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QUANTIFYING REACTIONS INFLUENCING QUALITY OF FOODS: TEXTURE, FLAVOR AND APPEARANCE[1]

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ABSTRACT

For engineers to maintain and improve processes applied to foods, it is necessary to quantify changes in food quality as a function of process parameters. Texture, flavor and appearance (color) are sensory properties which generally have been described qualitatively and only recently have quantitative models been developed and applied. This paper reviews the application of sensory analysis for generating quantitative data on the effect of processing on texture. Generally there is a lack of data on the effect of operational parameters on texture, and, in many cases, the data which have been generated were not correctly analyzed. The requirement that engineers designing machines or processes for foods need to develop a better appreciation for the rheological properties of foods is emphasized. Finally, a systematic approach to quantifying effects of processing on texture of food must be used if food processors are to design processes based on consumer appeal.

INTRODUCTION

Texture, flavor and appearance are perhaps the most important characteristics of foods because they are attributes the consumer can readily assess. Although the consumer is becoming increasingly aware of attributes such as nutritional content (including presence of toxic factors) and microbiological quality, these attributes are more difficult to assess, quantify and use as a basis upon which to distinguish between competing products. As engineers designing

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machines and processes for the food industry, we are forced to consider the impact of operations on quality attributes. Frequently development of operations (machine and process design) is a trial and error procedure because there is a lack of quantitative data on the effects of process parameters on quality attributes.

Perhaps the most widely studied quality attributes as a class are nutrients. Quantitative data generated to yield reaction rates at a reference temperature and a temperature dependence parameter (either Z-value or E_a; see Lund 1975) make it possible to determine the time/temperature treatment which should be applied to canned foods yielding the maximum retention of nutrients while fulfilling a design criterion for destruction of spores of *C. botulinum* (Lund 1977; Teixeira *et al.* 1975; Saguy and Karel 1979; Martens 1980; Hildenbrand 1980; Ohlsson 1980a). There are two obvious reasons why nutrients have been widely studied: (1) nutrients are important for maintenance of health. Since the main objective of the food processing sector should be the supply of an adequate quantity of required nutrients, it has been necessary to ascertain the effects of processing on nutrients, and (2) many nutrients can be assessed objectively with instrumental methods. This allows quantification of the effects of processing and ultimately the design of processes to maximize nutrients.

The quality attributes which are considered in this paper, however, have generally not received the same success in quantification as nutrients. There are many reasons for this including the observation that "food quality is in the mind of the observer." Response to food quality may be a conditioned response and is subject to many variables outside the control of the food processor. However, progress is being made in quantifying the response of texture, flavor and appearance of foods to process parameters. Eventually our knowledge in this area will allow us to design processes to achieve any desired set of quality attributes.

Use of Sensory Analysis

Very often sensory analysis (taste panels) are used to assess texture, flavor and appearance of food products as a function of type or severity of process. There are well established procedures for conducting sensory analysis in order to generate reliable information.

Recently Moskowitz (1981) and Trant *et al.* (1981) demonstrated a potential problem in correlating hedonic responses (like/dislike) from taste panels with physical and chemical measurements of food

quality. The problem arises in that physical and chemical measurements are usually linearly correlated to intensity whereas panel assessment based on hedonic response is nonlinear with intensity. Figure 1 is an example from Trant *et al.* (1981) on the sweetness of lemonade as a function of level of added sucrose. The objective measurement (refractive index) is linearly related to intensity (added sucrose) and is nearly paralleled by the intensity response from the trained panel. The hedonic response, however, clearly demonstrates a nonlinear (in fact, quadratic) response to intensity. Moskowitz (1981) suggested using a quadratic equation to determine "liking" if hedonic responses are determined. This model should contain all first and second order terms (including cross products when more than one attribute is measured with hedonic responses). Trant *et al.* (1981) also reemphasized the necessity of examining the data from each judge as well as data on the arithmetic mean of the panel. In some instances

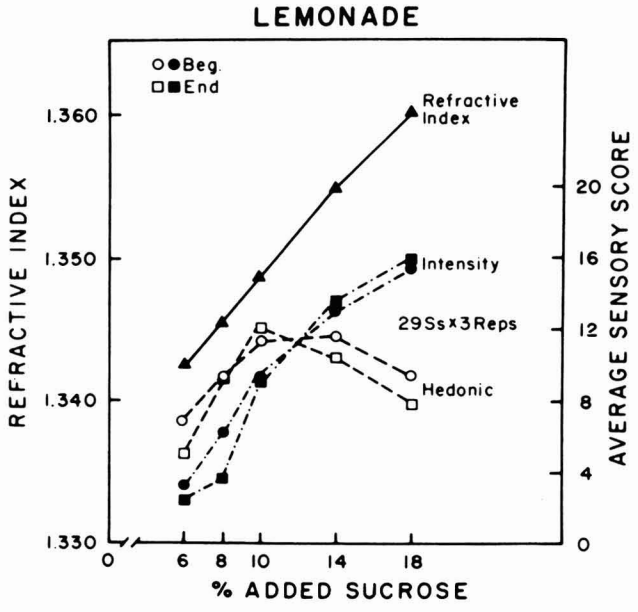

FIG. 1. AVERAGE SWEETNESS INTENSITY

(17 = extremely sweet; 1 = no sweetness) and average hedonic response (17 = like extremely; 1= dislike extremely) as a function of added sucrose in lemonade, at the beginning and end of the 7-wk experiment (29 judges, 3 replications), compared to refractive indices. Reprinted from *Journal of Food Science 46*, 583 (Trant *et al.* 1981)

quantitative data from sensory analysis has been an artifact of arithmetic (Pangborn 1980). Statistical analysis including examination of individual responses should be applied to sensory analysis just as it is applied to instrumental analysis.

Perhaps the most widely studied parameters are temperature and time of processing on quality attributes. For example, two studies in which sensory analysis was used to generate reaction rate and temperature-dependence parameters are those by Ohlsson (1980b) and Hayakawa *et al.* (1977). Ohlsson (1980b) investigated quality attributes of minced products including fish, liver, beef, vegetables, tomato sauce and vanilla sauce as affected by thermal processing. Sensory analysis was performed for appearance, odor, taste and off-taste by a trained panel using an 8 point intensity rating for each attribute. An example of the data generated by this procedure is presented in Fig. 2a on appearance of sterilized fish pudding. A linear response be-

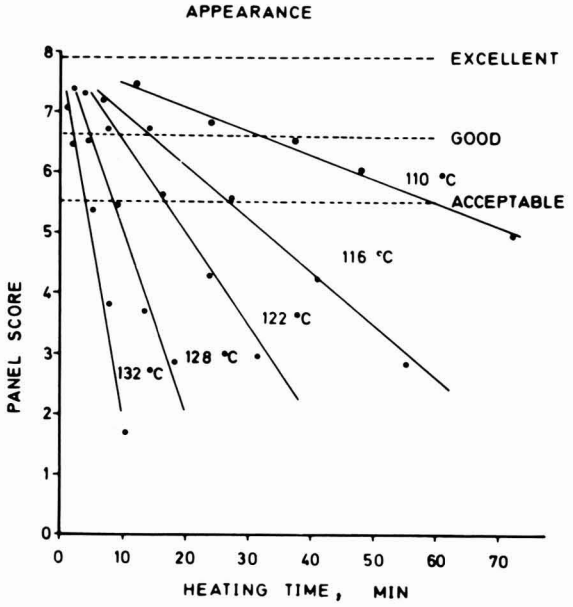

FIG. 2a. APPEARANCE OF STERILIZED FISH PUDDING

Average values and linear regression line of the panel scores plotted for each temperature against the corrected heating time. The panel score levels determined to correspond to excellent, good, and acceptable appearance are also given. Reprinted from *Journal of Food Science 45*, 836 (Ohlsson 1980b) Copyright © by Institute of Food Technologists.

tween panel score and heating time was obtained, and in an independent test the panel determined the score for an excellent, good and acceptable product. Ohlsson used the time at each temperature for the response to go from "good" to "acceptable" as a measure of the "time constant" for the reaction. The temperature dependence of time constant (given the symbol D) was determined using the Thermal Death Time Model by regressing log D on temperature (°C) as shown in Fig. 2b. Results for the temperature dependence of the sensory quality attributes and some additional attributes measured objectively are given in Table 1. The experimentally determined Z-values

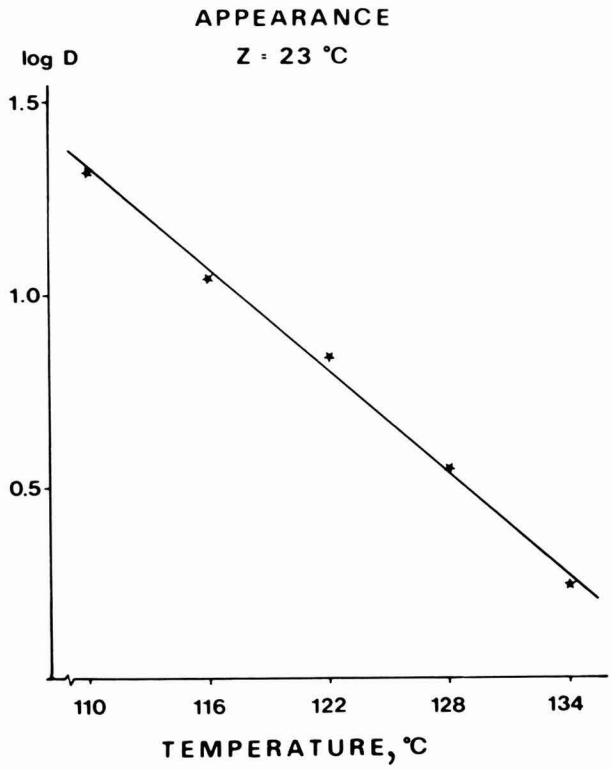

FIG. 2b. APPEARANCE OF STERILIZED FISH PUDDING

The common logarithm of the heating time required for the panel score of the appearance to go from good to acceptable as a function of the heating temperature. The regression line is also illustrated together with the calculated z-value. Reprinted from *Journal of Food Science 45*, 836 (Ohlsson 1980b) Copyright © by Institute of Food Technologists.

(°C temperature change necessary for D to change by one order of magnitude) ranged from 13-34°C with an average of 23°C ($E_a \sim 30$ kcal/mol). The Z-values are in the range for chemical reactions occurring in foods (Lund 1975). Ohlsson concluded that it is justifiable to describe quality deterioration in the temperature interval of 110°C to 134°C as a first order reaction. Based on the data, this conclusion is not supported since the response of the panel was linearly dependent on heating time, not logarithmically. Thus the panel response itself is zero order. Inferences on the kinetic model of quality deterioration from sensory data must be made with caution unless the relationship between panel score and intensity has been quantified.

Hayakawa *et al.* (1977) used sensory analysis to determine kinetic parameters for "quality score" for green beans, corn, peas and asparagus subjected to various time/temperature treatments. They used the time at various temperatures to reach a quality score of 4 (out of 8) as an indication of the time constant for overall quality. The temperature dependence of overall quality (expressed as Z-value) was 28°C for green beans and peas and 32°C for corn. Data on asparagus could not be evaluated because the authors concluded that some minimum heat treatment was necessary to produce thermally processed asparagus with acceptable quality. This illustrates the complexity of using sensory analysis to determine quantitative parameters.

Athough there have been many studies using sensory analysis to evaluate the effects of processing on quality attributes in foods, few have generated data which could be quantified in the form of a model

Table 1. Z-values for quality characteristics in various products[a]

	Z-Value (°C) for:					
Characteristic	Fish Pudding	Liver Paste	Strained Beef	Strained Vegetables	Tomato Sauce	Vanilla Sauce
Odor	29	26	22	18	27	13
Off-odor	—	—	20	—	18	22
Appearance	23	33	21	21	16	21
Taste	24	34	19	24	17	20
Off-taste	29	29	20	24	18	22
Consistency	28	29	—	—	—	20
Hardness	—	26	—	—	—	—
Coarseness	—	25	—	—	—	—
Lightness	25	21	22	21	28[b]	20

[a]Temperature range: 100°C to 134°C
[b]Redness
From: Ohlsson (1980b)

useful for predicting effects of processing. Although the original intent of this paper was to consider texture, flavor and appearance, I have used the author's prerogative to reduce the scope of the paper. In fact there are instrumental methods for measuring all of these attributes. The problem lies in correlating these objective measurements with quality perceived by the consumer. For flavor and appearance, characterization and quantification of reactants and products must be determined objectively and ultimately correlated with sensory analysis. Changes in these attributes should most closely parallel conventional chemical kinetics since flavor and appearance (color) are responses to chemical stimuli (flavor) or stimuli generated by a chemical (color). Thus, particularly for flavor and color, a dependence on environmental parameters (such as temperature) should closely parallel that for nutrients, for example. In the case of texture, however, the nature of the changes and the perception of texture by consumers does not appear so straightforward. Thus the remainder of this paper will focus on the state of knowledge on quantifying textural changes in foods.

Objective Measurement of Texture

If rheological measurements are to reflect the texture of a product perceived by the consumer, it follows that information on the conditions of evaluation in the mouth are important. Kapsalis and Moskowitz (1978) presented their views on the relation between instrumental tests and sensory analysis and concluded "that conditions between sensory and instrumental texture measurements are usually of an associative, indirect (at times even coincidental) nature, reflecting underlying effects which may be operating in the same or different directions on the two sides of the correlation." Thus correlation and prediction do not imply equality or, more importantly, an understanding of the mechanisms behind the association.

