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D.R. HELDMAN
and
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DENSITY AND COMPRESSIBILITY OF SELECTED FOOD POWDERS MIXTURES[1]

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ABSTRACT

The density and compressibility of various mixtures of granular and powdered sucrose, granular malic acid, soy protein and cornstarch, at 1:3, 1:1 and 3:1 were determined and compared to those of the pure components. Unless the particles were of similar size and properties (e.g., granular sucrose and malic acid) and density and/or compressibility could not be deduced from those of the pure ingredient, as a result of the formation of new bed structures. This was particularly the case in powders exposed to elevated relative humidity conditions, in this work $a_w = 0.65$. *Because compressibility can reflect changes in the bed structure as well as deformation of the particles themselves, no correlation could be found between loose bulk density and compressibility. Therefore, each mixture ought to be treated as a new powder the bulk properties of which are determined by their particular microstructure and the water moisture of their components.*

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[1] Supported by the Particulate and Multiphase Processes Program of the NSF Grant Nos. CPE-8206765 and CBT-8520370.

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INTRODUCTION

It has long been established that the bulk density, compressibility and flowability of food powders are related physical properties (Peleg 1983). They are largely influenced by the powder chemical composition and particle size and to an even greater extent by moisture. The effect of these factors has been extensively studied in various food powders (Sone 1972; Schubert 1987). It was found, Moreyra and Peleg 1981, that in general, the more cohesive the powder is, as a result of small size or moisture absorption, the lower is its bulk density (up to a limit) and the higher is its compressibility. Anticaking agents or flow conditioners that effectively reduce powders cohesiveness and improve flowability frequently also cause, as could be expected, an increase in bulk density and a decrease in compressibility (Peleg and Hollenbach 1981).

There are little published analyses on the effects of mixing on the bulk properties of food powder mixtures. A notable exception is the behavior of conditioned powders, i.e., a powder to which an anticaking agent has been added. Such powders are mixtures by definition, and according to modern classifications are known to the "ordered mixtures" in the senses that all the agent's particles are found absorbed on the host particles surface (see below). Consequently, such powders behave as if they were made of a single component with particles of a different kinds. Photographs and a discussion of the various microstructural features of conditioned powders and food powder mixtures have recently been published by Peleg and Hollenbach (1984) and Barbosa Canovas *et al.* (1985). Because, as mentioned, quantitative data on the behavior of mixtures is rather scarce, the objective of this work was to determine the bulk properties of mixtures of common ingredients and to characterize their features in light in interparticle interactions and the moisture of their components.

EXPERIMENTAL

Materials

Commercial granular sugar (from a local store), malic acid (Alberta Gas Chemical, Parsippany, NJ), precipitated isolated soy starch (Cargill, Inc., Minneapolis, MN) and domestic cornstarch (Staley Mfg. Co., Oak Brook, IL) were mixed at ambient conditions to form mixtures of various weight ratios. Part of these mixtures were tested without further treatment. Another part was tested after equilibration at ERH of 65% using an evacuated desiccator with saturated $NaNO_3$ solution (Rockland 1975; Greenspan 1977). Some of the granular sugar was pulverized by a laboratory mill (Micro-Mill, Technilab Instruments, Pequanoc, NJ) and sieved to obtain a USTM +70 −140 mesh fraction (105–74 micron). This powdered sucrose was mixed and tested in the same manner as the

TABLE 1.
THE MOISTURE (IN PERCENT) OF THE POWDER MIXTURES WHOSE BULK PROPERTIES WERE EVALUATED IN THIS WORK

Powder A[1]	Powder B[1]	ERH (%)	Weight fraction of A (%)				
			0 (Pure B)	25	50	75	100 (Pure A)
Granular sucrose	granular malic acid	ambient	0.20	0.30	0.18	0.14	0.10
		(65)	0.20	0.21	0.24	0.20	0.20
Granular sucrose	cornstarch	ambient	10.7	7.7	5.1	2.5	0.1
		(65)	11.4	8.8	6.2	2.8	0.2
Powdered sucrose (+140-200 mesh)	cornstarch	ambient	10.6	7.2	4.4	2.4	0.1
		(65)	11.4	9.5	7.5	5.9	0.1
Granular sucrose	soy protein	ambient	6.3	5.6	3.7	2.1	0.1
		(65)	10.3	7.4	4.9	2.4	0.2
Powdered sucrose	soy protein	ambient	6.3	5.8	4.1	1.7	0.1
		(65)	10.7	7.9	4.8	2.6	0.1
Cornstarch	soy protein	ambient	10.6	12.8	10.0	9.0	6.3
		(65)	11.4	11.5	11.6	11.2	10.3

[1] The sieve size of the powders was: granular sucrose - +30–50 mesh, powdered sucrose - +140–270 mesh, malic acid - +30–50 mesh, cornstarch - +140–200 mesh and soy protein - +50–70 mesh.

granular sugar. The mixing ratios and the powders' moisture are summarized in Table 1. They were mixed in a laboratory V-tumbling mixer (Patterson-Kelley Co., East Stroudsburg, PA) for about 10–15 min.

Density and Compressibility Determination

Loose bulk density was determined by pouring the powder into a cylindrical container, scraping off the excess and dividing the net weight of the powder by the volume of the container. Compressibility was determined by compression of a powder specimen by an Instron Universal testing machine. The specimen was confined in a metal cell and was covered by a specially designed lid that enables point loading during the compression. Detailed description of the cell and the procedure can be found in Moreyra and Peleg (1980). The force deformation curves, in the range of up to about 5 kg cm^{-2} were converted to normalized density-normal stress relationships. These were found to fit the following equation (Malave *et al.* 1985).

$$\frac{\rho(\sigma)-\rho_O}{\rho_O} = C_1 + C_2 \log \sigma_N \qquad (1)$$

where $\rho(\sigma)$ is the density at a normal stress σ, ρo the initial bulk density and C_1 and C_2 constants. The constant C_1 represents the density increase under a unit stress and the constant C_2 its compressibility.

All density and compressibility data are means of three replicates.

RESULTS AND DISCUSSION

Density-Structure Relationships in Powder Mixtures

Theoretically, binary powder mixtures can have different kinds of microstructure depending on the relative particle size of the components. Few of these are shown schematically in Fig. 1. They basically depict two major types, the so called "random mixtures", Fig. 1 a,b,c and d and "ordered mixtures", Fig. 1e and f, (Hersey 1975). The term "random" refers to the fact that all the particles, irrespective of their species have the same probability of occupying any given location in the powder bed. The term "ordered" refers to the fact that the particles of the finer component can only be found attached to the surface of the larger particles and as a result lose their ability to occupy any point in space in a random fashion. This, it should be pointed out, is a rather simplistic classification scheme. It has recently been suggested that more appropriate terms are

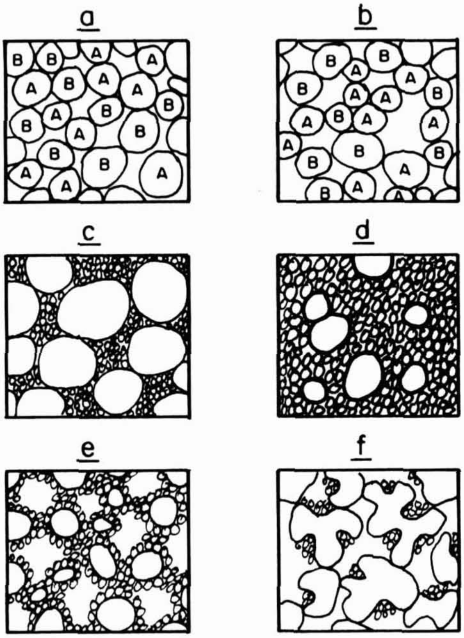

FIG. 1. SCHEMATIC VIEW OF VARIOUS TYPES OF POWDER MIXTURE MICROSTRUCTURE
(A and B represent particles of similar size but of different chemical species).
See text for explanation.

