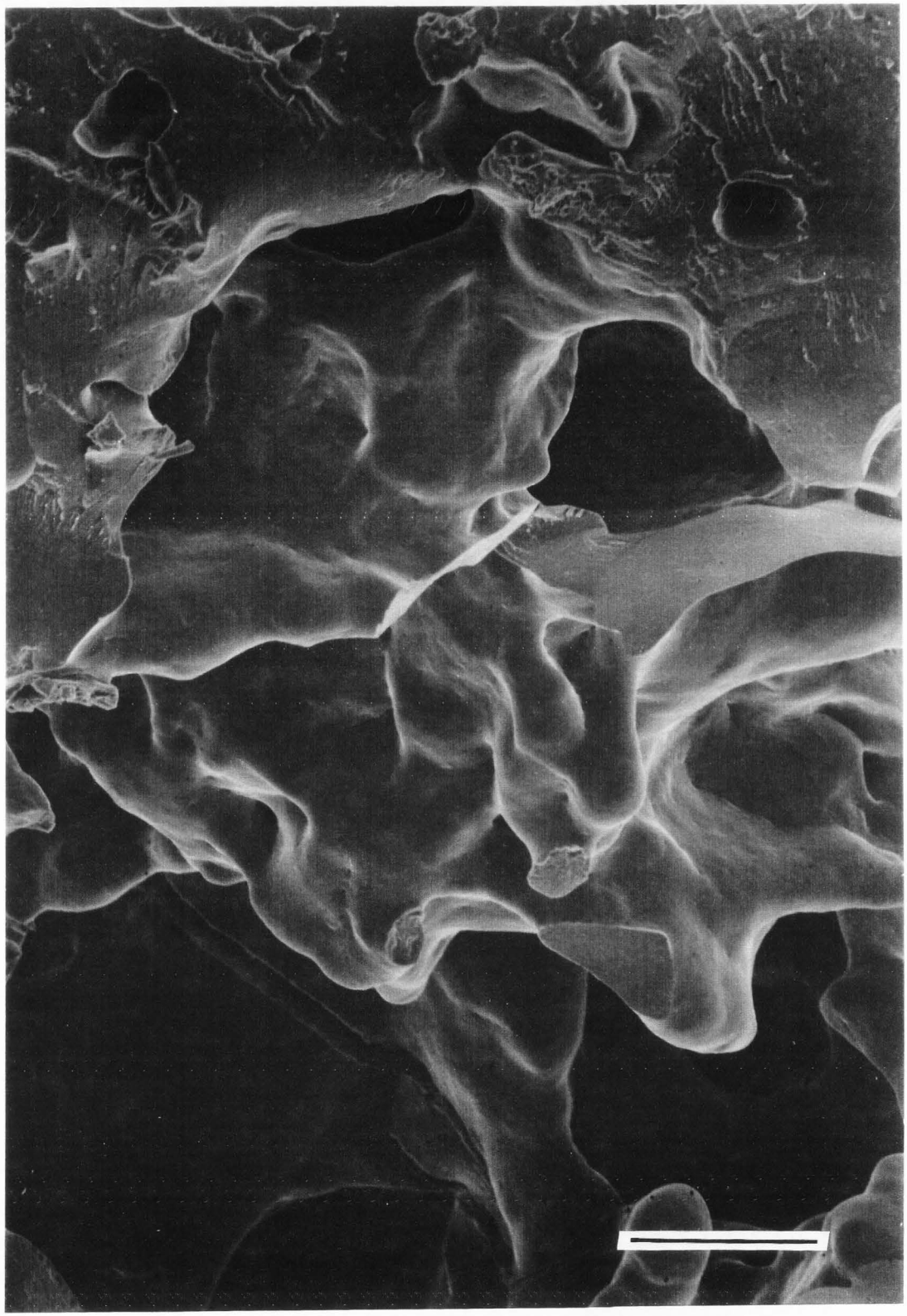


Table 4). The well gelatinized, smooth, continuous matrix of starch was due to maximum gelatinization of starch at 26% moisture content and low protein content and lactose/protein ratio. Air cells were absent, resulting in a dense and compact structure.

CONCLUSIONS

This study led to the following conclusions:

1. Moisture content had a significant linear effect on all the response variables and had a significant quadratic effect on expansion ratio, product temperature, color, viscosity, and water absorption index.

2. Protein content had a significant linear effect on product temperature, color, shear stress, viscosity, and water absorption index and had a significant quadratic effect on bulk density.

3. Lactose/protein ratio had a significant linear effect on product temperature, viscosity, and water absorption index and did not have a significant quadratic effect.

4. Feed rate did not have a significant linear or quadratic effect on any response variable.

5. Moisture content and feed rate had significant interaction effects on expansion ratio, color, and water absorption index. Moisture content and protein content had significant interaction effects on product temperature and viscosity. Moisture content and lactose/protein ratio and protein content and lactose/protein ratio had significant interaction effects on viscosity. Lactose/protein ratio and feed rate had a significant interaction effect on product temperature.

6. Expansion ratio, bulk density, and shear stress

were observed to be correlated. These properties along with color were chosen to determine extrusion conditions most likely to produce extrudates with properties suitable for a snack-type product. The combined set of extrusion conditions was 20% moisture content, 16% protein content, 0.347 lactose/protein ratio, and 55.0 gm/min feed rate.