Tremendous progress has been made in the last ten years in identifying events that occur in the oral cavity leading to evaluation of quality attributes. For oral evaluation of viscosity, Shama and Sherman (1973a) presented the "master curve" for shear stress versus shear rate (Fig. 3). The data were generated by comparison of objective flow curves for 13 Newtonian fluids with a panel's judgment for comparative viscosities in the mouth of different pairs of test samples. The dashed line rectangles in the figure represent the boundaries of shear stress/shear rate in the different groups of samples. The two solid curves represent the region of sensory stimuli

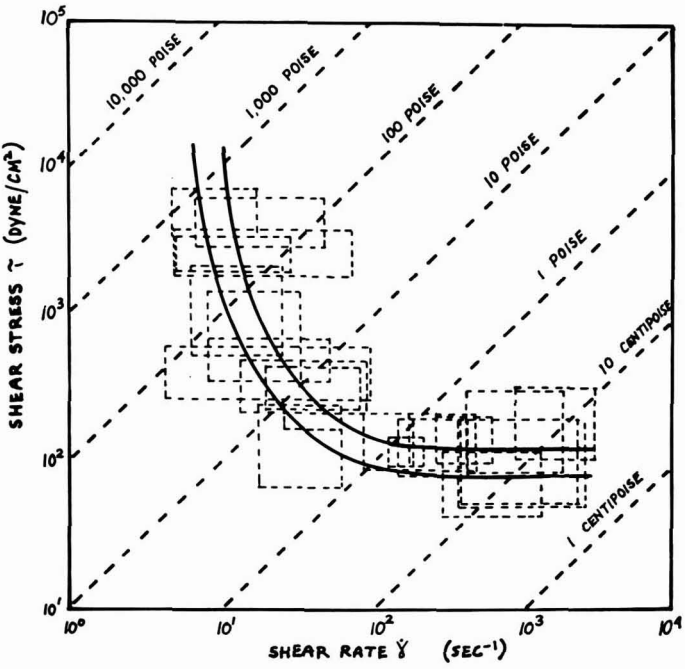

FIG. 3. BOUNDS FOR SHEAR STRESS AND RATE ASSOCIATED WITH ORAL EVALUATION OF VISCOSITY

Reprinted from J. Texture Studies (Shama and Sherman 1973a).

operating in the mouth. The conclusion from the study is that in the low-viscosity products ($\mu < 70$ cp), the perceived stimulus may be the shear rate at an approximately constant shear stress of 100 dynes/cm^2, while in the high-viscosity products ($\mu > 70$ cp), the stimulus may be the shear stress at an approximately constant shear rate of 10 s^{-1}. Christensen (1979) in studies on oral assessment of liquids with differing deviations from Newtonian behavior concluded that the untrained human subject's perception of viscosity may represent some sort of average viscosity over a range of shear rates. To complicate the situation further, when nonoral sensory cues of solution viscosity were minimized, the untrained observer was less able to perceive differences. This suggested that nonoral sensory modalities may be more sensitive to viscosity than oral perception.

Obviously an ingested liquid is subjected to many changes in the mouth. Variables of pH, heat, saliva and enzyme-modification of the liquid all affect oral perception of viscosity. Rha (1979a) reviewed the

importance of these factors in evaluating liquid foods. Rha (1979b) presented an excellent discussion of viscoelastic properties of food as related to micro- and molecular structures.

For solid foods it is also important to select an objective method and conditions of the test which adequately correlate to sensory properties. The data of Sherman and Shama (1973b) on textural characteristics of White Stilton and Gouda cheeses illustrate the importance of selecting the correct crosshead speed and taking the data in the right region of % compression. Figure 4 gives the force/deformation curves for White Stilton (dot-dash lines) and Gouda (solid lines) cheeses for crosshead speeds in the range of 5 cm/min to 100 cm/min. The sensory textural panel always rated Gouda as being harder than White Stilton. However, examination of Fig. 4 reveals that the objective test gives higher force values for Gouda than White Stilton at crosshead speeds of 20, 50 and 100 cm/min in compression ranges of 38-62%, 35-75% and 25-80%, respectively. The shaded area between the two curves defines the range of force-compression-crosshead speed conditions to be used with these products when good correlations with sensory assessment are desired. Szczesniak (1979) presented an excellent review of recent developments in sensory analysis for solving consumer oriented texture problems. She reviewed the problems in nomenclature, sensory stimuli and conditions actually occurring in the oral cavity, stress/strain conditions for testing, relationship between objective measurement and consumer preference, and consumer testing techniques. She concludes that many of these areas are inadequately researched and that progress has been very slow. The greatest progress has been made in nomenclature and developing information on the conditions for conducting various objective tests but future work will definitely need to involve psychologists and psychometricians.

Use of objective assessment of texture for developing consumer acceptable products can be illustrated by considering Texture Profile Analysis (TPA) and texturogram. Szczesniak *et al.* (1975) described a consumer texture profile technique similar to TPA (Brandt *et al.* 1963) which yielded a quantitative description of the product as perceived by the consumer and also permitted the description of an ideal texture for the specific food. Application of the method to puddings is shown in Fig. 5. Figure 5a presents the profile for the product and Figure 5b gives the deviations from the "ideal." Clearly this technique indicates those characteristics that need to be changed in order to improve the product. Although this technique serves to quantify textural characteristics particularly from the perspective of the consumer the technique does not appear to be in much use.

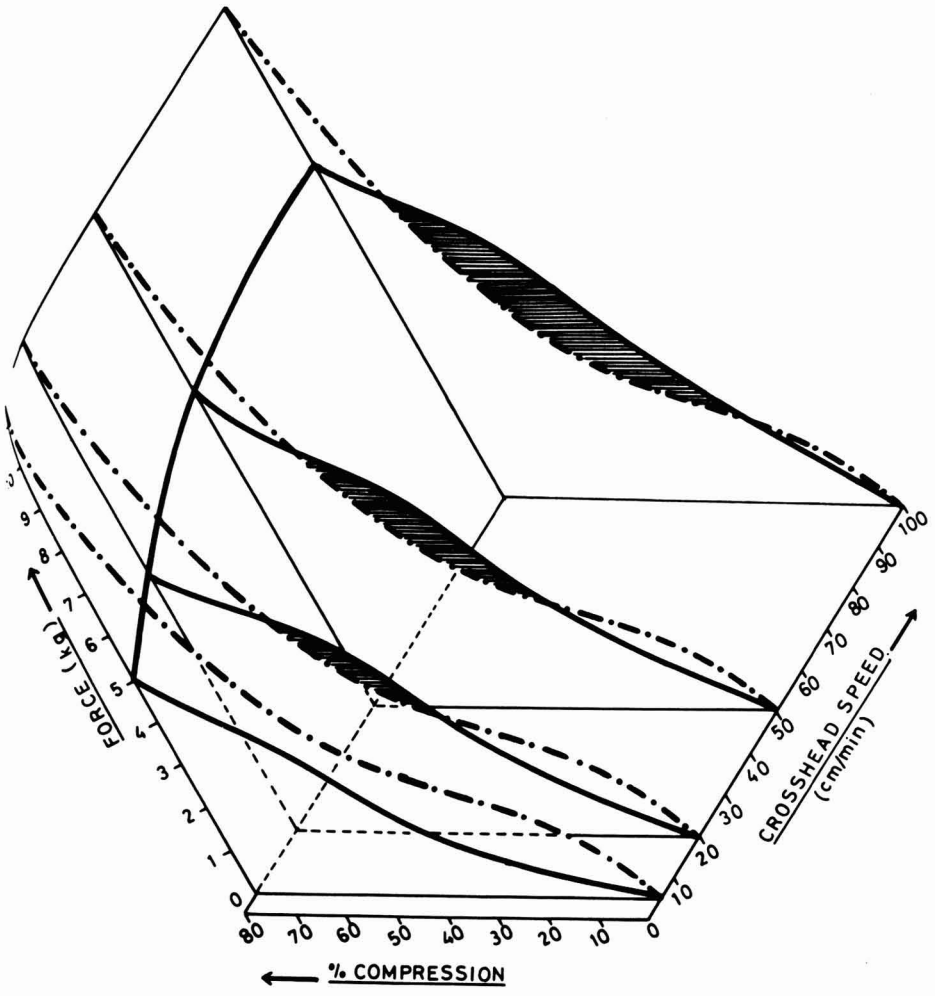

FIG. 4. THREE-DIMENSIONAL PLOT OF FORCE-COMPRESSION-RATE OF LOADING DATA FOR WHITE STILTON AND GOUDA CHEESES

—·—·— White Stilton. _____ Gouda. (Shama and Sherman 1973b)

An interesting combination of objective and sensory panel evaluations was presented by Okabe (1979) on the palatability of rice in Japan. Using the General Foods Texturometer, Okabe characterized the hardness and stickiness of rice and determined the preference for each sample by a trained panel. A combination of the results produced

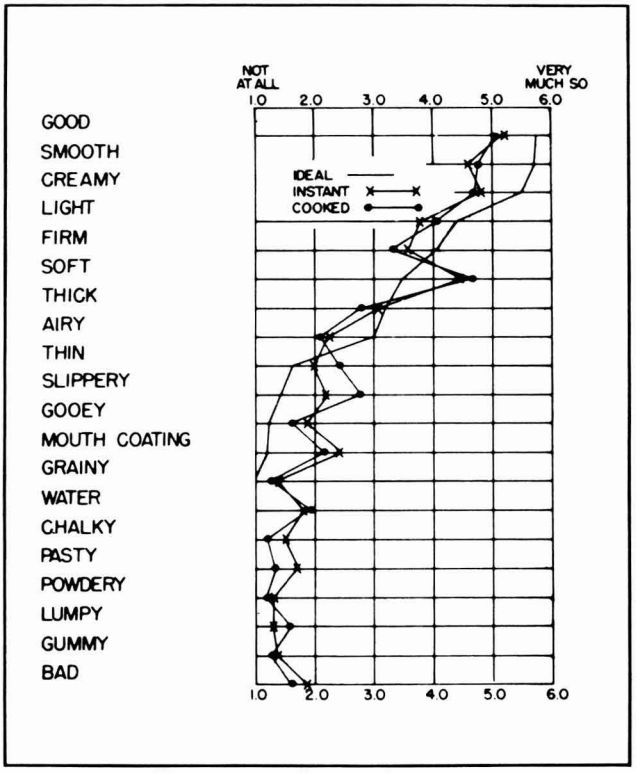

FIG. 5a. CONSUMER TEXTURE PROFILES FOR PUDDINGS, SHOWING DATA FOR THE IDEAL PUDDINGS AND ACTUAL TEST PRODUCTS

Reprinted from Journal of Food Science *40*, 1253 (Szczesniak *et al.* 1975) Copyright © by Institute of Food Technologists.

the Texturogram presented in Fig. 6. The zones of the Texturogram are ranked in order of decreasing acceptability, A through E, and each acceptability zone is further subdivided into five hardness groups designated 1 through 5. The most preferred characteristics of cooked rice by the Japanese is Zone A2. The advantage of developing this type of quantitative analysis of texture is that samples subjected to various treatments (e.g., cooking conditions including time/temperature/moisture, storage conditions, variety of rice) can be evaluated objectively and immediately assessed for their consumer preference.

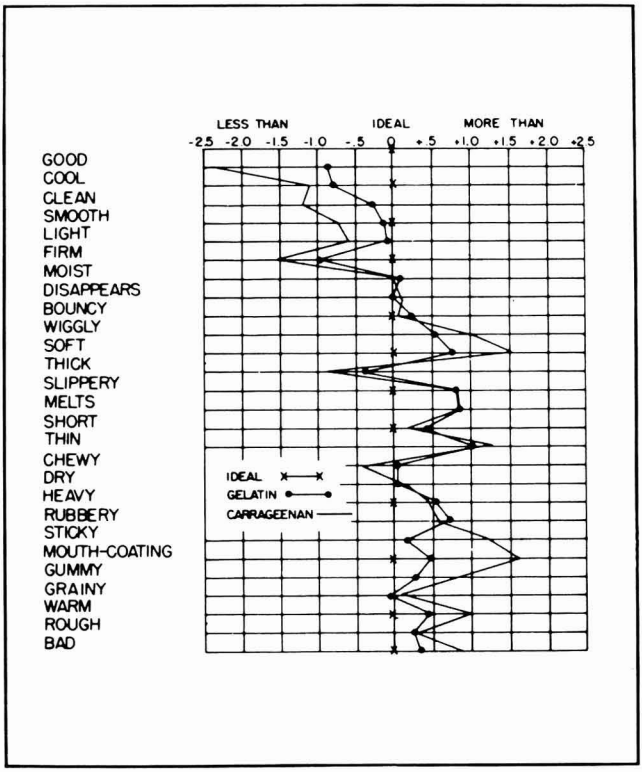

FIG. 5b. DEVIATIONS FROM IDEAL CONSUMER TEXTURE PROFILE FOR DESSERT GELS

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Structure of Foods

There is an extremely large body of literature on the structure of foods and no attempt will be made to review the literature here. However, the point should be made that an engineer who is designing a machine or a process must know the nature of the product. Reeve (1971) presented an excellent review of the relationships of histological structure to texture of fresh and processed fruits and vegetables. Sefa-Dedeh and Stanley (1979) presented a similar review on the textural implications of the microstructure of legumes. Stanley and Tung (1976) presented an excellent review on methodology for studying microstructure of food and discussed the structure of muscle