"noninteractive" and "interactive" mixtures (Egerman and Orr 1983). It was also demonstrated that there are food and nonfood powders, the structure of which has the combined features of both types, e.g. partially ordered random mixtures, etc. For more details and micrographs of such mixtures see the work of Barbosa-Canovas *et al.* (1985).

Another feature of the mixtures shown in Fig. 1 that is of direct concern to the present report is the space filling characteristics of the structure. Fig. 1a and b depict mixtures the density of which is expected to be equal to that of either component (A and B) provided their particle density is roughly the same. Fig. 1c depicts an array where the density of the mixture is bigger than that of the component with the larger particle size since the interparticle space is filled with fines. The maximum possible density of such mixtures and the concentration of the fines that will produce it can easily be calculated when the density of the two components is known. If the bulk density of the coarse and fine components are ρ_c and ρ_f respectively, then the maximum possible density, ρ_{max}, of the mixture provided that each component retains its bulk characteristics, is given by:

$$\rho_{max} = \rho_c + \epsilon\rho_f \tag{2}$$

where ϵ is the void ratio.

The results for the mixtures tested in this work are shown in Table 3, (based on the data shown in Tables 1 and 2). Other theoretical density aspects of noninteractive powder mixtures are reviewed and discussed by Stovall *et al.* (1986). Figure 1d depicts an array where the distribution between smaller and larger particles is reversed. Both arrays, i.e. those shown in Fig. 1c and d, it should be added, are "random" or "noninteractive" and will have a tendency to segregate with the larger particles migrating to the top of the bed and the smaller particles to the bottom. Fig. 1e and f show different and new arrays, both of "ordered" or "interactive" mixtures. The one shown in Fig. 1e can have a density lower than that of the component with the larger particle size while that shown in Fig. 1f will always have higher density than that of the larger component.

TABLE 2.
APPROXIMATE SIZE, DENSITY AND POROSITY OF THE POWDERS USED IN THIS STUDY

Powder	Approx. Mean Size (μ)	Bulk Density ($g.cm^{-3}$)	Approx. Porosity (-)
Granular Malic Acid	440	0.90	0.4
Granular Sucrose	440	0.94	0.4
Soy Protein	250	0.28	0.8
Cornstarch	90	0.65	0.6
Powdered Sucrose	80	0.65	0.6

Compressibility-Structure Relationships

The relationship between powder cohesiveness and compressibility is demonstrated schematically in Fig. 2. The fit of Eq. 1 of the experimental data had regression coefficients in the range $0.982 < r^2 < 0.998$ with corresponding significance levels of $P \leq 0.01$ or better.

Compressibility, in many powders, is a measure of internal cohesion, and to some extent of the particles deformability as well. (Moreyra and Peleg 1981; Peleg 1983). In mixtures, low compressibility can be simply a result of the fact that all or most of the interparticle space is filled with fines (Fig. 1c). Similarly, high compressibility can reflect a "lubricating effect" caused by the fines presence at the large particles surface, particularly when the former have a

TABLE 3.
MAXIMUM THEORETICAL DENSITY OF MIXTURES PROVIDED THE FINER COMPONENT PARTICLES ONLY OCCUPY THE VOIDS BETWEEN THE LARGER PARTICLES

	G. malic acid	G. sucrose	soy prot.	cornstarch
G. malic acid	-	[no change]	N.T.	N.T.
G. sucrose	[no change]	-	-	-
soy protein	1.0 (11%)	1.0 (11%)	-	-
cornstarch	1.2 (22%)	1.2 (22%)	0.8 (65%)	-
P. sucrose	1.2 (22%)	1.2 (22%)	0.8 (65%)	[no change]

1) Based on Eq. 2 and Table 2.
2) Numbers in parenthesis are the finer powder concentration in the hypothetical case where it fills all the voids.
G - Granular, P - Powdered, N.T. - Not tested in this work.

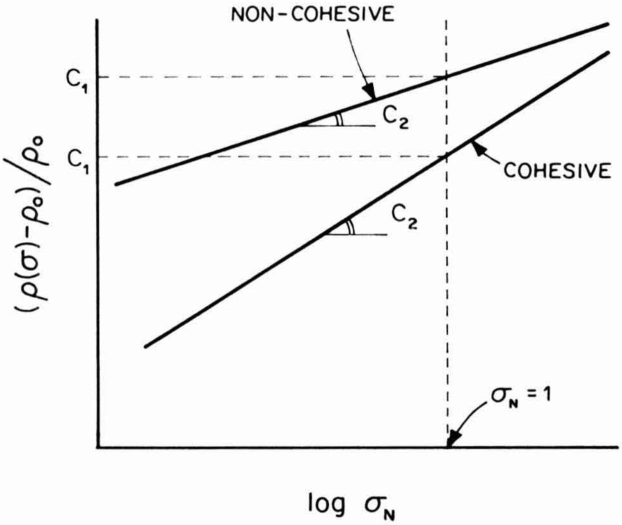

FIG. 2. SCHEMATIC PRESENTATION OF THE EFFECT OF POWDER COHESION ON ITS DENSITY AND COMPRESSIBILITY

$P(\sigma)$ is the compact density under normal stress σ_N, P_O is the initial loose bulk dencity C_2 - the slope of the line refers to compressibility (see Eq. 1).

regular shape and smooth surface texture, Fig. 1e. It can, of course, also be due to increased cohesion resulting from the fines tendency to absorb moisture for example.

The various possible effects of composition on the density and compressibility of powder mixtures are summarized in a schematic way in Fig. 3. It shows that the observed density and compressibility can be either above or below the hypothetical straight line that connects the densities, or compressibilities, of the pure components. A third theoretical possibility is that the observed density concentration or compressibility-concentration curve intersects and crosses this line in one or more points. The interpretation of such curves would be that the mixture at selected compositions behaves as a powder of a new kind. In the reported study no such pattern was observed (see below) and therefore this issue, although of interesting implications, will not be further discussed.