REFERENCES

Adams, J.P. 1985. Statistical analysis in a food science and engineering laboratory course. Food Technol. 39(4):103.

Aguilera, J.M. and Kosikowski, F.V. 1976. Soybean extruded product: a response surface analysis. J. Food Sci. 41:647.

Aguilera, J.M. and Kosikowski, F.V. 1978. Extrusion and roll-cooking of corn-soy-whey mixtures. J. Food Sci. 43(1):225.

Aguilera, J.M., Rossi, F., Hiche, E., and Chichester, C.O. 1980. Development and evaluation of an extrusion-texturized peanut protein. J. Food Sci. 45:246.

Aguilera, J.M. and Stanley, D.W. 1986. Extrusion processing as applied to proteinaceous materials. In Food Engineering and Process Application. Unit Operation. Vol. 2. M. LeMaguer and P. Jelen (eds.), p.131. Elsevier Applied Science Publishers, New York, NY.

Anderson, R.A., Conway, H.F., Pfeifer, V.F., and Griffin, Jr., E.L. 1969. Gelatinization of corn grits by roll and extrusion cooking. Cereal Sci. Today 14(1):4.

Anderson, V.L. and McLean, R.A. 1974. Design of Experiments, p. 353. Marcel Dekker, Inc., New York, NY.

Anonymous. 1987. Spray Drying Nozzles and Applications. Technical Manual No. 402. Spraying Systems Co., Wheaton, IL.

AOAC. 1984. Official Methods of Analysis, 14th ed. Association of Official Analytical Chemists, Washington, DC.

Badrie, N. and Mellowes, W.A. 1991. Texture and microstructure of Cassava (Manihot esculenta Crantz) flour extrudate. J. Food Sci. 56(5):1319.

Badrie, N. and Mellowes, W.A. 1992. Soybean flour/oil and wheat bran effects on characteristics of cassava (Manihot esculenta Crantz) flour extrudate. J. Food Sci. 57(1):108.

Barraquio, V.L., Fichtali, J., Ng Kwai Hang, K.F., and

van de Voort, F.R. 1988. Acid coagulation of skim milk powder by extrusion processing. Can. Inst. Food Sci. Technol. 21:305.

Barraquio, V.L. and van de Voort F.R. 1991. Sodium caseinate from skim milk powder by extrusion processing: Physiochemical and functional properties. J. Food Sci. 56:1552.

Bhattacharya, M., Hanna, M.A., and Kaufman, R.E. 1986. Textural properties of extruded plant protein blends. J. Food Sci. 51(4):988.

Bhattacharya, S., Das, H., and Bose, A.N. 1988. Effect of extrusion process variables on in-vitro protein digestibility of fish-wheat flour blends. Food Chem. 28:225.

Bhattacharya, S., Das, H., and Bose, A.N. 1990. Effect of extrusion process variables on microstructure of blends of minced fish and wheat flour. J. Food Sci. Technol. 27(1):22.

Bisson, J.P., Bussiere, M., Fournet, G., and Jacquenod, P. 1986. Process for preparing an expanded dry product based on casein and use of this product. French Patent No. FR 2582192A1.

Bisson, J.P., Bussiere, M., Fournet, G., and Jacquenod, P. 1988. Preparation of a casein-based puffed product. United States Patent No. US 4744993.

Bjorck, I., Asp, N.G., and Dahlqvist, A. 1984. Protein nutritional value of extrusion-cooked wheat flours. Food Chem. 15:203.

Borgstrom, U. 1990. New method for the manufacture of caseinate. Nordisk Mejeriindustri. 17(6):285.

Boulle, M. 1987. A new caseinate manufacturing process for high concentration caseinate. In Extrusion Technology for the Food Industry. O'Conor (Ed.). p. 96. Elsevier Applied Science Publishers, New York, NY.

Burgess, L.D. and Stanley, D.W. 1976. A possible mechanism for thermal texturization of soybean protein. J. Inst. Can. Food Sci Technol. Aliment. 9(4):228.

Cheryan, M. 1986. Ultrafiltration Handbook. p. 235. Technomic Publ. Co., Inc., Lancaster, PA.

Chilton's Food Engineering. 1977. p. 99. Chilton Book Co., Chilton Way, Radnor, PA. p. 99.

Chinnaswamy, R. and Hanna, M.A. 1988. Relationship between amylose content and extrusion-expansion properties of corn starches. Cereal Chem. 65(2):138.

Chinnaswamy, R. and Hanna, M.A. 1990. Macromolecular and functional properties of native and extrusion-cooked corn starch. Cereal Chem. 67:490.

Clark, J.P. 1978. Texturization by extrusion. J. Text. Studies. 9:109.

Cleyn, R. and Trout, R. 1984. Enzymatic/ultraviolet method for measuring lactose in milk, a collaborative study. J. Assoc. Anal. Chem. 67:637-640.

Colonna, P., Doublier, J.L., Melcion, J.P., de Monredon, F., and Mercier, C. 1984. Extrusion cooking and drum drying of wheat starch. I: Physical and macromolecular modifications. Cereal Chem. 61:538.

Cuddy, M.E. and Zall, R.R. 1982. Performance of lipid-dried acid whey in extruded and baked products. Food Technol. 36(1):54.

Cumming, D.B., Stanley, D.W., and deMan, J.M. 1972. Texture-structure relationships in texturized soy protein. II. Textural properties and ultrastructure of an extruded soy bean product. Can. Inst. Food. Sci. Technol. 5:124.