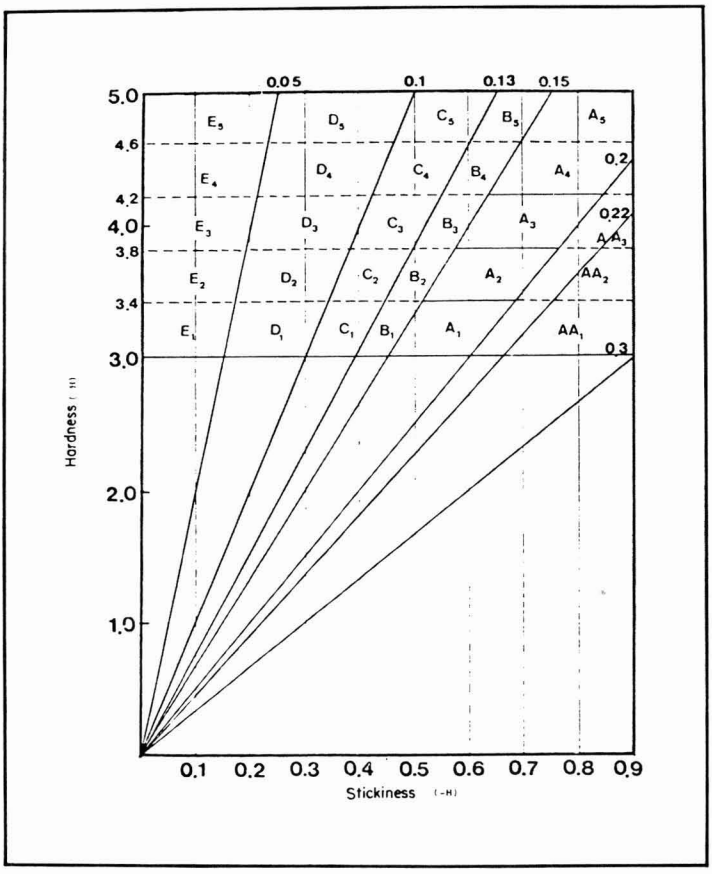


FIG. 6. TEXTUROGRAM FOR COOKED RICE, INDICATING ZONES OF ACCEPTABLITY

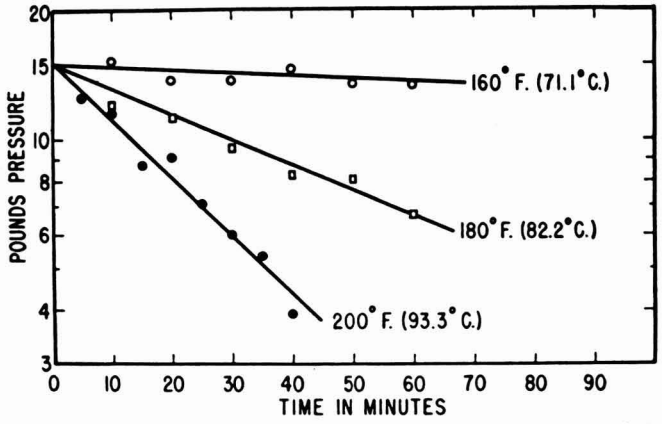
A = excellent, B = good, C = slightly poor/acceptable, D = poor, E = unacceptable, AA = glutinous rice. Reprinted from J. Texture Studies Okabe (1979). Copyright © by Food & Nutrition Press, Inc.

systems, food phytosystems, dairy products and cereal and oilseed products. These are examples of the literature available on structure of foods which provides the engineer with an appreciation of the nature of tissue. Furthermore, a qualitative description of the effect of process variables on the structure of the tissue gives the engineer insight into which variables are most important. Unfortunately quantitative relationships between structural changes and perceived texture or objectively measured textural parameters are not generally available. This is an area which requires further research.

Quantifying the Effects of Processing on Texture

Although there has been significant progress in relating objective and subjective tests for food texture, there have been relatively few attempts to develop parameters which can be used to quantify the effect of processing on texture. The "kinetics" of change in texture as a function of process have been reported in some studies but there has been no attempt to collect these results and to generalize. Bourne (1976) pointed out that softening that accompanies heating of plant tissue usually follows a "first-order" response. An example for softening of cucumbers is given in Fig. 7 and for thermal softening of legumes in Fig. 8.

Although no attempt was made to do an exhaustive literature search, several papers were found which had sufficient data to permit calculating an Arrhenius Activation Energy (E_a) for textural changes in various food systems. The results of those calculations are given in Table 2. Several observations can be made upon examination of the data. First, most of the studies have been done on high moisture samples. The effect of water activity on texture has not received much attention. More studies simlar to that by Katz and Labuza (1981) are needed. They investigated the effect of water activity on the sensory crispness and mechanical deformation of snack food products. Their results for popcorn using intensity rating for crispness and hedonic



From Nagel and Vaughn (1954)

FIG. 7. REDUCTION IN FIRMNESS OF CUCUMBERS ON HEATING

Reprinted from Food Res *19*, 613 (Nagel and Vaughn 1954)

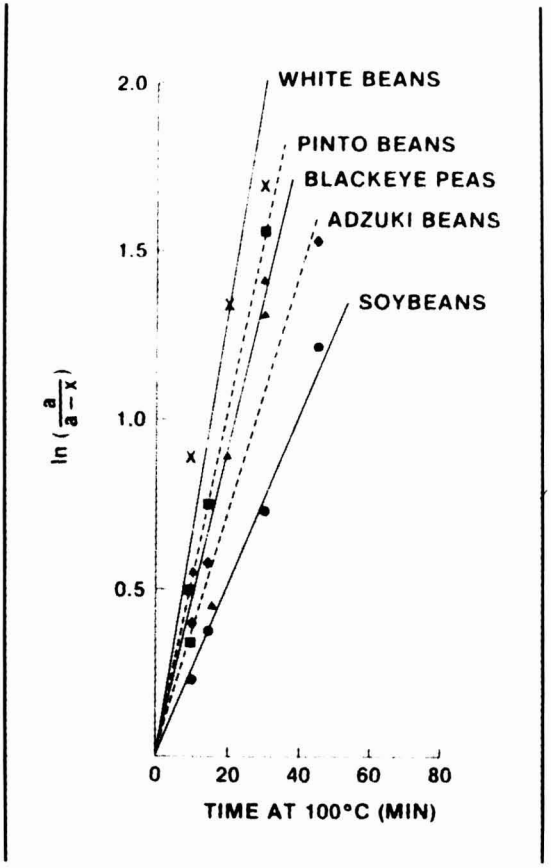


FIG. 8. THERMAL SOFTENING OF LEGUMES

plotted according to the first-order equation, $\ln[a/(a-x)] = k,t$, where t = time of heating at 100°C; a = maximum force of legume heated to 100°C; (a—x) = maximum force on legume heated at 100°C for t min; and k_1 = first-order reaction rate constant. Reprinted from Food Technology *33*(10), 77 (Sefa-Dedeh and Stanley 1979) Copyright © by Institute of Food Technologists.

rating are shown in Fig. 9. They identified a critical water activity (a_c) below which the product was unacceptable. For all of their products, a_c was in the water activity range of 0.35 to 0.50. They pointed out that this is also the range for initiation of amorphous to crystalline changes in simple carbohydrate containing food systems and for mobilization of soluble food constituents (i.e. above the monolayer).

The second observation based on Table 2 is that there are no studies cited in which moisture is migrating into the sample resulting in

Table 2. Z-values for texture changes in food systems

Component	pH	Temp. (°C)	E_a (kcal/mol)	Reference
Beef semitendinosus muscle	nat.[a]	55-59	140	Machlik and Draudt (1963)
Kangaroo tail tendon	1.8	50-70	67	Weir (1949)
	4.2		145	
	7.2		152	
	12.5		77	
Kangaroo tail tendon	6.0		141	
Kangaroo tail tendon with 0.1 mol/1 NaCl	6.2		95	
1.0 mol/1 NaCl	6.3		93	
2.0 mol/1 NaCl	6.2		83	
4.0 mol/1 NaCl	6.1		87	
sat. with NaCl	6.0		75	
External portion aponeurctic sheet	nat.	60-70	106	Winegarden *et al.* (1952)
Ligamentum muscle	nat.	60-70	76	Winegarden *et al.* (1952)
Deep pectoral muscle	nat.	50-60	44	Bauten and Harris (1972)
Semimembranous muscle	nat.	60-100	23	Tuomy *et al.* (1963)
Peas	nat.	77-93	20	Mansfield (1974)
Cucumbers	nat.	71-93	22	Nagel and Vaughn (1953)

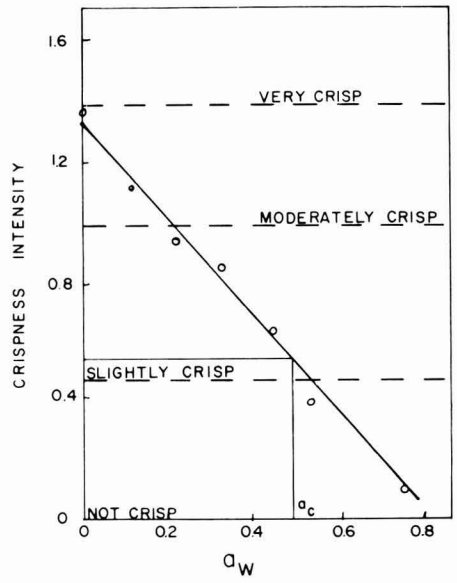


FIG. 9a. SENSORY CRISPNESS INTENSITY OF POPCORN VERSUS WATER ACTIVITY

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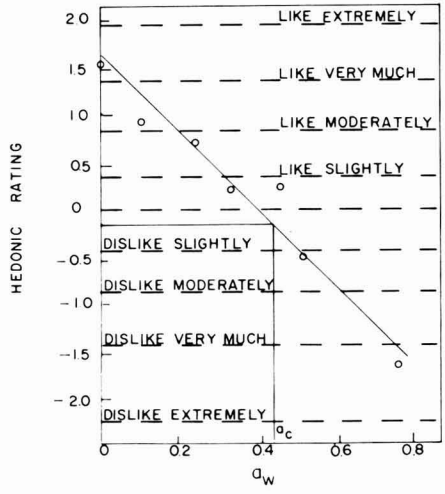

FIG. 9b. TEXTURAL HEDONIC RATING OF POPCORN VERSUS WATER ACTIVITY

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modification of texture. There are few studies of textural changes as a result of simultaneous heat and moisture transfer in the sample. Although cooking of dry legumes and cereal grains are examples of processes involving simultaneous moisture migration and softening, there has been no attempt to develop a generalized model to describe the processes.

Finally, generally temperature dependence of textural characteristics of meat (muscle) is greater than that for plant tissue. The apparent activation energy for muscle is in the range associated with structural changes induced by proteins, whereas for plant tissue it is in the range associated with chemical reaction. For muscle tissue, the temperature dependence of change in texture is extremely complex because of the collagen shrinkage reaction and the collagen-gelation tranformation. In addition there are changes in the structure of the muscle protein itself. For plant tissue, hydrolysis of cell wall constituents, swelling due to expansion of gases and heat induced changes in water holding capacity (e.g. gelatinization of starches), can all affect texture. Although there are complex changes which occur during heating, it would appear that an attempt should be made to model textural changes. Perhaps a first-order model with an Arrhenius temperature dependence would be a good first approximation.

SUMMARY

Food engineers need quantitative data on effect of processing variables on texture in order to systematically design food processing machines and processes. While there are considerable data and process models on the effect of process parameters on microorganisms, enzymes, nutrients, color, and flavor, there are very few models for texture. Although there have been significant developments in instrumentation and sensory analysis of texture, the correlation between the two is still in need of additional research. There has been a good attempt to study the conditions of stress/strain which actually occurs in the oral cavity so that objective tests can be designed to simulate the action in the mouth. As the science of materials testing develops further, it will be possible to be quantitative in descriptions of texture.

In general, the food engineer is not sufficiently acquainted with the mechanical properties of the material being processed and does not

have an appreciation of the microstructure of the material. Some data are available on the kinetics of change in texture especially as a function of heating temperature. However, the data are so insufficient that it is tenuous at best to make generalities. Textural changes in muscle tissue generally exhibit activation energies over 50 kcal/mol, whereas for plant tissue, it is between 15 and 40 kcal/mol. One of the problems that has not been adequately examined is the effect on textural response of a distribution of heat treatments as in conduction heating foods. When the outside is overcooked and the center is undercooked, how does that influence perception of texture by the consumer? Does the consumer take some sort of average textural characteristic in that case?

The effect on texture of simultaneous heat and mass transfer as in soaking/cooking of dry legumes and cereal grains has not been sufficiently studied although models do exist describing water absorption as a function of time and temperature. Finally, the effect on texture of other variables such as moisture content (or water activity), pretreatment of the product, and pH, have not received adequate attention. Hopefully these studies will be designed so they yield quantitative models. In this way, eventually a data bank will be generated which can be examined for generalizations and trends.

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RESPONSE SURFACE EXPERIMENTATION[1]

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INTRODUCTION

There are many reasons for experiments, but two are particularly significant for this paper. Many experiments either test a hypothesis or study the response characteristics of a system. Hypothesis testing experiments are generally asking whether there is a true difference between two or more items. The items may be different varieties of a particular crop, they may be samples drawn from a production line that are being compared against a standard for quality control, or they may be a product submitted to a taste panel for comparison. The test may compare separate items or it might test for differences between treatment levels of alternative mechanisms.

Response surface experiments attempt to identify the output or response of a system as a function of the explanatory variables. For example, an experimenter might attempt to identify the influence of cooking practices on the nutritional quality of food. Alternatively, a manufacturer might study the cost of production as a function of mixing time, processing temperature and product composition. Generally the results of response surface experiments are either reported as a mathematical model or used to optimize the system response.

The response can be thought of as a surface over the explanatory variables' experimental space. Consequently, the term response surface has been associated with experiments intended to identify or evaluate one or more response variables as a function of the independent variables. Response surface in this paper will specifically mean the results of experiments to identify a mathematical/statistical relationship between explanatory variable levels and the response.

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The originial publication in response surface methodology was by Box and Wilson (1951). These authors noted that experimental runs within a designed experiment are sometimes run sequentially, that a frequent reason for undertaking response surface experiments is to search for an optimum response and that generally the mathematical form for the response is not known.

Response surface experimentation as it is practiced today is not confined by any of these assumptions. Several published agronomic studies were based on response surface experiments in which all of the trials or runs were simultaneously conducted. For example, the influence of the amount of several different fertility elements on yield may be evaluated in several plots during one growing season and reported as a response surface function. Response surface experiments are run for many purposes in addition to finding optimum or improved response levels. Finally, response surface experiments are being run when a specific statistical model for the response is known. Kinetic models are routinely being utilized in single (Kittrell 1970) and multiple response surface experiments (Ziegel and Gorman 1980). Many authors have argued that when an appropriate model based on reasonable assumptions has been developed, it should be used rather than a general response model.

Box and coworkers continued to develop and publish on reponse surface methodology. Perhaps the most popular paper was published by Box and Hunter (1957) and introduced the concept of rotatability. In the same year, Box published a book on the application of response surface techniques to production optimization. Box and Lucas (1959) departed from using polynomial equations to represent the response. The nonpolynomial approach was further extended by Atkinson and Hunter (1958). Many other authors have contributed ideas for response surface methodology, but the concepts published by Box and coworkers seem to predominate.