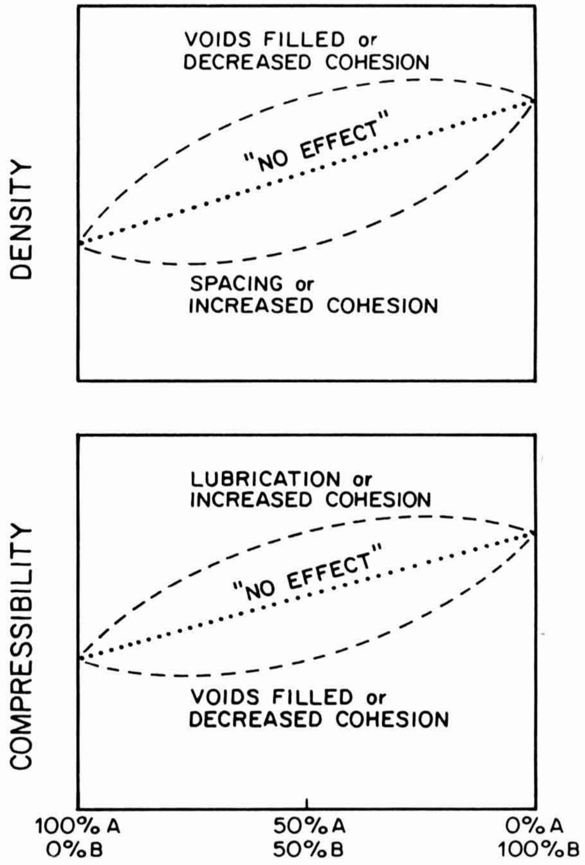

FIG. 3. SCHEMATIC VIEW OF VARIOUS TYPES OF DENSITY-COMPOSITION AND COMPRESSIBILITY-COMPOSITION RELATIONSHIP IN POWDER MIXTURES (Not to scale).

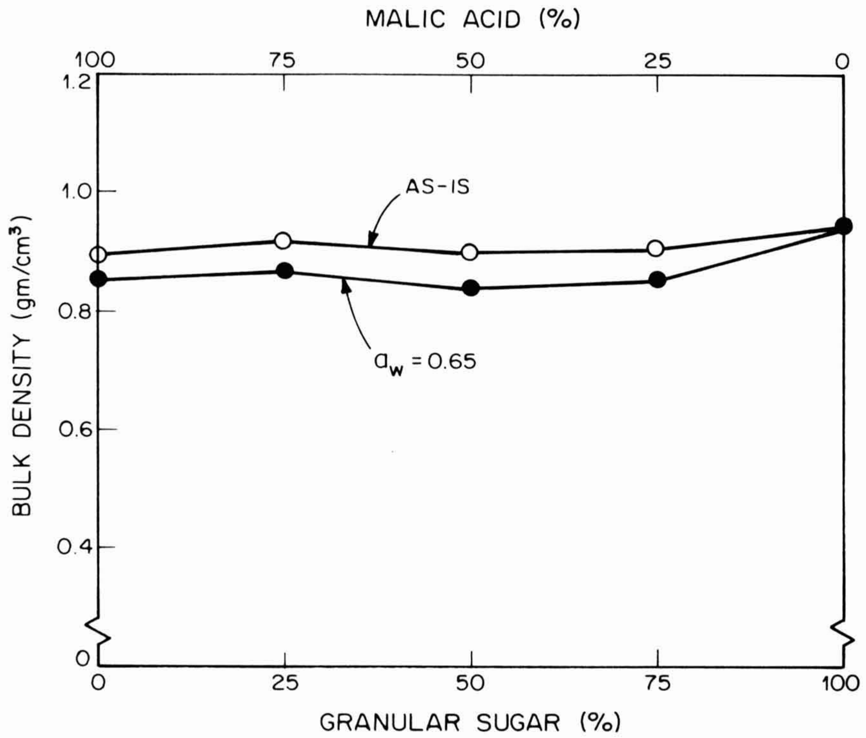

FIG. 4. BULK DENSITY OF GRANULAR SUCROSE AND GRANULAR MALIC ACID MIXTURES AT VARIOUS WEIGHT RATIOS

Density and Compressibility vs. Composition in Selected Food Powder Mixtures

The relationship between density and compressibility and the mixture ratio of sucrose (granular and powdered), malic acid (granular) soy protein and cornstarch are shown in Fig. 4–15. (The reproducibility of the density and compressibility measurements is very high, i.e., on the order of 1–2 and about 5%, respectively). The moisture as well as other physical properties of the ingredients and the mixtures are summarized in Tables 1 and 2. The data in the figures are of mixtures prepared and tested in their original moisture at ambient temperature, marked "as-is" and of the same mixtures after equilibration at $a_w = 0.65$, also at room temperature. Since the reproducibility of the density and compressibility is very high (i.e., on the order of 1 and 5%, respectively), the density-composition relationships clearly show two main characteristic patterns. The first, that of granular malic acid and sucrose (Fig. 4) and that of cornstarch and powdered sucrose (Fig. 14), is that of a line parallel to the weight fraction (composition) axis. Since the ingredients in both cases had similar particle size and density (Table 2), it appears that their particles, as far as density is concerned, are interchangeable in the powder bed spacial array. It also appears that exposure of the particles to moisture did not affect this characteristic feature as can also be seen from these figures.

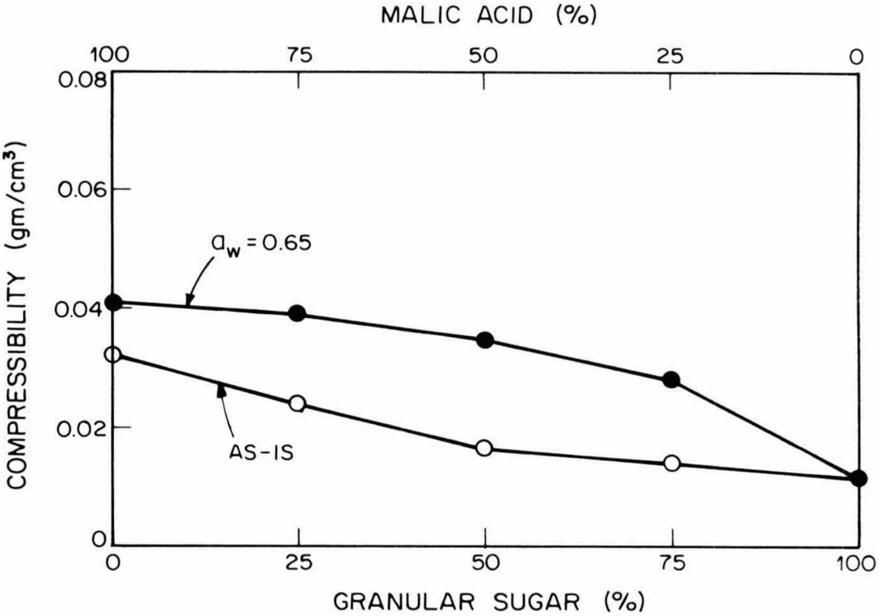

FIG. 5. COMPRESSIBILITY OF GRANULAR SUCROSE AND GRANULAR MALIC ACID MIXTURE AT VARIOUS WEIGHT RATIOS

The second type is that of concave downward relatioship indicating that the mixtures had a more open bed structure than that of the individual components. This was observed, at various degrees of curvature, in mixtures of granular sucrose with soy protein (Fig. 6) and soy protein with cornstarch or powdered sucrose mixtures (Fig. 10 and 12). In all these mixtures there was also a notable difference between the particle size of the components, Table 2. Since despite this fact no increase in density was observed, it appears that interparticle space filling by fines was not an influential factor among these powders. The curves concave downward shape can be explained by the relative cohesiveness of the fine powders which apparently induce the formation of new bed structures and in some cases, different types of "ordered" mixtures (See Barbosa-Canovas *et al.* 1985). The case of granular sucrose-cornstarch was somewhat exceptional in the sense that two points, 25% and 50% starch, were above the "no interaction" line (Fig. 8). This may be an indication that starch functions as a weak conditioner of dry sugar. Confirmation of such a hypothesis, however, requires data with much