Cumming, D.B., Stanley, D.W., and deMan, J.M. 1973. Fate of water soluble soy protein during thermoplastic extrusion. J. Food Sci. 38(2):320.

Dahl, S.R. and Villota, R. 1991. Twin-screw extrusion texturization of acid and alkali denatured soy proteins. J. Food Sci. 56(4):1991.

Danowski, K., Jakubowski, A. and Zgainski, J. 1979. Studies on the new extrusion-cooking process for manufacture of vegetable meat analogs. Proc. European Meeting of Meat Res. Workers, No. 25, 12.6:897.

Davidson, V.J., Paton, D., Diosady, L.L., and Larocque, G. 1984. Degradation of wheat starch in a single-screw extruder. Characteristics of extruded starch polymers. J. Food Sci. 49:453.

Diosady, L.L., Paton, D., Rosen, N., Rubin, L.J., and Athanassoulias, C. 1985. Degradation of wheat starch in a single-screw extruder: Mechanokinetic breakdown of cooked starch. J. Food Sci. 50:1697.

Downey, G. and Burgess, K.J. 1979. Modification of the aqueous solubility of edible fibers composed of casein and carrageenan. Irish J. Food Sci. Technol. 3(1):33.

Early, R. 1991. The Technology of Dairy Products. p. 167. VCH Publ. Inc., New York, NY.

Eerikainen, T. and Linko, P. 1989. Extrusion cooking modelling, control and optimization. In Extrusion Cooking. C. Mercier, P. Linko, and J.M. Harper, (Eds.). p. 157. American Assoc. of Cereal Chem., St. Paul, MN.

El-Dash, A.A., Gonzales, R., and Ciol, M. 1983. Response surface methodology in the control of thermoplastic extrusion of starch. J. Food Eng. 2:129.

Faubion, J.M. and Hoseney, R.C. 1982a. High-temperature short-time extrusion cooking of wheat starch and flour. I. Effect of moisture and flour type on extrudate properties. Cereal Chem. 59(6):529.

Faubion, J.M. and Hoseney, R.C. 1982b. High-temperature short-time cooking of wheat starch and flour. II. Effect of protein and lipid on extrudate properties. Cereal Chem. 59(6):533.

Faubion, J.M., Hoseney, R.C., and Serb, P.A. 1982. Functionality of grain components in extrusion. Amer. Assoc. of Cereal Chem. 27:212.

Fennema, O.R. 1985. Food Chemistry. p. 112. Marcel Dekker, Inc., New York, NY.

Fichtali, J. and van de Voort, F.R. 1990. Pilot plant production of caseins using extrusion processing. I. Acid casein production. Milchwissenschaft 45(9):560.

Fichtali, J., van de Voort, F.R., and Khuri, A.I. 1990. Multiresponse optimization of acid casein production. J. Food Proc. Eng. 12(4):247.

Fornal, L., Soral-Smietana, M., Smietana, Z., and Szpendowski, J. 1987. Chemical characteristics and physico-chemical properties of the extruded mixtures of cereal starches. Staerke. 39(3):75.

Gomez, M.H. and Aguilera, J.M. 1984. A physiochemical model for extrusion of corn starch. J. Food Sci. 49:40.

Guiraud, R.J.E. and Arnaud, M.R.C. 1977. Process for preparation of food products from aqueous suspensions of casein, with texturizing and stabilizing the milk proteins, and new products thus obtained. French Patent No. FR2333448.

Gujska, E. and Khan, K. 1991. Functional properties of extrudates from high starch fractions of navy and pinto beans and corn meal blended with legume high protein fractions. J. Food Sci. 56(2):431.

Harper, J.M. 1979. Food extrusion. Crit. Rev. Food Sci. Nutr. 11(2):155.

Harper, J.M. 1981. Extrusion of Foods. Volume 1 and 2. CRC Press, Boca Raton, FL.

Harper, J.M. 1986. Extrusion texturization of foods. Food Technol. 40(3):70.

Harper, J.M. 1989. Food extruders. In Extrusion Cooking. C. Mercier, P. Linko, and J.M. Harper, (Eds.). p. 1. Am. Assoc. of Cereal Chem., St. Paul, MN.

Harper, J.M., Rhodes, T.P., and Wanninger, L.A. Jr. 1971. Viscosity model for cooked cereal doughs. Chem. Eng. Prog. Symp. Series. 67(108):40.

Holay, S.H. and Harper, J.M. 1982. Influence of extrusion environment on plant protein texturization. J. Food Sci. 47:1869.

Hoseney, R.C., Zeleznak, K., and Abdelrahman, A. 1983. Mechanism of popcorn popping. J. Cereal Sci. 1:43.

Hsieh, F., Peng, I.C., and Huff, H.E. 1990. Effects of salt, sugar and screw speed on processing and product variables of corn meal extruded with a twin-screw extruder. J. Food Sci. 55(1):224.

Jones, S.A. 1989. Effects of extrusion cooking parameters on confectionary systems. In Progress in Extrusion Cooking. K. Paulus, and J.C. Cheftel, (Eds.). p. 126. Proc. of the COST 91 bis European workshop, Univ. of Montpellier, May 18-19, 1989. Bund fur Lebensmittelrecht und Lebensmittelkunde, Bonn.