The purpose of this paper is to summarize selected statistical models, experimental design and analysis methods for response surface experiments. This paper is not intended to be a comprehensive review of response surface literature. Instead, it is intended to be an introduction to a subset of the reported methods that are frequently utilized by experimenters. This paper only considers models that are linear in the parameters and designs for experiments in which the explanatory variables are completely independent of one another. Experiments with interrelated explanatory variables, mixture experiments, have been reviewed previously (Thompson 1981).

There are several good text books and review articles on response

surface methodology. The commonly mentioned text or reference books are the ones by Davies (1954), Cochran and Cox (1957) and John (1971). Good review articles have been published by Hill and Hunter (1966) and Mead and Pike (1975). Probably the most important advances in response surface methodology since the 1975 review have been in experimental designs. Some of these will be mentioned in a later section.

EQUATIONS FOR REPRESENTING RESPONSE SURFACES

It has been argued by a number of statisticians (Mead and Pike 1975; Bliss 1970; and Hill and Hunter 1966) that all models for biological or agricultural systems are essentially empirical. The only difference is the extent to which the models appear to be consistent with known biological phenomena.

When possible, a model developed from an intimate understanding of the biological mechanism should be utilized. The agreement of a series of observations with the mechanism based model is a necessary but not sufficient test of its validity. If the mechanism based model requires more parameters than an empirical model or if it is nonlinear in the parameters, a more empirical model may be favored. An experimenter should not avoid models that are nonlinear in the parameters for that reason only. Methods for designing experiments and estimating parameters for nonlinear models are available (Cochran 1973; Mead and Pike 1975). However, an efficient experimental design requires good advance estimates of the parameters and an iterative process is required for parameter estimation. In this section some of the empirical equations that are linear in the parameters are reviewed.

Polynomial Functions

Polynomials of first degree (Eq. 1) and second degree (Eq. 2) are the most frequently used response functions. These functions estimate the response, E(y) as a function of the parameters, β_i and the explanatory variables, x_i.

$$E(y) = \beta_o + \sum_{i=1}^{k} \beta_i x_i \qquad (1)$$

$$E(y) = \beta_o + \sum_{i=1}^{k} \beta_i x_i + \sum_{i \leqslant j}^{k} \beta_{ij} x_i x_j \qquad (2)$$

The number of parameters, p, in these equations is k + 1 (first degree) and (k +1) (k +2)/2 (second degree).

There are a number of reasons for the popularity of the polynomial response functions. The quadratic or second degree function is very easily formed by the addition of terms to the linear or first order function. The optimum can easily be defined mathematically. Estimates of the parameter values in the polynomial equations can be made by the method of least squares without complex calculations. Some authors have argued that the polynomial is similar to the first few terms of a Taylor series expansion of the true response function. However, in application polynomials seem to be used as the simplest smoothing curve.

First order models rarely adequately represent biological phenomena. However, these models are useful for screening experiments. The purpose of screening experiments is to identify the most significant explanatory variables. Sometimes the first order models effectively represent the data when used with variable transformations.

There are several disadvantages of polynomials which sometimes are overlooked by authors. Extrapolation outside the range of the independent variable values used to estimate parameters is impossible. Polynomials are smoothing functions with no biological justification which would be required for any extrapolation. The second degree or quadratic polynomial is symmetrical about the optimum. This can severely restrict the ability of this polynomial to fit nonsymetric responses. Second and higher degree polynomial models tend to be sensitive to outliers. Another disadvantage of polynomial functions is that they do not include a form that can asymptotically approach a constant response level. This response form is frequently observed in biological and agricultural data.

Cubic and higher order polynomials can be used to represent response data, but they have rarely been reported. Higher order responses are very difficult to explain in biological terms. Most experimenters prefer to attribute any variation explained by these higher order terms to random variation.

Power Functions

Numerous power functions have been proposed. These equations, like polynomials, are strictly empirical, but eliminate some of the

disadvantages of polynomials. Like the polynomial models the power functions cannot be extrapolated. The power functions also share, to a lesser extent, the same advantages attributed to polynomial functions.

Equations 3 and 4 illustrate two of the many power function forms that have been utilized by researchers.

$$E(y) = \beta_o + \sum_{i=1}^{k} \beta_i x_i^{r_i} \tag{3}$$

$$E(y) = \beta_o + \sum_{i=1}^{k} \beta_i (x_i - x_{0,i})^{r_i} \tag{4}$$

The exponent, r_i, may assume either positive or negative values depending on the data, but their values must be selected before the other parameters can be estimated by linear regression. Negative values for r_i generate inverse terms which will represent peaks in the reponse. The base level for the explanatory variable, $x_{o,i}$, in Eq. 4 allows the experimenter to adjust the location of the peak response.

Positive powers of r_i may assume either integer values (polynomials) or noninteger values for an infinite family of curves. Bliss (1970) discussed various modifications of these two equations and methods for fitting data values to the curve. Generally the constants r_i and $x_{0,i}$ must be selected by the experimenter based on general data characteristics. The parameters represented by the β's can be found by least squares regression.

Powers of the response variable, y, have also been considered (Box and Cox 1964; Lindsey 1972; Wood 1974). This is a data transformation that is sometimes useful for improving the representation by the model.

Exponential Relationships

Equations 5, 6 and 7 represent only a few of the exponential relationships that have been used for response curves.

$$E\,[\log(y)] = \beta_0 + \sum_{i=1}^{k} \beta_i\, x_i \tag{5}$$

$$E(y) = \beta_0\,[1 - \exp(-\beta_1 x_1)] \tag{6}$$

$$E\left[\log\left(\frac{y_0 - y}{y}\right)\right] = \beta_0 + \sum_{i=1}^{k} \beta_i x_i \qquad (7)$$

Most of the equations that have been proposed for exponential relationships relate a single explanatory variable to the response as in Eq. 6. Exponential relationships based on several explanatory variables are generally formed by taking a logarithm transformation of the responses as was done in Eq. 5 and 7. The simplest form of the exponential functions, Eq. 5, is very useful when the response may change by one or more orders of magnitude over the region of interest. Higher order terms may be included on the right side of Eq. 5. The exponential form in Eq. 6, sometimes attributed to Mitscherlich (1930) is widely used for responses that asymptotically approach a maximum level, β_0. Equation 7 is useful when the response variable represents a proportion (Berkson 1944).

Numerous other models and data tranformations have been proposed. Several authors (Box and Tidwell 1962; Dolby 1963; Hoerl 1954) have discussed the selection of appropriate tranformations.

RESPONSE SURFACE DESIGNS

Most of the literature on response surface experimental designs focuses on polynomial models. The emphasis has been on first and second order polynomials with a limited amount of work on third order designs. This discussion includes only first and second order designs because even when second order models are found to be inadequate higher order models are not usually selected. Instead, the experimenter frequently investigates transformations and power equations. Generally the response can be adequately represented with fewer parameters than would be required in a third order polynomial. If needed, specific information on third order designs has been reported by Draper (1960a, 1960b, 1961 and 1962) and Herzberg (1964). Additional information on first and second order designs is available in Cochran and Cox (1957), Cox (1958), Davies (1971) and Vojda (1967).

The explanatory variable levels are coded for an experimental design. The center point for each explanatory variable level is given a code of zero. The highest and lowest levels of interest for each independent variable are coded plus or minus one respectively for three level designs. For designs with more than three levels, the highest and lowest levels of interest are given maximum and mini-

mum codes respectively. Linear transformations can easily be written for converting actual values to codes and codes back to actual values. The use of coded levels has several advantages. Experimental designs can be written without knowing the interest range for each explanatory variable. The magnitude of regression coefficients can be directly compared because the range for each variable is identical. Response patterns can also be better estimated when all of the explanatory variables have the same center values and a similar range.

The combination of an assumed model with p parameters and n observations resulting from experiments at each of the design points can be represented in matrix form as shown in Eq. 8.

$$\bar{y} = X\bar{\beta} + \bar{\epsilon} \tag{8}$$

The vectory $\bar{y}$ is the n observations; X is a matrix with n rows, one for each design point, and p columns, one for each term of the statistical model; $\bar{\beta}$ is a column vector of the p unknown parameters and $\bar{\epsilon}$ is a vector of the n random errors in each of the respective experimental observations. The matrix X must be of rank p.

Criteria for comparing experimental designs have been developed. The D- and G-optimality are frequently cited. A design is D-optimal if the determinant $(X'X)^{-1}$ is minimized by the proposed design (Ash and Hedayat 1978). The D-efficiency of a design is the 1/p power of the ratio of the X'X determinant for the design to the X'X determinant for the optimum design with the same number of points. The G-optimality criteria (Snee and Marquardt 1976) is expressed as a percent efficiency and is corrected for the number of points in the design.

$$\text{G efficiency} = \frac{100k}{nd} \tag{9}$$

where d is the maximum value of $v = \bar{x}(X'X)^{-1}\bar{x}'$ over the n points included in the design for k explanatory variables. The G-optional design minimizes the maximum prediction variance.

Wheeler (1972) suggested that a G-efficiency of 50% or better is adequate for practical purposes. Snee (1975) agreed but suggested that the value of d should also be no greater than 1.0. An alternative criteria (Wynn 1972; Atwood 1972) requires the D-efficiency to equal or exceed $100\,(n(n-1)(n-2)\ldots(n+1-p)/n^p)^{1/p}$ percent and the G-efficiency to equal or exceed $100\,(n+1-p)/n$ percent.

Several other criteria have been proposed for comparing experimental designs (Ash and Hedaijat 1978; Fedorov 1972; Galil and Kiefer 1977; Kiefer 1974), but these are less frequently cited. The robust criterion (Box and Draper 1975) is perhaps of greater signficance to biological research than to research where greater variable control and measurement is possible. A design is considered robust if it is insensitive to wild observations. Box and Draper (1975) showed a close association between G-efficiency and robustness.

First Order Designs

Experimental designs specify the order of experimental units, runs or points and the explanatory variable levels for each run. By using coded levels for each variable, the designs are dependent only on the number of variables and the selected response equation.

Experimental designs must assure the experimenter that adequate information will be collected to estimate each of the parameters in the proposed model. In addition, there should be several degrees of freedom available for estimating one or more error terms or residuals. In response surface experimentation it is desirable to estimate a pure or experimental error term and a lack-of-fit error term. The pure error term represents the variation that is experienced when repeating a run at the same design point. This level of variation may change in different locations of the experimental space or with time. If the experimental variation or pure error is known to be independent of time, it can be estimated before or after the response surface experiment. If it is thought to be reasonably independent of location in the experimental design, it can be estimated from replications of a single experimental point. Frequently, the experimental or pure error term is estimated from replicated experiments at the center of the design space (all independent variables at a coded level as zero). This assumes that the pure error term may change with time but will be reasonably uniform throughout the experimental region.

Many of the designs do not include replicated points from which the pure or experimental error can be estimated. Unless the pure error level is known from previous experiments, these points should be added to the design. Alternatively, the entire design can be replicated. This eliminates the possibility of a single outlier rendering the experiment useless (Box and Draper 1975).

First order polynomials include k + 1 parameters. Therefore, the experimental designs must include this number of points plus the number of points needed to adequately estimate the error. The most

accurate parameter estimates are possible with experimental points at the extreme limits of each of the explanatory variables. This suggests a two level factorial experiment which includes 2^k experimental points. For experiments with two or three explanatory variables this is a reasonable design. However, as the number of explanatory variables increase, the number of experiments required for a two level factorial becomes unreasonable. For this reason fractional replications of factorial experiments are generally recommended for first order designs with four or more explanatory variables.

A full factorial experiment in k explanatory variables with two levels is referred to as 2^k factorial design. This notation not only defines the experiment but indicates the number of experiment units or points. Fractional replicates of factorial experiments always include an integer power of one-half times the number of points included in a full factorial experiment. Thus, a fractional replicate might include one-half, one-fourth, one-eighth or one-sixteenth of the number of points in the full factorial experiment. These experiments are represented by 2^{k-m} factorial experiments. The m in this notation is the power of one-half representing the fractional replication. As before this notation indicates the number of experimental units in the fractional factorial design. For example a one-fourth (m =2) replicate of a factorial experiment in 6 explanatory variables (k = 6) is represented by the notation 2^{6-2} and contains 16 experimental points.

Full factorial experiments generate adequate information to estimate all the parameters in a linear polynomial plus parameters for all possible interactions among the explanatory variables. For example, with three explanatory variables a full two-level factorial experiment includes 8 experimental points and adequate information to estimate the constant, the 3 main effects, the 3-2 factor interactions and the 3 factor interaction among these variables. No additional data values remain for estimating error so it is normal to assume that the 3 factor interaction is negligible and perhaps make the same assumption about the 2 factor interactions. Instead of estimating the values of these parameters, the information is pooled to estimate the error term.

Fractional replicates of factorial experiments do not contain sufficient information to estimate all of the parameters in the linear equation and parameters for all possible interactions. This is acceptable because our goal is to estimate only the parameters in the first order equation with adequate additional information to permit estimating the error which will be attributed to lack-of-fit. A fractional factorial experiment must generate the necessary information for estimating the parameters in the first order model and assure that

these estimates will not be influenced by responses which should be attributed to interactions among the explanatory variables.

The data from a fractional replicate of a factorial experiment contains no information for estimating one or more effects (parameters) in the linear plus interactions model and some of the remaining estimates represent the sum of two or more factors. The effects that cannot be estimated are called "defining contrasts." The factors that are summed for a given parameter estimate are called aliases or sometimes they are referred to as being confounded. For example, in Table 1 the defining contrast is the 3 factor interaction, $x_1x_2x_3$, and the aliases of each main effect is the 2 factor interaction involving the other 2 variables. Thus, if this experiment were run the parameter estimated for the x_1 term would include the effect from the x_2x_3 interaction. The parameters for the other two independent variables would likewise include the effect of the corresponding interactions. The constant estimated from this data would not be confounded with other information.