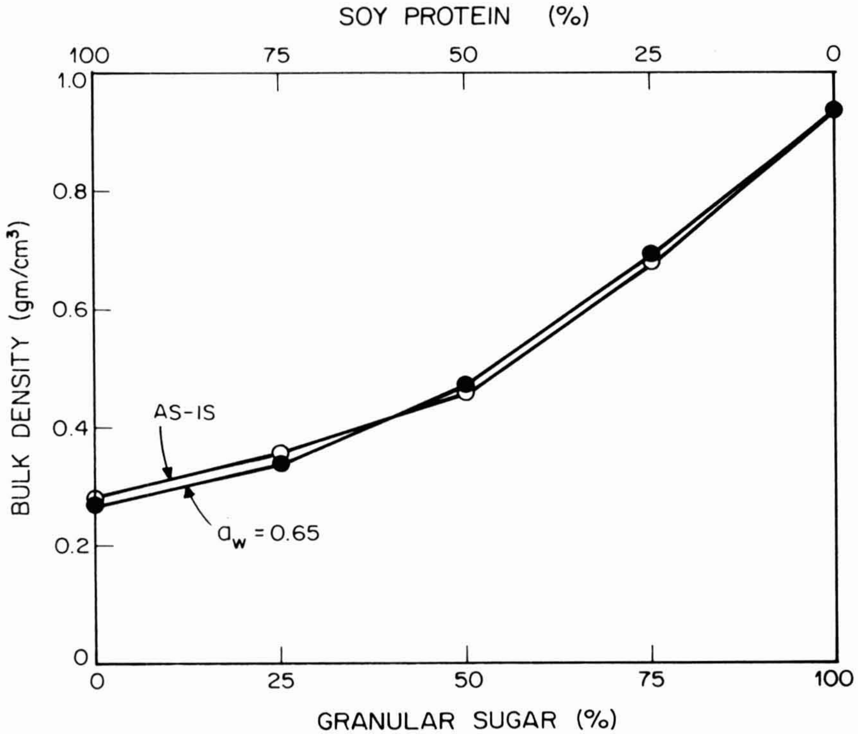

FIG. 6. BULK DENSITY OF GRANULAR SUGAR AND SOY PROTEIN MIXTURE AT VARIOUS WEIGHT RATIOS

lower starch concentrations, i.e. in the range of up to about 5% starch, in order to see whether a density increase on the order of 10–20% will result.

The compressibility data revealed all three possible patterns, namely "no effect", and both upward and downward concavities. As could be expected, the granular sucrose-malic acid mixtures showed "no-effect" (Fig. 5). This was quite expected because the two, despite their very different chemical properties, are very similar from a physical point-of-view. Both have hard crystalline structure and the samples studied in this work, also had the same mean particle size. (Table 2). The other mixtures where "no effect" was observed were the soy protein-cornstarch mixtures (Fig. 11). Since their density was not much influenced by the composition ratio (Fig. 10) this observation does not tell much about the structural features of the mixtures. It is also possible that since the two components are "floury", the structural changes that result from mixing do not show up as clearly as in mixtures of powders of distinctly different mechanical properties, e.g., crystalline and noncrystalline.

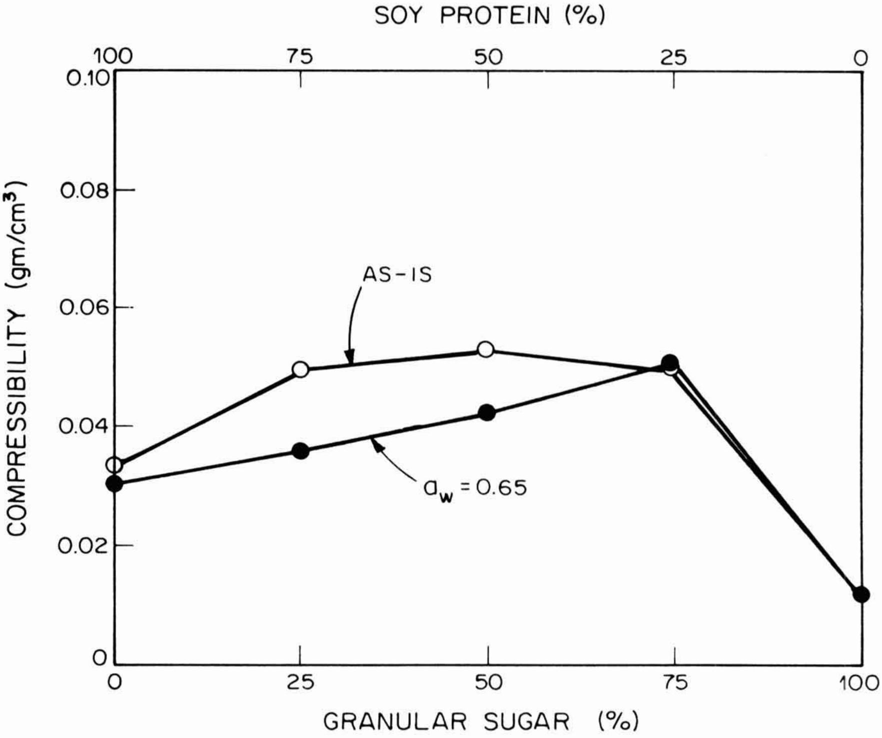

FIG. 7. COMPRESSIBILITY OF GRANULAR SUGAR AND SOY PROTEIN MIXTURE AT VARIUS WEIGHT RATIOS

Mixtures of granular sucrose and either soy protein or starch had higher compressibility than that of the individual components. This is clearly expressed in the concave upward shape of the compressibility-composition plots, Fig. 7 and 9. This observation can be explained as a result of smoothening of the sucrose surface by the presence of the small and fairly round soy and starch particles as can be seen from these figures. The effect of water activity was very noticeable in the case of starch and somewhat reversed in the case of soy protein. When the absolute moisture is taken into consideration, however, see Table 1, it becomes clear that the very high compressibility of the granular sucrose-starch mixtures was, at least partly, due to the higher cohesiveness of these mixtures and perhaps to a certain extent to plasticization of the starch particles. The same was observed in the powdered sucrose-starch mixtures (Fig. 15). While the compressibility of the dry mixtures was hardly only slightly higher than that of the ingredients, as could be expected from the unchanged initial density, it was significantly higher in the "wet" mixtures. This can be attributed to the significant increase in the mixtures cohesiveness as well as to the "lubricating" or plasticizing effect of