Kim, C.H. and Maga, J.A. 1987. Properties of extruded whey protein concentrate and cereal flour blends. Lebensm. Wiss. Technol. 20:311.

Kinsella, J.E. 1978. Texturizing proteins: Fabrication, flavoring, and nutrition. CRC Crit. Rev. Food Sci. Nutr. 10(2):147.

Lai, C.S., Davies, A.B., and Hoseney, R.C. 1985a. The effect of yeast protein concentrate and some of its components on starch extrusion. Cereal Chem. 62:293.

Lai, C.S., Davies, A.B., and Hoseney, R.C. 1985b. Effects of whole yeast and various fractions on some properties of extruded starch. Cereal Chem. 62:423.

Lawton, B.T., Henderson, G.A., and Derlatka, E.J. 1972. The effects of extruder variables on the gelatinization of corn starch. Can. J. Chem. Eng. 50:168.

Lawton, J.W., Davis, A.N., and Behnke, K.C. 1985. High-temperature, short time extrusion of wheat gluten and a bran-like fraction. Cereal Chem. 62(4):267.

Ledward, D.A. and Mitchell, J.R. 1989. Protein extrusion-More questions than answers? In Food Structure-Its Creation and Evaluation. J.M.V. Blanshard, and J.R. Mitchell (Eds.). p. 219. Butterworths, London.

Lee, C.M. 1986. Fabricated seafood products: Surimi manufacture and fabrication of surimi based products. Food Technol. 40(3):115.

Linko, P., Kervinen, R., Karppinen, R., Rautalinna, E.K., and Vainionpaa, J. 1985. Extrusion cooking for cereal-based intermediate moisture products. In Properties of Water in Foods in Relation to Quality and Stability. D. Simatos, and J.L. Multon, (Eds.). p. 465. Martinus Nijhoff Publ., Dordrecht, Neth.

Li Sui Fong, J.C. 1980. Effects of extrusion cooking on the availability of lysine. Revue Agri. et Suc. de l'Ile Maurice. 59(2):63.

Maga, J.A. and Lorenz, K. 1978. Sensory and functional properties of extruded corn-soy blends. Lebenson. Wiss. Und-Tech. 11:185.

Maga, J.A. and Reddy, T. 1985. Coextrusion of carp (Cyprinus carpio) and rice flour. J. Food Proc. Pres. 9:121.

Mandigo, R.W. 1986. Restructuring of muscle foods. Food Technol. 40(3):85.

Martinez-Serna, M.D. and Villota, R. 1991. Reactivity, functionality, and extrusion performance of native and chemically modified whey proteins. In Food Extrusion Science and Technology. J.L. Kokini, C.T. Ho, and M.V. Karwe, (Eds.). p. 175. Marcel Dekker, Inc., NY.

Masters, K. 1985. Spray Drying Handbook. George Godwin. n.p.

Maurice, T.J., Burgess, L.D., and Stanley, D.W. 1976. Texture-structure relationships in texturized soy protein. III. Textural evaluation of extruded products. Can. Inst. Food Sci. Technol. 9(4):173.

Maurice, T.J. and Stanley, D.W. 1978. Texture-structure relationships in texturized soy protein. IV. Influence of process variables on extrusion texturization. Can. Inst. Food Sci. Technol. 11(1):1.

Mercier, C. and Feillet, P. 1975. Modification of carbohydrate components by extrusion-cooking of cereal products. Cereal Chem. 52:283.

Meuser, F., Van Lengerich, B., and Reimers, H. 1984. Kochextrusion von Staerken. Vergleich experimenteller Ergebnisse zwischen Laborextrudern und Produktionsextrudern mittels Systemanalyse. Staerke. 36:194.

Millauer, C. and Weidmann, W.M. 1984. Einstaz von Extrudern in der Staerke-Industrie. Staerke. 36:228.

Montgomery, D.C. 1976. Design and Analysis of Experiments. p. 340. John Wiley and Sons, New York, NY.

Mulvaney, S. 1993. Personal communication. Cornell University, Ithaca, NY.

Murray, B.P. and Stanley, D.W. 1980. Improved utilization of fish protein - co-extrusion of mechanically deboned salted minced fish. Can. Inst. Food Sci. Technol. 13(3):125.

Mustakes, G.C., Albrecht, W.J., Bookwalter, G.N., McGhee, J.E., Kwolck, W.F., and Griffin, E.L. 1970. Extruder-processing to improve nutritional quality, flavor and keeping quality of full-fat soy flour. Food Technol. 25:1290.

Neumann, P.E., Jasberg, B.K., Wall, J.S., and Walker, C.E. 1984. Uniquely textured products obtained by co-extrusion of corn gluten meal and soy flour. Cereal Chem. 61:438.

Nicklason, P.M. and Pigott, G.M. 1989. Simultaneously heating, forming and extruding of surimi-based products. J. Food Eng. 9:219.

Noguchi, A. 1989. Extrusion cooking of high-moisture foods. In Extrusion Cooking. C. Mercier, P. Linko, and J.M. Harper, (Eds.). p. 91. Am. Assoc. of Cereal Chem., St. Paul, MN.

Olkku, J. and Vainionpaa, J. 1980. Response surface analysis of HTST extrusion texturized starch-protein-sugar paste. In Food Process Engineering. Vol. 1, Food Processing Systems. P. Linko, Y. Malkki, J. Olkku, and J. Larinkari, (Eds.). p. 821. Applied Science Publ., London.