Table 2 is a repeat of the experiment shown in Table 1, but includes the resulting levels for each of the interaction terms for each experimental run. The 3 factor interaction is at the high level for all 4 runs. This is the reason that no parameter value could be calculated for the three factor interaction from the results of this experiment. This is

Table 1. A ½ replicate of a 2^3 factorial experiment design

Experimental Run Number	Design Level For Each Independent Variable		
	x_1	x_2	x_3
1	1	1	1
2	-1	-1	1
3	-1	1	-1
4	1	-1	-1

Table 2. Factor levels for the experimental runs of the 2^{3-1} factorial experiment

Run Number	x_1	x_2	x_3	x_1x_2	x_1x_3	x_2x_3	$x_1x_2x_3$
1	1	1	1	1	1	1	1
2	-1	-1	1	1	-1	-1	1
3	-1	1	-1	-1	1	-1	1
4	1	-1	-1	-1	-1	1	1

also the reason that this 3 factor interaction is called the defining contrast for this fractional replicate of the 2^3 factorial experiment. The experimental runs selected from the full factorial experiment were those for which the 3 factor interaction had a +1 value. All experiments in which this interaction had a -1 value were excluded from the one-half replicate. Observe that the levels for factor x_1 and for the interaction x_2x_3 are identical for all 4 experimental runs. The same is true for x_2 and the interaction x_1x_3 and for x_3 and the interaction x_1x_2. This is the reason that these terms are said to be confounded and the parameters estimated for each of the independent variables in the first order equation would include the effect of the corresponding interaction term.

The goal when designing fractional factorial experiments for first order models is to assure that the defining contrasts are of no interest and that the alias terms for each independent variable are insignificant. Unless the experimenter has apriori knowledge indicating that some of the two factor interactions have no influence, the alias for each independent variable and the defining contrasts should all be interactions involving three or more factors. Thus the experimental design shown in Table 1 would not be satisfactory although it is a useful illustration.

The defining contrast and the alias for each independent variable (main effect) can be identified before the design is recorded as in Table 1. The defining contrast can be arbitrarily selected by the experimenter because it identifies which experiments will be selected from a full factorial for the fractional factorial design. Multiplying the defining constrast by any of the independent variables, or interaction terms will identify the alias. In the previous example the defining contrast was $x_1x_2x_3$. Multiplying this by the first independent variable, x_1, gives $x_1x_2x_3$. Any variable multiplied by itself has a value of one (1^2 or -1^2) so the alias is the interaction x_2x_3.

After a suitable defining contrast has been identified, the full factorial experiment can be recorded with an additional column for the level of the defining contrast. For this the half replicate of the full factorial can be selected either as those experimental runs in which the defining contrast is at +1 or the experimental runs at which it has a -1 value. If the experimental runs for which the defining contrast has a -1 are selected, the parameters for the explanatory variables in the model equation will be estimating the effect of the explanatory variables in the model equation will be estimating the effect of the explanatory variable less the effect of the alias interaction term. If the

runs with a +1 defining contrast level are selected, the parameters estimate the sum of the effects. Normally, the interaction among all the explanatory variables is selected for the defining contrast in a one-half replicate design.

The preceding comments can be generalized to one-fourth or smaller fractional replicates of a 2^k factorial experiment. For a one-fourth replicate, two defining contrasts must be selected. The product of these contrasts, the generalized interaction, also will act as a defining contrast and cannot be estimated from the experimental data. Since there are 3 defining contrasts, each effect (explanatory variable or interaction term) other than the defining contrasts has 3 aliases. Each of these can be found by multiplying the effect times one of the defining contrasts. For a one-eighth replicate, 3 defining contrasts are selected and their products generate 4 generalized interactions (3 two factor products and 1 three factor product). Thus, each effect has seven aliases. This can be generalized to a one-sixteenth or smaller replicate.

The defining contrasts selected in a one-fourth or smaller replicate frequently do not include the largest interactions. For example selecting the interaction involving all of the explanatory variables and an interaction involving all but one of the explanatory variables would generate an explanatory variable as one of the defining contrasts. Two factor interactions should be avoided for defining contrasts because they will cause two explanatory variables to be aliases.

Table 3 identifies some possible designs and indicates their characteristics. Designs with resolution III permit all explanatory variable parameters in the first order equation to be estimated, but some or all of the parameters include the effect of one or more two factor interactions. In other words none of the main effects have an alias of another main effect, but some of the main effects do have two factor interaction aliases. Design resolution IV does not include any explanatory variable aliases that are either explanatory variables or two factor interactions. Resolution V allows no explanatory variables or 2 factor interactions to have an alias that is an explanatory variable or a 2 factor interaction. Several of the designs suggested in Table 3 are identified in Tables 4 through 6.

Designs including the minimum possible number of experimental runs (k + 1) have been identified by Plackett and Burman (1946) and Box and Hunter (1961). These reports include designs for as many as 100 explanatory variables. These designs include no degrees of freedom for estimating the error (pure or lack-of-fit). Replicated experiments at the center point (all explanatory variables at a coded level of

Table 3: Designs for first order response surface variables

Number of Explanatory Variables	Number of Linear Polynomial Parameters	Factorial Replicate	Design Characteristics				
			Designation	Number of Experiments	Degrees of Freedom	Selected Defining Contrasts	Resolution
2	3	Full	2^2	4	1	none	V
3	4	Full	2^3	8	4	none	V
4	5	Full	2^4	16	11	none	V
		½	2^{4-1}	8	3	$x_1x_2x_3x_4$	IV
5	6	Full	2^5	32	26	none	V
		½	2^{5-1}	16	10	$x_1x_2x_3x_4x_5$	V
		¼	2^{5-2}	8	2	$x_1x_2x_5, x_3x_4x_5$	III
6	7	½	2^{6-1}	32	25	$x_1x_2x_3\ x_4x_5x_6$	V
		¼	2^{6-2}	16	9	$x_1x_2x_3x_5$, $x_1x_2x_4x_6$	IV
		⅛	2^{6-3}	8	1	$x_1x_3x_5, x_1x_4x_6$ $x_2x_4x_5$	III
7	8	½	2^{7-1}	64	56	$x_1x_2x_3x_4x_5x_6x_7$	V
		¼	2^{7-2}	32	24	$x_1x_2x_3x_4x_5$	IV
		⅛	2^{7-3}	16	8	$x_1x_2x_3x_6x_7$ $x_1x_2x_3x_4, x_1x_2x_5x_6$ $x_1x_3x_5x_7$	IV
8	9	¼	2^{8-2}	64	55	$x_1x_2x_3x_5x_7$ $x_1x_2x_4x_6x_8$	V
		⅛	2^{8-3}	32	23	$x_2x_3x_4x_8, x_2x_4x_6x_7$ $x_1x_2x_3x_5x_6$	IV
		1/16	2^{8-4}	16	7	$x_1x_2x_3x_4, x_1x_2x_5x_6$ $x_1x_2x_7x_8, x_1x_3x_5x_8$	IV

Table 4. A 2^{5-1} fractional factorial design

Point Number	Explanatory Variable Level				
	x_1	x_2	x_3	x_4	x_5
1	+1	-1	-1	-1	-1
2	-1	+1	-1	-1	-1
3	-1	-1	+1	-1	-1
4	+1	+1	+1	-1	-1
5	-1	-1	-1	+1	-1
6	+1	+1	-1	+1	-1
7	+1	-1	+1	+1	-1
8	-1	+1	+1	+1	-1
9	-1	-1	-1	-1	+1
10	+1	+1	-1	-1	+1
11	+1	-1	+1	-1	+1
12	-1	+1	+1	-1	+1
13	+1	-1	-1	+1	+1
14	-1	+1	-1	+1	+1
15	-1	-1	+1	+1	+1
16	+1	+1	+1	+1	+1

Defining contrast $x_1x_2x_3x_4x_5 = +1$

Table 5. A 2^{5-2} fractional factorial design

Point Number	Explanatory Variable Level				
	x_1	x_2	x_3	x_4	x_5
1	-1	-1	-1	-1	+1
2	+1	+1	-1	-1	+1
3	+1	-1	+1	-1	-1
4	-1	+1	+1	-1	-1
5	+1	-1	-1	+1	-1
6	-1	+1	-1	+1	-1
7	-1	-1	+1	+1	+1
8	+1	+1	+1	+1	+1

Defining contrasts $x_1x_2x_5 = x_3x_4x_5 = 1$
(selected)
$x_1x_2x_3x_4 = 1$
(generalized interaction)

zero) could be utilized to estimate the pure error and provide a lack-of-fit estimate with one degree of freedom. The experimental designs listed in Table 3 include degrees of freedom for estimating the lack-of-fit but generally should be augmented with replicated center point experiments for estimating the pure error.

Table 6. A 2^{8-4} fractional factorial design

Point Number	Explanatory Variable Level							
	x_1	x_2	x_3	x_4	x_5	x_6	x_7	x_8
1	-1	-1	-1	-1	+1	+1	+1	+1
2	-1	-1	-1	-1	-1	-1	-1	-1
3	+1	+1	-1	-1	-1	-1	+1	+1
4	+1	+1	-1	-1	+1	+1	-1	-1
5	+1	-1	+1	-1	+1	-1	-1	+1
6	+1	-1	+1	-1	-1	+1	+1	-1
7	-1	+1	+1	-1	-1	+1	-1	+1
8	-1	+1	+1	-1	+1	-1	+1	-1
9	+1	-1	-1	+1	-1	+1	-1	+1
10	+1	-1	-1	+1	+1	-1	+1	-1
11	-1	+1	-1	+1	-1	+1	+1	-1
12	-1	+1	-1	+1	+1	-1	-1	+1
13	-1	-1	+1	+1	+1	+1	-1	-1
14	-1	-1	+1	+1	-1	-1	+1	+1
15	+1	+1	+1	+1	-1	-1	-1	-1
16	+1	+1	+1	+1	+1	+1	+1	+1

Defining contrasts: $x_1x_2x_3x_4 = x_1x_2x_5x_6 = x_1x_2x_7x_8 = x_1x_3x_5x_8 = 1$ (selected)

$x_3x_4x_5x_6 = x_3x_4x_7x_8 = x_5x_6x_7x_8 = x_2x_4x_5x_8 = x_2x_3x_6x_8$

$x_1x_3x_6x_7 = x_2x_4x_6x_7 = x_2x_3x_5x_7 = x_1x_4x_5x_7 = x_1x_4x_6x_8 = x_1x_2x_3x_4x_5x_6x_7$ (generalized interaction)

Second Order Designs

Most of the second order response surface experiments being reported today have utilized either a central composite design or a design attributed to Box and Behnken (1960). In addition, designs developed by Hoke (1974) and the noncentral composite (San Cristobal) designs proposed by Rojas (1963, 1972) are reviewed here. Numerous other designs have been proposed and may eventually see extensive application. These other designs will only be briefly mentioned here because they have not been found clearly superior (Lucas 1976, 1978). Nalimov (1970), Pesotchinsky (1975) and Mitchell and Bayne (1978) have attempted to develop optimum or near optimum designs for second order equations. These designs may be useful in special situations. However, most of them achieve higher efficiencies at a cost of more experimental runs and designs that cannot be easily communicated. Doehlert (1970) developed a class of uniform shell designs that are interesting but are not as efficient (Lucas 1976) as the designs presented here.

Central Composite Designs. Central composite designs were first proposed by Box and Wilson (1951). Important advances in these designs were proposed by Box and Hunter (1957) and in numerous other articles by Box and coworkers. Central composite designs include three types of experimental points. A 2^k or a 2^{k-m} fractional factorial like the resolution V (five) first order designs (Table 3) constitutes one group of points for the central composite second order design. The number of these points located at the vertices of a square, cube, hypercube, or fraction of a hypercube is referred to as n_c. The coded independent variable levels for these points are ± 1. Generally a full factorial is utilized if the number of explanatory variables, k is less than 5. If k is between 5 and 7, a ½ is recommended and if k is 8 or greater, a ¼ replicate is recommended (Dykstra 1960).

The defining contrast for the fractional factorials must be carefully selected. An experimental objective usually is to estimate parameters for second order interaction terms as well as the main effects and square terms in the second order polynomial. Therefore resolution V (five) designs are preferred. If lower resolution designs are selected, the defining contrast in Table 3 may not be optimum (Hartley 1959). The experimenter must decide which interactions are most likely to be insignificant and then select a design that leaves those terms as aliases either with main effects or other interaction terms.

The second group of points in a composite design are called star points. These experimental points have coordinates $(\pm \alpha, 0, \cdots, 0)$, $(0, \pm \alpha, 0, \cdots, 0)$, $\cdots$, $(0, \cdots, 0, \pm \alpha)$. The number of these points, n_a is 2 k. Generally the value for α is selected to make the design rotatable. A rotatable design has uniform variance at any given radius from the center of the design. The rotatable condition is satisfied by Eq. 10.

$$\alpha = n_c^{1/4} = 2^{\frac{(k-m)}{4}} \tag{10}$$

The third group of points included in a central composite design are replicated points at the center of the design. These points all have the coordinates $(0, \cdots, 0)$. These center points provide a means for estimating the experimental error and provide a measure of lack of fit with one degree of freedom. The number of center points to be included is sometimes selected for experimenter's convenience or to assure adequate degrees of freedom in the estimate of the experimental error. The number of center points can also be fixed by requiring the design to be orthogonal. Orthogonal designs generally provide the greatest amount of information for estimation of the parameters. For the

design to be orthogonal, the number of center points included in the design, n_o must be given by Eq. 11.

$$n_o = \frac{4\ \alpha^2\ (n_c + \alpha^2)}{n_c} - n_a \tag{11}$$

Or if the design is also rotatable

$$n_o = 4(2^{(k-m)/2}+1) - 2k$$

If the square root of n_c is an integer, the design can be both orthogonal and rotatable. Another method for defining the number of center points is to require the design to provide uniform information. This condition is satisfied when the number of center points is given by Eq. 12.

$$n_o = (n_c + 4\alpha^2 + \frac{4\alpha^4}{n_c})\ \lambda - n_c - n_a \tag{12}$$

where

$$\lambda = \frac{k + 3 + \sqrt{9k^2 + 14k - 7}}{4(k + 2)}$$

The number of points specified by this equation must be rounded to the nearest integer number to give a near uniform information design. The same is true for orthogonal designs when k is an odd integer (3, 5, 7, · · ·).

Table 7 gives the characteristics of uniform and orthogonal rotatable central composite designs for 2 through 8 independent variables.