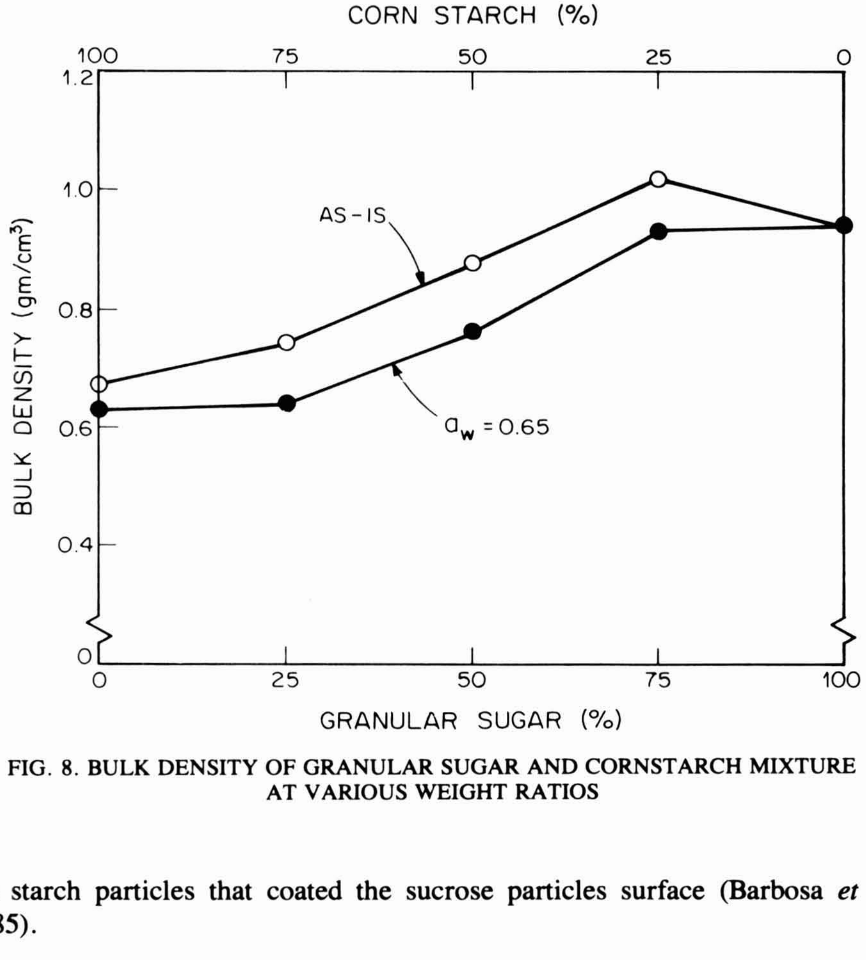

FIG. 8. BULK DENSITY OF GRANULAR SUGAR AND CORNSTARCH MIXTURE AT VARIOUS WEIGHT RATIOS

the starch particles that coated the sucrose particles surface (Barbosa *et al.* 1985).

CONCLUSIONS

It has been shown that the bulk density and compressibility of binary mixtures of various compositions can change in different ways depending on the physical and bulk properties of the components, the difference in particle size and the moisture content of the mixture. None of the mixtures studied had density that exceeded that of its components. Thus, it can be concluded, interparticle void filling was not a dominant factor that affected the mixtures density. The density of mixtures made of powders having similar solid and bulk charcteristics remained unchanged irrespective of composition. When the particles were very different the mixture density was lower than that of its components (on the order of up to about 30%). This was a result of a new bed structure formation. Compressibility showed similar trends. It either remained unchanged in mixtures of

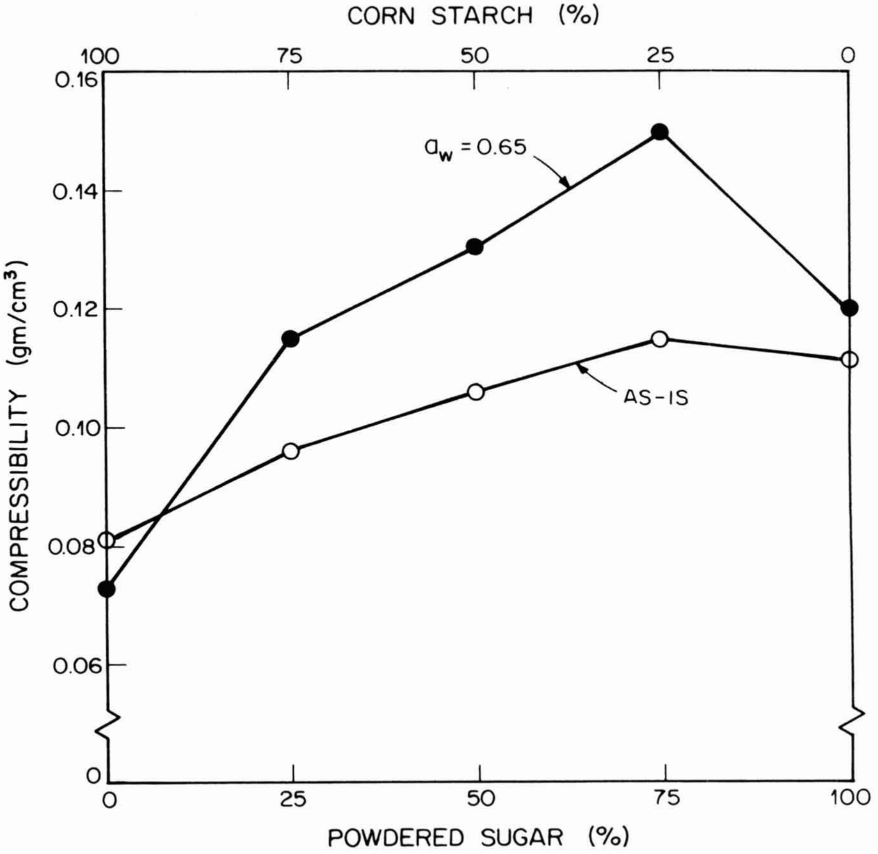

FIG. 9. COMPRESSIBILITY OF GRANULAR SUGAR AND CORNSTARCH MIXTURE AT VARIOUS WEIGHT RATIOS

similar particles, or was higher than that of the components as a result of increased interparticle cohesion and/or plasticization effects. The effect of moisture on the compressibility of certain mixtures could be drastic, reaching an order of over 50%. No such effect on bulk density has been observed. Since each mixture was unique with respect to its particles properties and structural features, no correlation could be derived between the effect of composition on neither density nor compressibility. This was particularly the case when large differences in moisture uptake by the components were also involved. It, therefore, appears that bulk properties of mixtures cannot be predicted by knowing, a priori, the properties of the ingredients.