Owusu-Ansah, J., van de Voort, F.R., and Stanley, D.W. 1983. Physiochemical changes in corn starch as a function of extrusion variables. Cereal Chem. 60:319.

Park, J., Rhee, K.S., Kim, B.K., and Rhee K.C. 1993. Single-screw extrusion of defatted soy flour, corn starch and raw beef blends. J. Food Sci. 58(1):9.

Peri, C., Barbieri, R., and Casiraghi, E.M. 1983. Physical, chemical and nutritional quality of extruded corn germ flour and milk protein blends. J. Food Technol. 18:43.

Pham, C.B. and Del Rosario, R.R. 1984. Studies on the development of textured vegetable products by the extrusion process. I. Effects of processing variables on protein properties. J. Food Technol. 19:535.

Philips, R.D., Chinnan, M.S., and Kennedy, M.B. 1984. Effect of feed moisture and barrel temperature on physical properties of extruded cowpea meal. J. Food Sci. 49:916.

Queguiner, C., Eliane Dumay, C., Cavalier-Salou, C., and Cheftel, J.C. 1989. Reduction of Streptococcus thermophilus in a whey protein isolate by low moisture extrusion cooking without loss of functional properties. Int. J. Food Sci. Technol. 24:601.

Queguiner, C., Eliane Dumay, C., Cavalier-Salou, C., and Cheftel, J.C. 1991. Applications of extrusion cooking to dairy products: Preparation of fat analogues by microcoagulation of whey proteins. In Food Extrusion Science and Technology. p. 373. J.L. Kokini, C.H. Ho, and M.V. Karwe, (Eds.). Marcel Dekker, Inc., New York, NY.

Racicot, W.F., Satterlee, L.D., and Hanna, M.A. 1981. Interaction of lactose and sucrose with cornmeal protein during extrusion. J. Food Sci. 46:1500.

Reinikainen, P., Suortti, T., Olkku, J., Malkki, Y., and Linko, P. 1986. Extrusion cooking in enzymatic liquefaction of wheat starch. Staerke. 38:20.

Rhee, K.C., Kuo, C.K., and Lusas, E.W. 1981. Texturization. In Protein Functionality in Foods. J.P. Cherry, (Ed.). ACS Symposium Series. Washington, DC. 147:51.

Rossi, M. and Perri, C. 1986. Effect of extrusion cooking on the structural and functional characteristics of sunflower protein. In Food Engineering and Process Application. Unit Operation. Vol. 2. M. LeMaguer, and P. Jelen, (Eds.). p. 197. Elsevier Applied Science Publishers, New York, NY.

Schroeder, K. 1982a. Protein-based snack and process for its production. German Federal Republic Patent No. DE 3039348A1.

Schroeder, K. 1982b. Foamed proteinaceous snack product. UK Patent No. 2085273A.

Simonsky, R.W. and Stanley, D.W. 1982. Texture-structure relationships in texturized soy protein. IV. Influence of pH and acylation on extrusion texturization. Can. Ist. Food Sci. Technol. 15(4):294.

Singh, R.K., Nielsen, S.S., Chambers, J.V., Martinez-Serna, M., and Villota, R. 1991. Selected characteristics of extruded blends of milk protein raffinate or nonfat dry milk with corn flour. J. Food Process. Preserv. 15:285.

Skierkowski, K., Gujska, E., and Khan, K. 1990. Instrumental and sensory evaluation of textural properties of extrudates from blends of high starch/high protein fractions of dry beans. J. Food Sci. 55(4):1081.

Stanley, D.W. 1989. Protein reactions during extrusion cooking. In Extrusion Cooking. C. Mercier, P. Linko, and J.M. Harper, (Eds.). p. 321. American Association of Cereal Chemists, St. Paul, MN.

Stanley, D.W. and deMan, J.M. 1978. Structural and mechanical properties of textured protein. J. Text. Studies. 9:59.

Szpendowski, J., Smietana, Z. and Zuraw, J. 1983. Production of extrusion texturized protein preparations. Milchwissenschaft. 38(10):577.

Tadmor, Z. and Klein, I. 1970. Engineering Principles of Plasticating Extrusion. Van Nostrand Reinhold Co., p. 450. New York, NY.

Taranto, M.V., Cegla, G.F., and Rhee, K.C. 1978. Morphological, ultrastructural, and rheological evaluation of soy and cottonseed flours texturized by extrusion and non extrusion process. J. Food Sci. 43:973.

Tossavainen, O., Hakulin, S., Kervinen, R., Myllymaeki, O., and Linko, P. 1986. Neutralization of acid casein in a twin-screw cooking extruder. Lebensm-Wiss. und Technol. 19(6):443.

Tuohi, J.J., Burgen, K.J., and Lambert, I. 1979. A comparison of the texture of expanded milk protein extenders and TVP. J. Food Technol. 14:473.

van de Voort, F.R., Stanley, D.W., and Edamura, R. 1984. Improved utilization of dairy proteins: coextrusion of casein and wheat flour. J. Dairy Sci. 67(4):749.

Van Zuilichem, D.J., Stolp, W., and Witham, I. 1977. Texturization of soya with a single-screw extruder. Presented at European Seminar on Extrusion Cooking of Foods, Center de Perfectionnement des Cadres des Industries Agricoles at alimentaire, Paris, France.