Table 8 lists the entire design for four factors in an orthogonal rotatable central composite design. This design like all other designs must be performed with the experimental runs in a randomized order. This is important from the standpoint of eliminating the time effect from compounding other effects and may be required to validate the statistical analysis.

San Cristobal Designs. San Cristobal designs (Rojas 1963, 1972) are an interesting variation of composite designs. These designs include the same 2^k factorial or 2^{k-m} fractional factorial points recommended for central composite designs. Two types of San Cristo-

Table 7. Rotatable central composite designs

Number of Independent Variables k	Number of Parameters in the 2nd Order Model p	Number of Cube points $2^{k-m} = n_c$	Star Points		Orthogonal Design		Uniform Information Design	
			Number n_a	Value α	Number of Center Points n_o	Total Number of Points	Number of Center Points n_o	Total Number of Points
2	6	4	4	1.414	8	16	5	13
3	10	8	6	1.682	9	23	6	20
4	15	16	8	2.0	12	36	7	31
5	21	32	10	2.378	17	59	10	52
5 half replicate	21	16	10	2.0	10	36	6	32
6	28	64	12	2.828	24	100	15	91
6 half replicate	28	32	12	2.378	15	59	9	53
7	36	128	14	3.364	35	177	21	163
7 half replicate	36	64	14	2.828	22	100	14	92
8	45	256	16	4.0	52	324	28	300
8 half replicate	45	128	16	3.364	33	177	20	164
8 quarter replicate	45	64	16	2.828	20	100	13	93

Table 8. Orthogonal central composite design for four independent variables

Point Number	Variable Levels			
	x_1	x_2	x_3	x_4
1	+1	+1	+1	+1
2	-1	+1	+1	+1
3	+1	-1	+1	+1
4	-1	-1	+1	+1
5	+1	+1	-1	+1
6	-1	+1	-1	+1
7	+1	-1	-1	+1
8	-1	-1	-1	+1
9	+1	+1	+1	-1
10	-1	+1	+1	-1
11	+1	-1	+1	-1
12	-1	-1	+1	-1
13	+1	+1	-1	-1
14	-1	+1	-1	-1
15	+1	-1	-1	-1
16	-1	-1	-1	-1
17	+2	0	0	0
18	-2	0	0	0
19	0	+2	0	0
20	0	-2	0	0
21	0	0	+2	0
22	0	0	-2	0
23	0	0	0	+2
24	0	0	0	-2
25	0	0	0	0
26	0	0	0	0
27	0	0	0	0
28	0	0	0	0
29	0	0	0	0
30	0	0	0	0
31	0	0	0	0
32	0	0	0	0
33	0	0	0	0
34	0	0	0	0
35	0	0	0	0
36	0	0	0	0

bal designs have evolved (Rojas 1979). The first type does not include center points but has 2k star points $(\alpha, 0, 0, \cdots, 0)$ $(0, \alpha, 0, \cdots, 0), \cdots, (0, 0,0, \cdots, \alpha)$ and $(-1, 0, \cdots, 0), (0, -1, \cdots, 0), \cdots, (0, 0, \cdots, -1)$. The second type has k star points $(\alpha, 0, 0, \cdots, 0), (0, \alpha, 0, \cdots, 0), \cdots, (0, 0, 0, \cdots, \alpha)$ and may have one center point. The α values for these designs (Table 9) were calculated to make the correlations among all pairs of quadratic regression coefficients β_{ii} and β_{jj}, $i \neq j$, equal to zero. All

Table 9. San Cristobal designs

Number of Independent Variables k	Number of Parameters in the 2nd Order Model p	Number of Cube Points $2^{k-m} = n_c$	Type One (Star Points at -1 and α)		Type Two (Star Points at α)		
			Value of α	Total Number of Points	Value of α	Number of Center Points	Total Number of Points
3	10	10	1.26	14	1.34	1	12
4	15	16	1.61	24	1.37	0	20
5 (half replicate)	21	16	1.84	26	1.66	1	22
6 (half replicate)	28	32	2.13	44	1.69	0	38
7 (half replicate)	36	64	2.38	78	1.97	1	72
8 (quarter replicate)	45	64	2.56	80	1.97	0	72

other correlations are close to zero except those between linear, β_i, and quadratic, β_{ii}, coefficients which are all less than 0.30.

The San Cristobal designs were originally developed for fertility experiments. They are a composite design, but permit no treatments below the (-1, -1$\cdots$, -1) level. Thus these designs are useful for exploring an experiment space that is above a given base level in each independent variable. (Alternately, it could explore below the base levels if $-\alpha$ values are used for star points.)

The San Cristobal designs require only 4 design levels for each independent variable instead of the 5 required by central composite designs. In some experiments this is an advantage; however both San Cristobal and central composite designs specify noninteger levels. Noninteger levels are not feasible for some independent variables.

Each of the San Cristobal designs assume the pure error level is known. Although the designs include sufficient points to test for lack-of-fit, there are no repeated points from which the pure error level can be calculated. If the pure error level is not known, one or more points in the design should be replicated.

Smaller near central composite designs have been proposed by Roquemore (1976). These designs are near orthogonal, near rotatable and contain either one or zero degrees of freedom for error. Only rarely are such designs suitable, but if the experiments are extremely expensive, the error level is known and lack of fit is not a serious problem then a Roquemore design should be considered.

Box-Behnken Designs. The Box and Behnken (1960) designs are a valuable alternative to the composite designs. These designs are based on concepts presented by Debaun (1959). Each independent variable is included in the design at 3 levels rather than the 5 levels required for a central composite design or the four levels for a San Cristobal design. The previous designs also require non-integer code levels while the Box-Behnken designs use only integer (-1, 0, +1) code levels. In many industrial and some academic designs, these are important considerations. Generally the number of experimental runs required by the three designs are similar. If the region of interest is best described as a hypersphere, the composite designs usually will be slightly more efficient. If the region of interest is more nearly a hypercube, the Box-Behnken designs are usually more effective (Thompson 1980; Lucas 1976).

In Table 10 designs suitable for investigating 3, 4, 5, 6, 7, and 9 independent variables are given. In this table the symbol $\pm$ 1 means that all combinations of plus and minus levels within this row are to

Table 10. Box-Behnken three level designs[1]

Number of Factors (k)	Design Matrix[2]						Number of Experimental Runs	
3	±1	±1	0				12	
	±1	0	±1					
	0	±1	±1					
	0	0	0				3	
								15
4	±1	±1	0	0			8	
	0	0	±1	±1				
	0	0	0	0			1	
	±1	0	0	±1			8	
	0	±1	±1	0				
	0	0	0	0			1	
	±1	0	±1	0			8	
	0	±1	0	±1				
	0	0	0	0			1	
								27
5	±1	±1	0	0	0		20	
	0	0	±1	±1	0			
	0	±1	0	0	±1			
	±1	0	±1	0	0			
	0	0	0	±1	±1			
	0	0	0	0	0		3	
	0	±1	±1	0	0		20	
	±1	0	0	±1	0			
	0	0	±1	0	±1			
	±1	0	0	0	±1			
	0	±1	0	±1	0			
	0	0	0	0	0		3	
								46
6	±1	±1	0	±1	0	0	24	
	0	±1	±1	0	±1	0		
	0	0	±1	±1	0	±1		
	0	0	0	0	0	0	3	
	±1	0	0	±1	±1	0	24	
	0	±1	0	0	±1	±1		
	±1	0	±1	0	0	±1		
	0	0	0	0	0	0	3	
								54
7	0	0	0	±1	±1	±1	0	
	±1	0	0	0	0	±1	±1	
	0	±1	0	0	±1	0	±1	
	±1	±1	0	±1	0	0	0	56

Table 10. Continued

Number of Factors (k)	Design Matrix[2]									Number of Experimental Runs	
			0	0	±1	±1	0	0	±1		
			±1	0	±1	0	±1	0	0		
			0	±1	±1	0	0	±1	0		
			0	0	0	0	0	0	0	6	
											62
	±1	0	0	±1	0	0	±1	0	0		
	0	±1	0	0	±1	0	0	±1	0	24	
	0	0	±1	0	0	±1	0	0	±1		
	0	0	0	0	0	0	0	0	0	2	
	±1	±1	±1	0	0	0	0	0	0		
	0	0	0	±1	±1	±1	0	0	0	24	
	0	0	0	0	0	0	±1	±1	±1		
	0	0	0	0	0	0	0	0	0	2	
	±1	0	0	0	±1	0	0	0	±1		
9	0	0	±1	±1	0	0	0	±1	0	24	
	0	±1	0	0	0	±1	±1	0	0		
	0	0	0	0	0	0	0	0	0	2	
	±1	0	0	0	0	±1	0	±1	0		
	0	±1	0	±1	0	0	0	0	±1	24	
	0	0	±1	0	±1	0	±1	0	0		
	0	0	0	0	0	0	0	0	0	2	
	±1	0	0	±1	0	0	±1	0	0		
	0	±1	0	0	±1	0	0	±1	0	24	
	0	0	±1	0	0	±1	0	0	±1		
	0	0	0	0	0	0	0	0	0	2	
											130

[1]Box and Behnken (1960)

[2]Lines, ———, separate blocks. The runs within a block must be run in random order and the sequence of the blocks must be random. No randomization of runs between blocks is required

be run. To illustrate this the 3 factor design has been written in detail in Table 11.

Possible points for blocking in these designs have been indicated in the table. All of the runs within a block should be randomized and the blocks, where there are more than 2, should be run in a random order. However, a given block can be completed before starting another block. If there is likely to be a time effect this can be detected by running the experiment in blocks and comparing the response level at the center location for each of the blocks. Central composite designs

Table 11. A three level Box-Behnken design for three independent variables[1]

Point Number	Variable Levels[2]		
	x_1	x_2	x_3
1	+1	+1	0
2	-1	+1	0
3	+1	-1	0
4	-1	-1	0
5	0	0	0
6	+1	0	+1
7	-1	0	+1
8	+1	0	-1
9	-1	0	-1
10	0	0	0
11	0	+1	+1
12	0	-1	+1
13	0	+1	-1
14	0	-1	-1
15	0	0	0

[1]Box and Behnken (1960)
[2]The dashed lines, -----, identify possible blocks. The order of the experimental runs within each block and the order of the blocks must be randomized

can also be run in blocks by first running the cube points with part of the center replicates and then running the star points with the remaining center replicates. A first order equation can be fit to the cube points to determine whether the remainder of the experiment or the second order equation is required. Blocks in both sets may or may not be rotatable or orthogonal.

Hoke Designs. Hoke (1974) proposed several design types for estimating quadratic responses that are based on irregular fractions of the 3^k factorial. Like the Box-Behnken designs, only three integer levels of each independent variable are required. The Hoke designs do not include replicated points for estimating the pure error level and some of the designs do not include any degrees of freedom for error estimation. The latter designs are only used when there are extreme restrictions on the number of experimental units and if there is previous information about the system response.

Two Hoke designs have been recommended because of their high efficiency levels for 3 through 8 independent variables (Lucas 1976). The number of experimental points required for the Hoke designs is significantly less than for the design previously discussed. The two recommended designs (Hoke 1974) are identified as D_2 and D_6. The D_2 design includes only p experimental points and D_6 design includes p +

Table 12. Hoke designs D_2 and D_6

Typical Design Point in Subset	No. of Points in Subset
-1, 0, 0, ···, 0	k
-1, -1, -1, ··· -1	1
-1, 1, 1, ···, 1*	k
1, 1, -1, ···, -1	$\frac{k(k-1)}{2}$
The following subset is included only in the D_6 designs.	
0, 1, 1, ···, 1	k

*if k = 3, this subset should be (-1, -1, 1) to avoid duplicating the following subset

Table 13. The D_6 Hoke design for four independent variables

Point Number	Variable Levels			
	x_1	x_2	x_3	x_4
1	-1	0	0	0
2	0	-1	0	0
3	0	0	-1	0
4	0	0	0	-1
5	-1	-1	-1	-1
6	-1	1	1	1
7	1	-1	1	1
8	1	1	-1	1
9	1	1	1	-1
10	1	1	-1	-1
11	1	-1	1	-1
12	1	-1	-1	1
13	-1	1	1	-1
14	-1	1	-1	1
15	-1	-1	1	1
16	0	1	1	1
17	1	0	1	1
18	1	1	0	1
19	1	1	1	0

k experimental points. Neither design includes replicated points for estimating pure error.

The D_2 and D_6 designs are given in general form in Table 12. Each subset in this table includes all design points that can be formed by reordering the code values in that subset. For example, k = 3, the first subset includes (-1, 0, 0), (0, -1, 0) and (0, 0, -1). The Hoke D_6 design when there are four independent variables is in Table 13.

Table 14. The number of experimental points specified and the G-Efficiencies for each design

Number of Independent Variables (k)	Number of Quadratic Equation Parameters (p)	Number of Experimental Points and (G-Efficiency)						
		Central Composite		San Cristobal		Box-Behnken	Hoke	
		Uniform	Orthogonal	1st	2nd		D_2	D_6
3	10	20 (0.75)	23 (0.65)	14 (0.88)	12 (0.95)	15 (0.89)	10 (1.0)	13 (0.81)
4	15	31 (0.83)	36 (0.71)	24 (0.83)	20 (0.75)	27 (0.95)	15 (1.0)	10 (0.79)
5	21	32 (0.75)	36 (0.66)	26 (0.83)	22 (0.96)	46 (0.91)	21 (1.0)	36 (0.82)
6	28	53 (0.84)	59 (0.75)	44 (0.76)	38 (0.74)	54 (0.92)	28 (1.0)	34 (0.83)
7	36	92 (0.72)	100 (0.66)	78 (0.54)*	72 (0.53)	62 (0.93)	36 (1.0)	43 (0.84)
8	45	93 (0.88)	100 (0.82)	80 (0.64)	72 (0.63)	(not recommended)	45 (1.0)	53 (0.85)
Error degrees of freedom		Yes	Yes	only lack of fit	only lack of fit	Yes	non	only lack of fit

*Does not meet the Wynn (1972) and Atwood (1972) criteria for G-efficiency

Table 14 compares the number of experimental points required for the quadratic response designs discussed in this section. The smallest fractional replicate of the cube points that will estimate all parameters was assumed for composite designs. Other important characteristics must be considered when selecting a design, but the table helps identify relative costs for these designs.