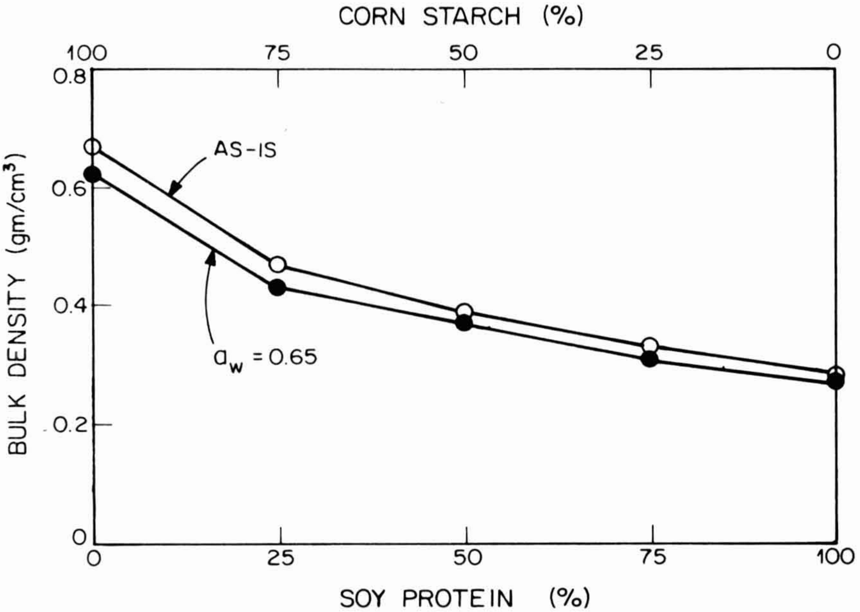

FIG. 10. BULK DENSITY OF SOY PROTEIN AND CORNSTARCH MIXTURE AT VARIOUS WEIGHT RATIOS

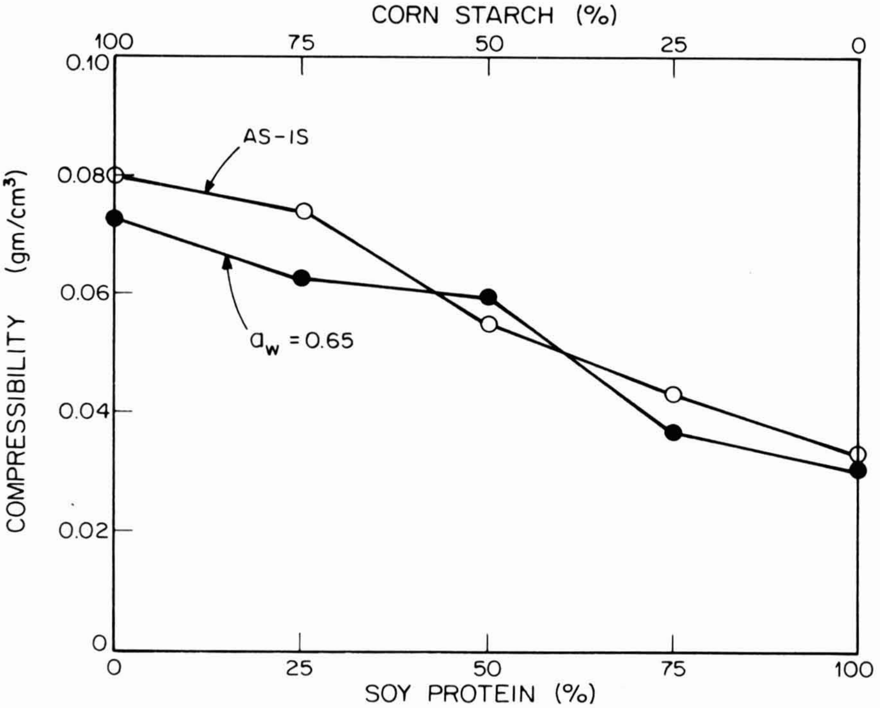

FIG. 11. COMPRESSIBILITY OF SOY PROTEIN AND CORNSTARCH MIXTURE AT VARIOUS WEIGHT RATIOS

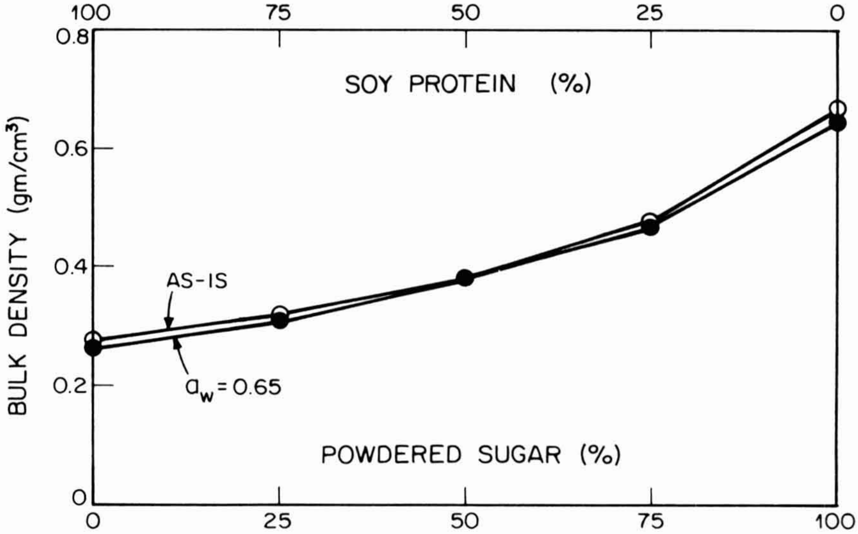

FIG. 12. BULK DENSITY OF SOY PROTEIN AND POWDERED SUCROSE MIXTURE AT VARIOUS WEIGHT RATIOS

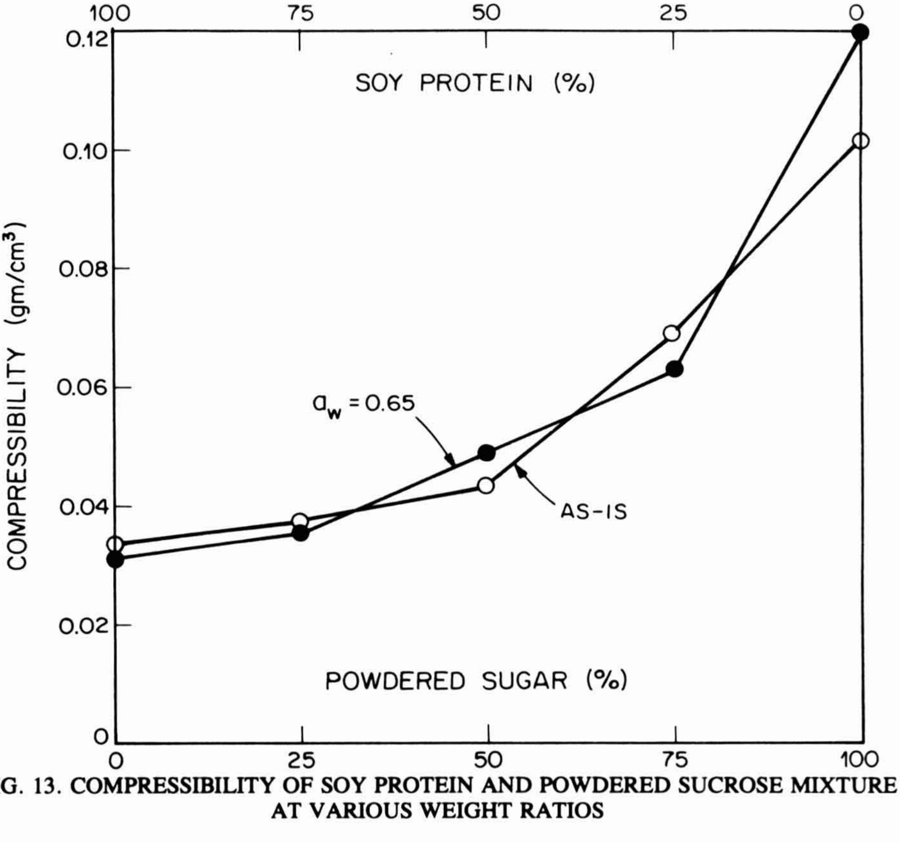

FIG. 13. COMPRESSIBILITY OF SOY PROTEIN AND POWDERED SUCROSE MIXTURE AT VARIOUS WEIGHT RATIOS

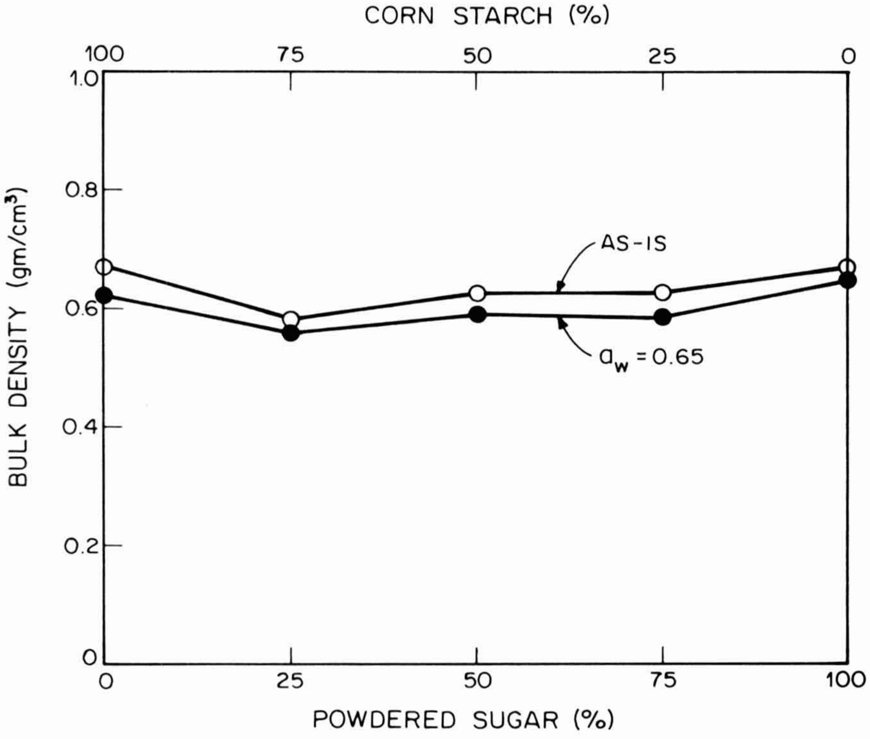

FIG. 14. BULK DENSITY OF CORNSTARCH AND POWDERED SUCROSE MIXTURE AT VARIOUS WEIGHT RATIOS

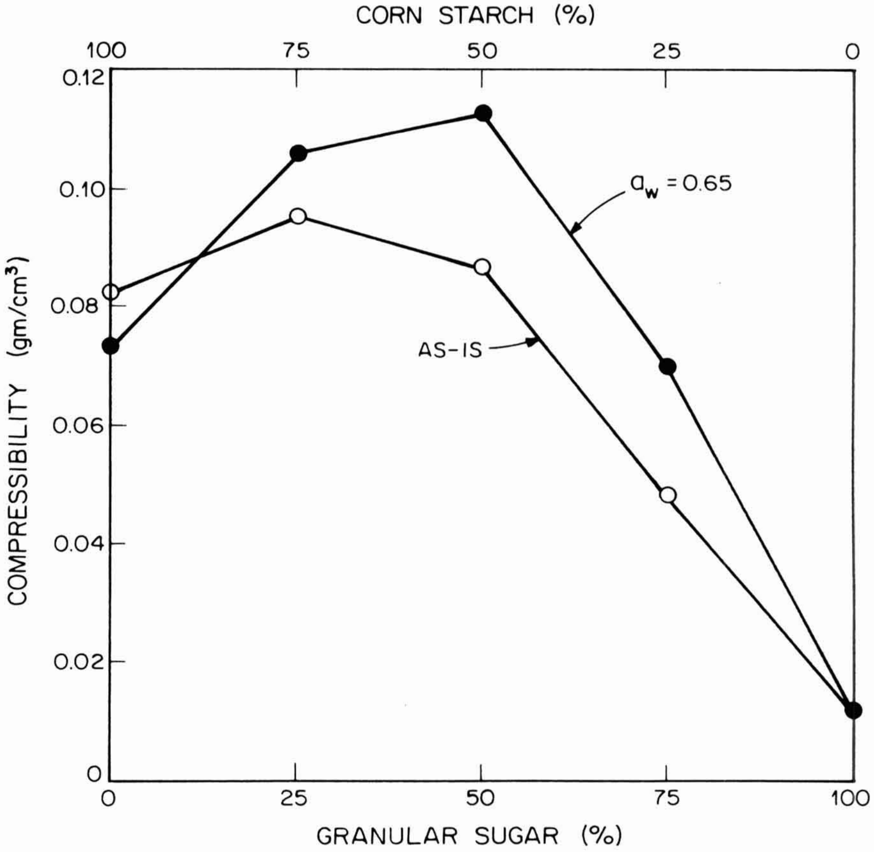

FIG. 15. COMPRESSIBILITY OF CORNSTARCH AND POWDERED SUCROSE MIXTURE AT VARIOUS WEIGHT RATIOS

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RESEARCH NOTE

ON-LINE MEASUREMENT OF DYNAMIC RHEOLOGICAL PROPERTIES DURING FOOD EXTRUSION

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ABSTRACT

Dynamic rheological properties of extruded rice dough were measured continuously using a Rheometrics On-Line Rheometer, an instrument designed for rubber and plastics applications. Storage and loss moduli were successfully measured over a moisture content range of 24 to 50% wet basis; however, the instrument is difficult to use in food research due to numerous problems: high pressure required to move material through the unit, internal dead spots causing build up of material, cleaning problems due to difficult disassembly. As a result of the development project discussed here, these problems are being addressed by Rheometrics (Piscataway, NJ) with the goal of producing an on-line rheometer acceptable to the food extrusion industry.

INTRODUCTION

Starch and protein based extrudates are highly viscoelastic materials which exhibit radical changes in rheological properties during processing. On-going chemical reactions (such as starch gelatinization and protein denaturation) and the presence of extrudate die swell (due to vapor flashing and primary normal stress differences), create serious measurement problems. Time-dependent changes in material properties make it impractical to produce material in an extruder and transport it to a rheology laboratory for testing. Thus, on-line measurement appears to provide the best means of determining material properties under extrusion processing conditions.