Wagner, J. and Sillard, M. 1989. Making caseinate by extruder. Food Eng. Int. 14(2):42.

Wen, W.F., Rodis, P., and Wasserman, B.P. 1990. Starch fragmentation and protein insolubilization during twin-screw extrusion of corn meal. Cereal Chem. 67:269.

Wiedmann, W. and Millauer, C. 1985. A process for producing caseinate. German Federal Republic Patent No. DE 3340116A1.

Williams, M.A., Horn, R.E., and Rugula, R.P. 1977. Extrusion: An in-depth look at a versatile process. J. Food Eng. 49(9):99.

Yagi, N., Kim, K.Y., and Nakaji, T. 1986. Process for preparing heat resistant milk protein having good processability and/or acid and base resistance, and a food product containing such protein. European Patent No. EP 0174192A2.

Zuber, F., Megard, D. and Cheftal, J.C. 1987. Continuous emulsification and gelation of dairy ingredients by HTST extrusion cooking: production of processed cheeses. Int. J. Food Sci. Technol. 22(6):607.

APPENDICES

Appendix 1. Experimental data for response variables

Run #	Trial #	ER	PT	BD	Color (b*)	Sh.St.	Vis. (cp)	WAI	Color (L*)
1	1	1.45	111.17	0.57	3.08	0.73	3432.0	6.03	38.52
2	4	1.37	110.62	0.50	3.19	1.18	7176.0	7.51	40.12
3	10	1.42	79.66	0.39	5.62	0.58	920.4	3.02	41.91
4	1	1.55	112.24	0.50	2.98	0.83	3354.0	6.18	40.20
5	3	1.57	88.38	0.56	4.47	0.93	1341.6	4.41	41.93
6	20	1.59	88.44	0.50	2.64	2.06	3237.0	5.63	39.52
7	5	1.73	104.52	0.34	6.85	0.28	1521.0	4.81	33.88
8	1	1.54	111.53	0.58	3.20	0.71	2964.0	5.87	37.88
9	21	2.17	112.50	0.36	6.45	0.27	2706.6	6.40	26.17
10	17	1.76	114.14	0.28	4.74	0.27	3705.0	7.34	31.17
11	1	1.37	109.80	0.55	3.23	0.87	3120.0	6.12	39.33
12	2	2.40	119.39	0.38	8.55	0.16	1365.0	6.09	27.28
13	8	1.84	106.91	0.40	4.54	0.27	2652.0	5.72	28.83
14	18	1.25	81.13	0.51	4.17	1.34	858.0	3.22	40.56
15	22	1.54	93.33	0.52	4.45	0.85	819.0	4.15	39.75
16	11	1.70	109.10	0.32	6.89	0.19	1466.4	5.85	33.17
17	1	1.35	109.14	0.51	3.04	0.78	4227.6	6.43	39.48
18	15	1.89	114.49	0.25	7.56	0.25	834.6	6.13	31.41
19	12	1.54	99.59	0.57	2.89	1.43	3135.6	4.25	41.95
20	23	2.20	117.18	0.38	13.35	0.09	897.0	5.31	27.76
21	7	1.47	105.27	0.53	3.15	0.58	2301.0	5.71	36.74
22	25	2.21	122.17	0.38	6.51	0.36	2379.0	6.40	24.81
23	6	1.44	110.47	0.52	3.95	1.09	4368.0	6.31	39.50
24	1	1.38	110.60	0.58	3.40	0.77	4110.6	5.92	38.89
25	19	2.01	113.70	0.46	8.23	0.22	756.6	5.22	26.56
26	13	1.98	121.91	0.30	4.75	0.34	2324.4	7.24	30.04
27	24	1.55	103.80	0.53	2.64	1.49	7433.4	6.25	37.85
28	16	1.80	97.99	0.59	3.45	1.13	7285.2	5.10	41.33
29	9	1.52	112.22	0.54	3.88	1.47	4875.0	6.43	40.76
30	14	1.58	85.26	0.46	5.87	0.72	975.0	4.45	41.09

ER : Expansion Ratio

PT : Product Temperature (°C)

BD : Bulk Density (gm/ml)

Sh. St : Shear Stress (lbf/mm^2)

WAI : Water Absorption Index

Vis. : Viscosity (cp)

Appendix 2. Analysis of variance tables for response variables.

ANOVA table for Expansion Ratio

Mean Squares	DF	P	
0.784831	2	0.0000	moisture content
0.0129406	2	0.6156	protein content
0.0537571	2	0.1592	lactose/protein
0.0426042	2	0.2251	feed rate
0.00855624	1	0.5734	moisture content*protein content
0.0138063	1	0.4759	moisture content*lactose/protein
0.174306	1	0.0202	moisture content*feed rate
0.0390063	1	0.2380	protein content*lactose/protein
5.62508e-005	1	0.9634	protein content*feed rate
7.01492e-005	1	0.9591	lactose/protein*feed rate
0.0258213	15		ERROR
0.00776	5		REPLICATE ERROR

ANOVA table for Temperature

Mean Squares	DF	P	
1457.23	2	0.0000	moisture content
147.744	2	0.0002	protein content
59.8505	2	0.0093	lactose/protein
0.140501	2	0.9849	feed rate
73.0597	1	0.0130	moisture content*protein content
27.014	1	0.1074	moisture content*lactose/protein
0.182754	1	0.8899	moisture content*feed rate
7.57627	1	0.3788	protein content*lactose/protein
34.6626	1	0.0714	protein content*feed rate
85.9205	1	0.0080	lactose/protein*feed rate
9.20991	15		ERROR
1.30438	5		REPLICATE ERROR