Table 14 also compares the G-efficiencies for these quadratic response designs. All of the designs meet the Wheeler (1972) criteria and all but one design meets the Wynn (1972) and Atwood (1972) criteria. Thus efficiency should not be a major factor when selecting a design from this group.

The numbers in Table 14 should not be used to directly compare the methods because of the differences in degrees of freedom for error. Generally the designs with fewer points do not have replicated points for estimating pure error. In the extreme case, Hoke D_2, neither pure error or lack of fit can be estimated. If these estimates are needed, more points would have to be added to the San Cristobal and Hoke designs and the efficiencies would decrease.

ANALYSIS

After the data has been collected in a response surface experiment, the next step usually is to estimate model parameters. Normally this estimation is done by linear least squares regression. Most computer systems have a number of regression routines available to the user and many statistics books outline regression procedures. The texts by Draper and Smith (1981) and by Weisberg (1980) are frequently cited authorities on regression. Most regression routines provide an analysis of variance but leave the subdivision of the residual term into pure experimental error and lack of fit to the experimenter. If a point is replicated n_o times, the analysis of variance for the regression should be similar to Table 15.

From the information in this table both the lack of fit and the value of the model equation can be tested with the F statistic. The value calculated for F_{1of} (Eq. 13) should be compared with the table value, $F(n\text{-}p\text{-}n_o\text{+}1, n_o\text{-}1, s_1)$. If the value of F_{1of} is the larger of the terms, there is a significant lack of fit with this particular model. The significance of the regression equation can be estimated by comparing the results of Eq. 14 with the table value, $F(p\text{-}1, n\text{-}p, s_1)$. Generally, the calculated F value from Eq. 14 should be several times the tabled value if the model is a good predictor of the experimental results (Wetz 1964).

$$F_{lof} = \frac{MS_l}{s^2} \tag{13}$$

$$F = \frac{MS_r}{s^2} \tag{14}$$

All these statistics assume either normally distributed errors or random experiment order. The statistics also assume that there are no biases contributed by the experimenters. In general, this means that the individuals performing the experiments and analyzing the results should have no more than the absolutely essential knowledge about characteristics of each experimental run. Knowledgeable experimenters will have preconceived ideas about the probable results and this inevitably biases the experiment.

There are several linear regression procedures and additional statistics that can be used for response surface analysis. These have been reviewed by Hocking (1976) and therefore will only be briefly mentioned here. Not all of the terms in a mathematical model are needed to explain typical experimental data. The two general regression approaches for selecting the most important terms are stepwise and all possible models. A stepwise procedure either adds model terms one at a time, selecting the term with the highest sequential F value at each step (forward selection) or starts with all terms in the equation and eliminates terms one at a time by removing the term with the lowest sequential F value (backward elimination). Stepwise regression is appropriate when the primary purpose of the model is to describe the experimental results. The statistics watched most closely

Table 15. Analysis of variance for response surface regression

Source of Variation	Sum of Squares	Degrees of Freedom (df)	Mean Square
Total (uncorrected)	$\overline{y}' \overline{y}$	n	
Mean (β_0)	$\frac{1}{n}\sum_{i=1}^{n} y_i^2$	1	
Total (corrected for the mean)	by subtraction, TSS	n-1	
Due to Regression/β_0	$R_eSS = \overline{\beta}' X' \overline{y}$	p-1	MS_r
Residual	by subtraction, RSS	n-p	
Pure Error	$ESS = \sum_{i=1}^{n_o}(y_{oi} - \overline{y}_o)$	n_o-1	s^2
Lack of Fit	by subtraction, LSS	n-p-n_o+1	MS_l

in a stepwise regression include the residual mean square (s^2), the sequential F, the coefficient of multiple determination (R^2), and the R^2 adjusted for the number of parameters in the equation, (R^2_a).

$$R^2 = \frac{R_eSS}{TSS} \tag{15}$$

$$R^2_a = 1 - (n-1)\ (1-R^2)/(n-p) \tag{16}$$

Generally, it is desirable to have the residual mean square as small as possible. However, terms that cause very little reduction in the residual mean square are probably contributing very little to the description of the data. The sequential F value indicates which terms are significant in the response equation. The coefficient of multiple determination and adjusted R^2 indicate the fraction of the variation about the mean response level that is being explained by the regression equation. If the change in the coefficient for the addition of a term is small, the term is of limited value as a descriptor of the experimental results.

All possible regression equations should be considered if the purpose of the model is prediction, process control or model discrimination. All possible regression programs try fitting the data to all possible combinations of the terms in the model equation. These tentative models are then compared and the best ones selected for validation. The statistics usually observed in an all possible regression analysis include the residual mean square, the adjusted R^2, the average prediction variance (J) and the total squared error (C_p) which is also called the C_p statistic.

$$J = (n+p)\ s^2/n \tag{17}$$

$$C_p = RSS/E(s^2)+2p - n \tag{18}$$

The average prediction variance, J, can be treated much like the residual mean square, but it has been adjusted to negate the normal reduction in value caused by increased number of parameters. The C_p statistic for a good model should have a value approximately equal to the number of parameters included in the model. Its value for a good model should never exceed the number of parameters and should be at least equal to p/2 (Daniel and Wood 1971).

Validation of models cannot be overemphasized. A model fit to one

set of data even though there are numerous degrees of freedom for error, can misrepresent the true response of the system. A small replicate of a factorial experiment should be run in the experimental space of the original experiment and those results compared with model predictions. If the model response level is plotted against the experimental response level for the validating experiments, the points should fall in a straight line intersecting the origin and having a slope of 1. Slightly positive intercepts and slopes somewhat less than 1 are usually found because of a bias inherent in regression procedures (Draper and Smith 1981; Davies and Hutton 1975).

Most model equations are too complex for the average person to visualize the characteristics of the response surface. If computer equipment is available for drawing contour plots representing the model equation, this should be done. Without the assistance of a computer to draw the contour plots, a canonical analysis should be considered (John 1971). If the experiment is to be scaled-up to a full scale process or equipment, the general contours observed in the experimental work will probably remain nearly constant. The response may shift or the optimum location in the independent variables may shift, but the shape of the contours usually remains similar between experiments and full scale operations.

NOTATION

Symbol	Definition
C_p	The total squared error, also called the C_p statistic.
$E(s^2)$	The estimated experimental variance based on a model with all parameters included.
ESS	Pure (experimental) error sum of squares.
E(y)	Expected or predicted response level.
J	The average prediction variance.
k	The number of independent variables in the experiment.
LSS	Lack of fit sum of squares.
m	Indicates a fraction replication of a factorial experiment. The level of the replication is $(\frac{1}{2})^m$. For example a ½ replicate is represented by m = 1 and a ¼ replicate by m =2.
MS_l	Lack of fit mean square.
MS_r	Mean square due to regression.
n	The total number of experimental points in the experimental design.

n_a The number of star points in a composite design.
n_c The number of experimental points in a 2^k factorial or 2^{k-m} fractional factorial part of a central composite design.
n_o The number of center points in a composite or 3 level factorial design.
p The number of parameters in the model equation.
R^2 Coefficient of multiple determination.
R_a^2 Adjusted R^2 value.
r_i Any positive or negative constant associated with variable i.
RSS Residual sum of squares.
R_eSS Regression sum of squares.
s_1 The level of significance being tested in an F test. Usually 0.95 or 0.99.
s^2 Pure error mean square or residual mean square. Estimates response variance.
TSS The total sum of squares corrected for the mean.
X An nxp matrix with row i containing the statistical model evaluated at design location i.
$x_{o,i}$ A base or center level for explanatory variable i.
x_i, x_j The level of explanatory variable i or j.
$\overline{y}$ A column vector of observations, (nx1).
y_i The observed response level for experimental run i.
y_o A base value for the dependent or response variable.
$\overline{y}_o$ The mean response at the center of the design.
y_{oi} The response to the i^{th} repeated experiment at the center of the design.

GREEK NOTATION

Symbol	Definition
α	The independent variable level for a star point in a composite design. If the design is rotatable, $\alpha = n_c^{1/4}$.
β_i, β_{ij}	Coefficient or parameter in a model.
$\overline{\beta}$	A column vector of estimated parameter values, (p × 1).
$\overline{\epsilon}$	An n × 1 column vector of random errors.
Σ	Summation.

Note: The mark ′ implies a transpose of a previous vector or matrix.

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EVALUATION OF TEXTURE AND WATER HOLDING CAPACITY IN COOKED MINCED FISH

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ABSTRACT

*A meat sausage type batter system consisting of mechanically deboned sucker (*Catostomidae fam.*) flesh and binder ingredients was evaluated for texture and water holding capacity (WHC) by various techniques. The Instron Universal testing instrument for texture and the centrifuge method for WHC were found to be the most reliable procedures to evaluate the cooked fish matrix.*

Although texture and WHC were improved by the addition of salt and soy protein isolate (SPI) or sodium caseinate (SC), the firmness characteristics of the cooked flesh matrix seemed to depend greatly on the nature of the fish and type of cooking.

INTRODUCTION

Sucker fish (*Catostomidae fam.*) is one of the underutilized freshwater species from the Great Lakes area with great production potential. Nevertheless, some of the defects which preclude their gaining consumer acceptance when marketed in the conventional foms are poor morphological characteristics, lack of appealing sensory properties, and an abundance of small bones.

Mechanical deboners have attracted a considerable amount of attention from major fish processors. However, in spite of the great processing possibilities offered by these machines, many problems have hindered the realization of their full utilization (Nakayama and Yamamoto 1977). It has been suggested that before underutilized fish

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can be evaluated for potential use in processed food products certain basic information on species is needed to help determine whether the fishery resource is acceptable as human food and can justify the processing equipment and facilities (Miyauchi 1975).

The work described herein consisted of a study of some of the methods for measuring texture and WHC as indicators of the functional behavior of the sucker flesh. The effects of some processing variables on texture and WHC of the mechanically deboned sucker flesh were also studied.

MATERIALS AND METHODS

A catch of about 600 lb of fresh sucker from Lake Huron was handled on ice for about 72 h before processing. The batch consisted of about 50% each Silver Redhorse sucker, *Moxostoma anisurum* (Rafinesque) and White sucker, *Catostomus commersoni* (Lacepede), (Eddy 1974).

Fish were dressed and mechanically deboned and the flesh was then packaged into Cryovac® bags and stored in a -29°C blast freezer for further use in small experimental batches.

The thawed minced flesh was then blended with different binders. Regular binder (RB) was defined, for the purpose of this study, as a mixture of ingredients used along with the minced sucker flesh in the following concentrations: salt 1.0%; sugar 1.0%; corn oil 1.0%; fish muscle 2.5%; ice 5.0%; monosodium glutamate 0.3%; sodium tripolyphosphate 0.15% and sodium ascorbate 0.04%. Other binders used were soy protein isolate (SPI), Cenpro-P, Central Soya, Chicago, IL. and sodium caseinate (SC), Milk Proteins Inc., Detroit, MI.

Blending of sucker flesh with binders was achieved in either a food cutter, model 84181D, for two min or in a Kitchen Aid food preparer, Model N-50, with a paddle attachment for 15 min. Both machines were manufacturered by Hobart Mfg. Co., Troy, OH.

Cooking of the fish paste in water bath was carried out by weighing 25g material into 50 ml open plastic centrifuge tubes in a 70°C water bath for 30 min. Smokehouse cooking was achieved after stuffing and linking the fish paste into frankfurter type casings (25 mm diam.) according to the following schedule: 10 min at 54.5°C, 25% RH; 20 min at 60°C, 35% RH; 20 min at 65.5°C, 40% RH; 20 min at 71.1°C, 65% RH and 15 min at 79.4°C, 65% RH.

Texture of the fish matrix was estimated on the smokehouse cooked product by using the following devices: (a) the Kramer Shear Press;

Food Technology Corp., Reston, VA, equipped with a compression cell; (b) The Instron universal testing instrument, model TTB, Instron Corp., Canton, MA, with a compression load cell, range 1 to 50 kg. and (c) The Universal Penetrometer, Arthur H. Thomas Co., Philadelphia, PA, equipped with a 35 g penetration cone. For the Kramer Shear Press (a) shear-compression measurements fish matrix samples (frankfurter size) were cut into 8 cm long cylinders weighed (≈60 g) and subjected to shear. The results were expressed as lbs force per gram of sample. For the Instron shear determination cylindrical samples were placed in a brace and sheared by a Warner-Bratzler type (triangular) blade. The instrument was calibrated with a 1 kg weight for full scale displacement with the drive and recording chart adjusted to 20 cm/min. The shear resistance force was expressed as kg force per cm of sample diameter. Penetration was measured as mm depth of penetration per cm diam. Five, 10, and 15 replicate measurements were done for the Kramer, Instron and penetration tests, respectively.

The WHC of the fish matrix was estimated by (a) the filter paper press method, according to the gravimetric adaptation by Karmas and Turk (1975) to the original technique by Wierbicki and Deatherage (1958); (b) a centrifuge technique, as described by Bremner (1977).

RESULTS AND DISCUSSION

Evaluation of texture characteristics on the fish matrix was carried out according to the following criteria. The higher the shear force value obtained by using the Instron or the Kramer instruments the more firm or hard the fish matrix. Firmness was associated with desirable texture of the product since sucker tended to be mushy. Using the penetrometer for softness of the product, the higher the penetration value obtained the poorer the firmness of the fish matrix.

The Instron and Kramer instruments seemed to be more reliable than the penetrometer in estimating the texture of the sucker matrix based on the fact that more significant differences among binder treatments were detected by these methods (Table 1). However, there were no significant differences in correlation between Instron and penetrometer with WHC measures by the centrifuge technique (Table 3). The Instron machine, in addition, proved to be more automated permitting faster determinations.