ANOVA table for Bulk Density

Mean Squares	DF	P	
0.0651989	2	0.0000	moisture content
0.0122461	2	0.0420	protein content
0.00149819	2	0.6265	lactose/protein
0.00449715	2	0.2660	feed rate
0.01	1	0.0929	moisture content*protein content
0.0049	1	0.2283	moisture content*lactose/protein
0.009025	1	0.1089	moisture content*feed rate
0.000899999	1	0.5982	protein content*lactose/protein
0.011025	1	0.0791	protein content*feed rate
0.000107027	1	0.8552	lactose/protein*feed rate
0.00310529	15		ERROR
0.00125667	5		REPLICATE ERROR

ANOVA table for Color

Mean Squares	DF	P	
33.4692	2	0.0000	moisture content
19.2113	2	0.0000	protein content
2.67353	2	0.0770	lactose/protein
1.63457	2	0.1884	feed rate
1.61926	1	0.1937	moisture content*protein content
1.41016	1	0.2235	moisture content*lactose/protein
13.1951	1	0.0015	moisture content*feed rate
2.03776	1	0.1477	protein content*lactose/protein
0.213906	1	0.6281	protein content*feed rate
1.13604	1	0.2722	lactose/protein*feed rate
0.874458	15		ERROR
0.02343	5		REPLICATE ERROR

ANOVA table for Shear Stress

Mean Squares	DF	P	
2.00807	2	0.0000	moisture content
0.447155	2	0.0184	protein content
0.0259422	2	0.7406	lactose/protein
0.0783149	2	0.4181	feed rate
0.283556	1	0.0872	moisture content*protein content
0.0855562	1	0.3308	moisture content*lactose/protein
0.247506	1	0.1079	moisture content*feed rate
0.0115562	1	0.7170	protein content*lactose/protein
0.00390625	1	0.8328	protein content*feed rate
0.0343871	1	0.5336	lactose/protein*feed rate
0.08468	15		ERROR
0.00361667	5		REPLICATE ERROR

ANOVA table for Viscosity

Mean Squares	DF	P	
8.66651e+006	2	0.0008	moisture content
2.66574e+007	2	0.0000	protein content
3.28645e+006	2	0.0286	lactose/protein
585572	2	0.4634	feed rate
6.70603e+006	1	0.0082	moisture content*protein content
3.8025e+006	1	0.0367	moisture content*lactose/protein
164511	1	0.6402	moisture content*feed rate
6.09448e+006	1	0.0109	protein content*lactose/protein
1840.41	1	0.9604	protein content*feed rate
29246.3	1	0.8433	lactose/protein*feed rate
722919	15		ERROR
270529	5		REPLICATE ERROR