WHC was evaluated in terms of water losses percent (Table 2); thus the higher the water losses the poorer was the water retention of the

Table 1. Mean and standard deviation of texture values as determined on the smokehouse cooked sucker matrix[1]

Procedure For Texture and Units	Binder Treatments								
	RB	2% SPI	3% SC	RB + 2% SPI	RB + 2% SC	RB + 2% NaCl	RB + 2% NaCl (Paddle Mix)	RB + 2% NaCl + 2% SPI	RB + 2% NaCl + 2% SC
Shear force by Instron, Kg-f/1.6 cm ϕ of fish core N = 8	$1.9^{a} \pm 0.2$	$2.6^{cde} \pm 0.3$	$2.4^{bcde} \pm 0.1$	$4.1^{h} \pm 0.3$	$3.4^{fg} \pm 0.1$	$2.2^{abcd} \pm 0.2$	$2.6^{de} \pm 0.2$	$3.6^{g} \pm 0.1$	$2.7^{e} \pm 0.1$
Shear force by Kramer, lb-f/g fish core. N = 5	$.45^{a} \pm 0.1$	$.87^{g} \pm 0.1$	$.70^{bcdef} \pm 0.1$	$1.0^{h} \pm 0.1$	$.73^{def} \pm 0.1$	$.72^{cdef} \pm 0.1$	$.75^{e} \pm 0.1$	$1.0^{h} \pm 0.1$	$.76^{fg} \pm 0.1$
Softness by penetrometer, mm penetration	$10.7^{g} \pm 0.4$	$9.5^{ef} \pm 0.6$	$9.5^{ef} \pm 0.2$	$7.2^{a} \pm 0.2$	$8.7^{cd} \pm 0.5$	$9.9^{f} \pm 0.6$	$9.9^{f} \pm 0.3$	$8.3^{bc} \pm 0.7$	$9.2^{def} \pm 0.3$

[1]Means in same row not followed by the same superscript are significantly different ($P<0.01$)

Table 2. Mean and standard deviation of water-holding capacity (WHC) of cooked sucker flesh, expressed as water losses percent

Procedure for Water-Holding Capacity (WHC)	Binder Treatments				
	RB	RB + 2% NaCl	RB + 2% NaCl (Paddle Mix)	RB + 2% NaCl + 2% SPI	RB + 2% NaCl + 2% SC
Filter paper press on water bath cooked fish N = 4	$51.8^{c} \pm 2.2$	$50.7^{bc} \pm 2.5$	$48.5^{abc} \pm 2.8$	$48.8^{abc} \pm 4.8$	43.1 ± 3.1
Filter paper press on smoke-house cooked fish N = 4	$43.1^{ab} \pm 1.5$	$38.7^{a} \pm 3.4$	$43.8^{b} \pm 2.3$	$42.5^{ab} \pm 0.9$	$42.5^{ab} \pm 2.3$
Centrifuge technique on water bath cooked flesh N = 3	$38.4^{d} \pm 0.5$	$37.3^{cd} \pm 0.5$	$36.0^{bcd} \pm 1.7$	$30.3^{a} \pm 1.1$	$33.9^{ab} \pm 1.0$
Centrifuge technique on smoke-house cooked flesh N = 3	$34.9^{bc} \pm 1.3$	$33.1^{bc} \pm 2.1$	$29.8^{ab} \pm 1.2$	$27.3^{a} \pm 1.0$	$28.3^{ab} \pm 1.0$

[1]Means in same row not followed by the same superscript are significantly different ($P<0.05$)

Table 3. Correlation coefficients (r values) between selected measures of functional qualty of sucker flesh

Parameter	1	2	3	4	5	6	7
1. Instron shear force	—						
2. Kramer shear force	.72**	—					
3. Penetrometer depth	.30	-.86**	—				
4. Cooking shrink	.27	-.10	.67**	—			
5. WHC - SM[1] by filter paper	.18	.05	-.02	-.25	—		
6. WHC - WB[2] by filter paper	-.63**	-.13	-.02	.26	-.50	—	
7. WHC - SM[1] by centrifuge	-.90**	-.85*	.92**	.98**	-.30	.23	—
8. WHC - WB[2] by centrifuge	-.99**	-.86**	.97**	.81*	-.21	.10	.91**

*significant at 5% level
**significant at 1% level
[1]SM = smokehouse oven cooked
[2]WB = water bath cooked

fish system. The centrifuge technique proved to be more accurate in predicting texture and easier to use than the filter paper press method. Some of the disadvantages of the latter, in this study, were the tedious removal of the fish flesh residue from the filter paper following the pressing step and the fast water evaporation during the handling of the filter paper before weighing. As can be noted in Table 3, the WHC as evaluated by the centrifuge method for flesh cooked 2 ways was significantly correlated with firmness or texture values by each technique. Such was not the case with WHC estimated by the filter paper method.

Fish flesh with RB was considered the control system for the binder effect evaluation. Table 1 shows an improvement in the texture characteristics of the sucker flesh by addition of either SPI, SC, or salt. The firmest product was obtained when 2% SPI plus 2% salt were used along with RB in fish emulsion. This effect was apparent by using any of the three texture techniques described in this study.

Table 2 shows the effects of some of the binders on WHC characteristics of the cooked sucker flesh. No important WHC improvement could be observed by using the filter paper press technique. However, a significantly better water retention in the product with RB plus 2% salt plus 2% SPI was detected when the more reliable centrifuge method was used.

Table 1 also shows that SPI had a greater effect on the texture characteristics of the fish product than SC. A similar effect of these two proteinaceous binders on cooked fish WHC had been observed by Karmas and Turk (1976).

The paddle mixing system used in adding the binder to the minced

sucker flesh showed no improvement in texture or WHC characteristics over the cutting emulsifying system.

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BOOK REVIEWS

Source Book of Flavors, H. B. Heath, AVI Publishing Co. Inc., Westport, Conn. 863 pp. 1981. $85.00.

This book touches on nearly every aspect of the flavor industry except for flavor applications. Subjects include a general perspective of the flavor industry, the flavor chemist, flavor research, chemistry of flavor formation in nature, theories of odor and taste, food colorants (both natural and synthetic), manufacturing methods, quality assurance, international flavor legislation, toxicology, consumer safety and worldwide labelling regulations. The text also includes nearly 150 pages of discussion on natural flavoring materials. Treatment varies from only a brief description of the plant to a rather detailed outline of the plant, typical processing techniques and specifications of the finished flavoring material. Depth of treatment is determined by the importance of the plant as a flavoring material. The last section of this book is basically a reprinting of Merory's book *Food Flavorings: Composition, Manufacture and Use.* This section includes formulations (and processing techniques) for natural flavorings and extracts, imitation flavorings and flavorings for specific applications and product groups. The value of reprinting Merory's text is questionable since technology and compounds permitted for use in artificial flavors has changed greatly since Merory first published his text in 1960. However, I do agree with Mr. Heath that the section still has value to teach principles and provide starting points for the novice.

The positive points of this book include the tremendous breadth of information compiled in one text. There truly is no previously published counterpart to this text. In addition, Mr. Heath has done a very good job of coverage in each of these areas. It is impressive that the text includes over 3,000 references. If the text does not include the necessary depth, references can lead the reader to more detail. The book is well written and reasonably free of errors.

The only shortcomings of the book are due to the breadth of the subject matter. A single author cannot be an expert in all aspects of a subject as broad as flavors. This results in some subjects (eg. flavor research and chemistry) not meeting the same standards as the rest of the text. Also, while one may commend Mr. Heath on his excellent treatment of flavors, his experience has primarily been with European flavors and the European market. His viewpoint at times, shows this slant.

It is this reviewer's opinion that the *Source Book of Flavors* would be a very valuable asset to any individual working in or with flavors. There is no other single book which rivals the coverage of this book. It is well worth the price.

DR. GARY REINECCIUS

Developments in Food Microbiology—1. R. Davies, Editor. Applied Science Publishers, Inc. Englewood, N.J. 07631. 1982.

This book represents the beginning of another in the Development Series offered by this particular publisher. I assume this is the first of a series as implied by the 1 in the title. The contents are a series of monographs by recognized authorities in the particular area in which they are writing. As with most books of this type, written by many, and edited by one, there are some inconsistencies between the chapters and style differences among the authors.

The book consists of six chapters, each by different authors. The first chapter, "Microbial Spoilage of Meats," by T. A. McMeekin, discusses, at some length, the microorganisms responsible for meat spoilage at refrigeration temperatures. This author reviews the information concerning the use of the terms "psychrotrophic" and "psychrophillic" to describe those microbes that can grow at refrigeration temperatures. It is the reviewers opinion that the use of these two terms is confusing and what these microbes are called is of interest only to us academicians. The important point, regardless of what the microbes are named, is that they have the capacity to grow on flesh foods in the cold and render the food unacceptable to man. The author illustrates his discussion with several excellent scanning electron micrographs showing microorganisms on the surface of flesh foods.

"The Nurmi Concept and Its Role in the Control of Salmonellae in Poultry" is the title of the second chapter. The authors, H. Pivnick and E. Nurmi, present some interesting data and discussion on the topic. They are convinced that the Nurmi concept does an excellent job of controlling Salmonellae in poultry. The data presented seems to support their conviction.

G. Hobbs and W. Hodgkiss author the third chapter on "The Bacteriology of Fish Handling and Processing." This review is an excellent one and summarizes in one spot a great deal of information on this topic. I found it to be both interesting and informative to peruse.

The use of ultra high temperature processing is discussed by K. L.

Brown and C. A. Ayres in Chapter 4 entitled "Thermobacteriology of UHT Processed Foods." The authors indicate, and I agree, that the literature dealing with this topic is voluminous and overwhelming. Nevertheless, they have done an excellent job in summarizing the literature pertinent to the topic.

One of the foremost authorities on the topic, L. L. McKay, has written chapter 5 on "Regulation of Lactose Metabolism in Dairy Streptococci." As he usually does, Dr. McKay has summarized the topic succinctly in an understandable manner. If you've been away from this topic for some time, read this chapter, it gives sound information. This chapter alone is worth the price of the book.

A topic of interest to all microbiologists interested in rapid enumeration of microbes is presented in Chapter 6. "New Developments in the Rapid Estimation of Microbial Populations in Foods" was written by J. M. Wood and P. A. Gibbs. These authors summarize in some detail the recent developments in enumerating microorganisms in food. Some interesting ideas and concepts are presented.

This book and others like it are useful as reference as they review the literature of recent years on a particular topic or topics. They do become outdated in a few years and of little use, thus the need to write another develops. I would recommend it as a reference and perhaps as a basis for a seminar but not as a text book for a course in food microbiology.

EDMUND A. ZOTTOLA, Ph.D.

Flavour '81, 3rd Weurman Symposium Proceedings of the International Conference. P. Schreier editor. Walter de Gruyter Co., Genthiner Str. 13, 1000 Berlin, West Germany. 1981. 780 pp.

Flavour '81 is a publication of the papers which were presented at the 3rd Weurman Symposium in April 1981, Munich, West Germany. The main topics encompassed by this symposium were: sensory methodology, application of sensory methods, instrumental analysis, formation of flavor, applications and technology and molecular aspects of flavor. A total of 57 individual presentations are included in this book. Since this work is an assembly of individual efforts, wide variation is found between individual papers in depth of presentation. The papers are, however, generally well written and informative.

It is of interest that only 7 of the 57 papers were authored by researchers from the USA. While one could be critical of the low US representation, this reviewer welcomes the broad coverage of

European work. The problems associated with obtaining and translating articles in foreign languages often results in their neglect.

The breadth of subject matter covered by this text is too broad to convey in a brief review. A few chapter titles would include: A critical review of threshold intensity and descriptive analysis in flavor research (by Pangborn); Sensory evaluation in a natural environment (by Koster); Perception and analysis: a perspective view of attempts to find casual relationships between sensory and objective data sets (by Powers); A search for a nervous code for odor quality combining analytical and electrophysiological methods: a model of a biotest (by Selzer and Christoph); Recent developments in high resolution gas chromatography (by Jennings); Isolation and properties of bitter-sensitive proteins via affinity chromatography (by Gatfield); Formation of flavor components from proline and hydroxyproline with glucose and maltose and their importance to food flavor (by Tressel *et al.*); sunlight flavours in Champagne wines (by Charpentier and Maujean); Aspects of the development of industrial flavor materials (by Emberger); and Bifunctional unit concept in flavour chemistry (by Ohloff).

In this reviewers opinion, anyone working in any area of flavor will find papers of interest in this compilation. While the book is expensive ($99) it contains a great deal of useful information and is well worth the price.

G. A. REINECCIUS

ERRATUM

A correction was discovered in the *Journal of Food Processing and Preservation* Vol. 6, No. 1 in the paper: Development and Stability of Intermediate Moisture Cheese Analogs from Isolated Soybean Proteins by M. Motoki, J. A. Torres and Marcus Karel. In Table 7, sample 5 is reported to give 9 days to organoleptic unacceptability. The correct value is 90 days.

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JOURNALS AND BOOKS IN FOOD SCIENCE AND NUTRITION

Journals

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JOURNAL OF FOOD BIOCHEMISTRY, H. O. Hultin, N. F. Haard and J. R. Whitaker

JOURNAL OF FOOD PROCESS ENGINEERING, D. R. Heldman

JOURNAL OF FOOD PROCESSING AND PRESERVATION, T. P. Labuza

JOURNAL OF FOOD QUALITY, A. Kramer and M. P. DeFigueiredo

JOURNAL OF FOOD SAFETY, M. Solberg and J. D. Rosen

JOURNAL OF TEXTURE STUDIES, P. Sherman and M. C. Bourne

Books

ANTINUTRIENTS AND NATURAL TOXICANTS IN FOOD, R. L. Ory

UTILIZATION OF PROTEIN RESOURCES, D. W. Stanley, E. D. Murray and D. H. Lees

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FOOD SCIENCE AND TECHNOLOGY, 3RD ED., M. Pyke

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HASSON, E. P. and LATIES, G. G. 1976. Separation and characterization of potato lipid acylhydrolases. Plant Physiol. 57, 142–147.

ZABORSKY, O. 1973. *Immobilized Enzymes*, pp. 28–46, CRC Press, Cleveland, Ohio.

Journal abbreviations should follow those used in *Chemical Abstracts*. Responsibility for the accuracy of citations rests entirely with the author(s). References to papers in press should indicate the name of the journal and should only be used for papers that have been accepted for publication. Submitted papers should be referred to by such terms as "unpublished observations" or "private communication." However, these last should be used only when absolutely necessary.

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