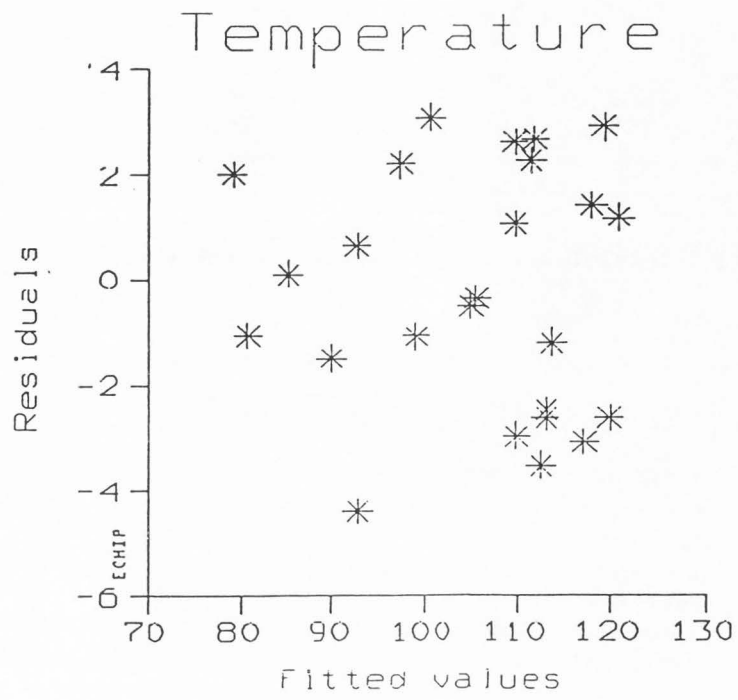
ANOVA table for Water Absorption Index

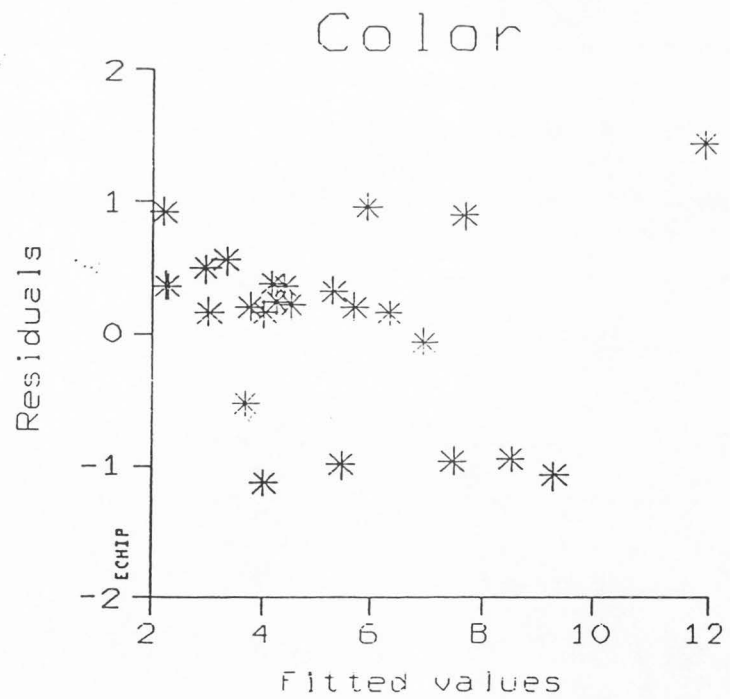
Mean Squares	DF	P	
7.6052	2	0.0000	moisture content
5.42436	2	0.0000	protein content
0.683076	2	0.0126	lactose/protein
0.0606772	2	0.6006	feed rate
0.1444	1	0.2801	moisture content*protein content
0.7056	1	0.0257	moisture content*lactose/protein
2.00223	1	0.0008	moisture content*feed rate
0.0841	1	0.4060	protein content*lactose/protein
0.330625	1	0.1107	protein content*feed rate
0.0585154	1	0.4866	lactose/protein*feed rate
0.115025	15		ERROR
0.0410966	5		REPLICATE ERROR

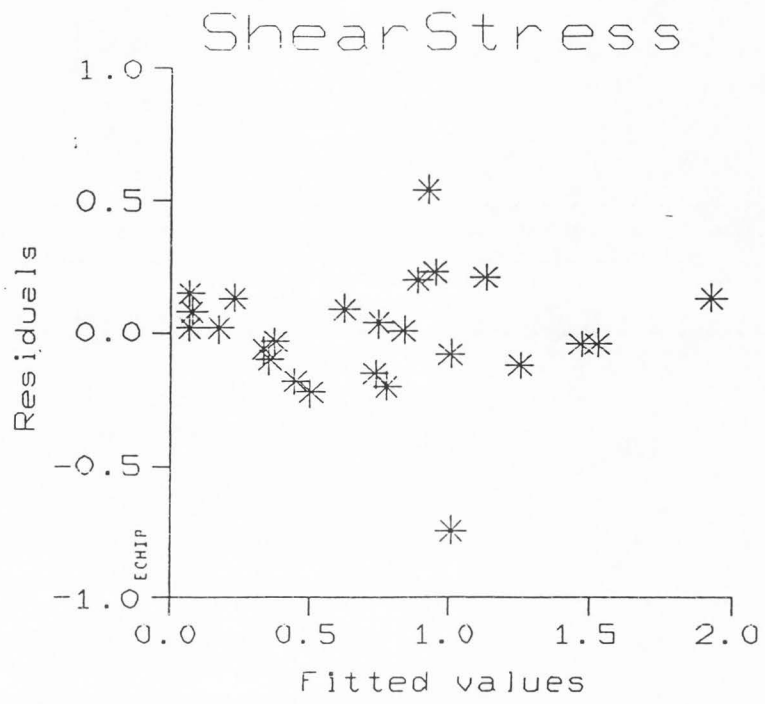
Appendix 3. Regression coefficients for response variables

Coefficients	Expansion Ratio	Product Temperature	Bulk Density	Color	Shear Stress	Viscosity	Water Absorption Index
b_0	1.5145	109.696	0.5245	3.6851	0.7485	3470.33	6.0756
b_1	-0.0498	-2.5222	0.0169	-0.3425	0.0931	106.34	-0.1722
b_2	-0.0055	-0.6221	-0.0032	0.22	-0.0343	-259.267	-0.1193
b_3	-0.2235	-14.312	0.0024	-2.2179	0.3107	-3417.27	-1.5877
b_4	-0.0027	-0.0054	-0.0008	-0.019	-0.0019	8.5757	0.0036
b_{12}	-0.0007	-0.0657	-0.0008	-0.0098	-0.0041	-19.92	-0.0029
b_{13}	-0.0336	-1.485	-0.02	0.3393	0.0836	-557.143	-0.24
b_{14}	0.0009	0.0009	0.0002	0.0081	-0.0011	-0.9013	-0.0031
b_{23}	-0.0434	-0.6049	0.0066	-0.3137	-0.0236	542.571	-0.0637
b_{24}	1.3e-05	-0.0101	-0.0002	-0.0008	0.0001	0.0733	0.0009
b_{34}	0.0005	0.5790	-0.0006	0.0666	-0.0116	-10.6818	-0.0151
b_{11}	0.0162	-0.1730	-0.0011	0.0904	-0.0081	-84.1722	-0.0328
b_{22}	-0.0007	-0.0151	-0.0018	0.0182	-0.0004	21.0863	0.0021
b_{33}	-6.2838	-51.085	1.2732	-34.971	-0.7522	-7706.38	-0.3317
b_{44}	0.0001	9.3e-05	-7.4e-05	0.0001	0.0004	0.9103	7.5e-05

Appendix 4. Residuals versus fitted values plots.



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-1.0
Residuels
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2.0
Fitted values