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Evaluating extrudate rheological properties at the extruder die is very significant. This information will be important in correlating final extrudate texture to extruder operating parameters and ingredients. Not only is this valuable as a process control tool, it will also help elucidate the basic chemical and physical mechanisms at work in the production of extruded food. Also, rheological problems in extrusion must be understood if significant progress is going to be made in the design, analysis and scale-up of food extruders.

Very few published references deal with the continuous measurement of rheological properties during food extrusion. Van Zuilichem *et al.* (1979) and Van Lengerich (1984) constructed slit viscometers to investigate the steady shear properties of extruded food materials. To our knowledge, the current effort represents the first attempt to continuously measure dynamic rheological properties during food extrusion.

This effort had two primary objectives:

(1) To evaluate the use of the Rheometrics On-Line Rheometer (ROR), designed for plastics and rubber, for food extrusion applications.

(2) To formulate design recommendations which would lead to ROR improvements making the instrument suitable for the food extrusion industry.

MATERIALS AND METHODS

The experimental samples were produced on a Baker Perkins twin screw extrusion system (Model: MPF-50D/25). The extruder has a 50mm diameter barrel and was operated with a length to diameter ratio of 15. Screw elements (primarily feed screws) were selected to generate a high pressure at the die head. Mass flow through the extruder was 44.6 kg/h (100. lbm/h) and the shaft speed was held constant at 300 rev/min. The raw material was rice flour, Comet variety.

Dynamic rheological properties were measured on the ROR manufactured by Rheometrics Inc., Piscataway, NJ. The instrument performs oscillatory testing of material between concentric cylinders separated by a 3.0 mm gap (note: some ROR units have a 1.5mm gap). The unit has numerous capabilities including frequency and strain sweep modes. Data--loss modulus (G′), storage modulus (G″), complex modulus, dynamic viscosity and tangent delta-- are output to a printer and (or) a plotter; the standard unit does not have disk storage capabilities.

A special adapter was constructed to interface the extruder and the ROR (Fig. 1). Flows from two extruder outlet ports were forced into one 3.33 cm inside diameter channel; then, material was diverted to the atmosphere (through valve 2) or into the ROR. An exit valve (valve 1) was also added to the ROR to reduce flashing problems which seriously impair the peformance of the instrument. An air actuated valve, at the inlet to the instrument, was left open and not needed in our experiments.

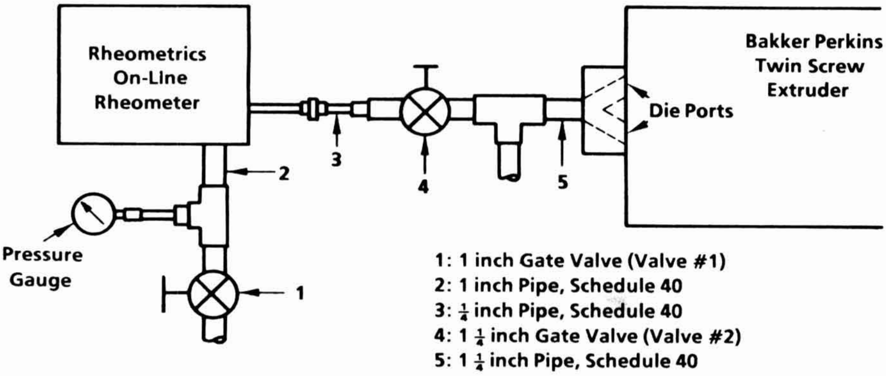

FIG. 1. PIPING SYSTEM FOR EXTRUDER-RHEOMETER INTERFACE

The ROR was completely disassembled and cleaned prior to testing. During operation, the extruder was brought to a stable condition with movement of material through valves 1 and 2 (Fig. 1). To conduct a test, valve 1 was closed and a frequency sweep initiated on the sample. When valve 1 was closed, valve 2 was adjusted (opening increased) to avoid a process upset in the operation of the extruder. The frequency sweep was conducted at ten percent strain so an adequate torque response would be obtained on the ROR. Strain sweeps, at 50 rad/s, were also conducted on various samples.

The overall testing procedure called for starting the system at high MC values and working down to low MC levels, then, back to high values. Operating in this manner should allow the dough to flush the ROR in going back to high MC levels. Internal ROR temperatures were set to correspond to the temperature measured at the extruder die head before initiating a rheological test. After the final experiment, the system was flushed with a food grade grease (Molykote, Dow Corning Corp.) using a small grease gun made for lubricating fittings. After grease insertion, and subsequent cooling, the system was completely disassembled for inspection.

Our philosophy in operating the ROR was to stress the instrument to the operating limit by measuring rheological properties at the lowest possible moisture content (MC). The intent was to simulate industrially relevant extrusion conditions where low MC levels and vapor flashing are the general rule.

RESULTS AND DISCUSSION

Due to the nature of the extrusion operation, we were unable to control temperature as an independent variable. Barrel temperatures were set at a constant value while MC was varied. Different MC levels caused variations in the viscous dissipation of energy resulting in the temperature fluctuation, 104 to 138 C, observed in the experimental data.

Typical values of the storage (Fig. 2) and loss moduli (Fig. 3) indicate expected results: increasing numerical values with increasing frequency and decreasing MC. Increasing temperature, above the starch gelatinization range, would tend to reduce G′ and G″ values. Periodic strain sweeps run on the material did not show a linear viscoelastic region for the material.

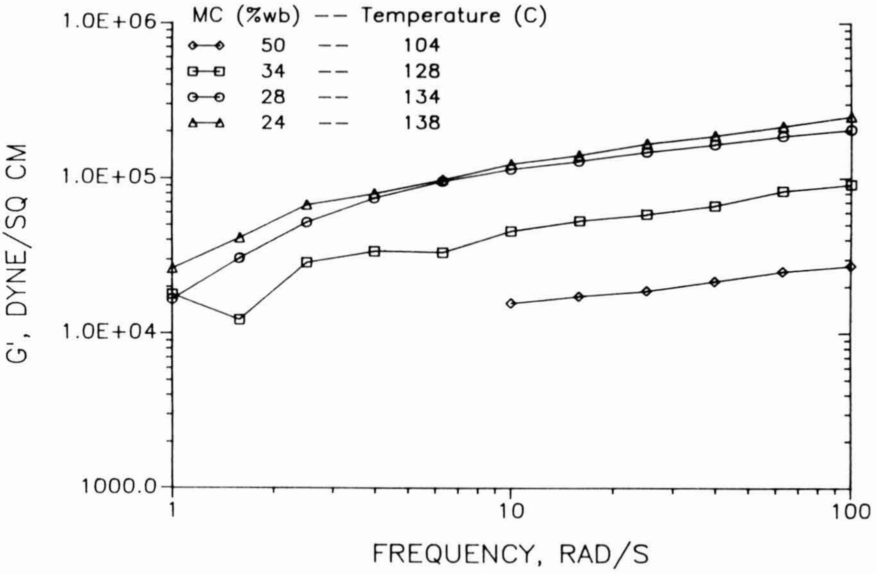

FIG. 2. RELATIONSHIP BETWEEN STORAGE MODULI AND MOISTURE CONTENT (OF EXTRUDED RICE DOUGH) AS A FUNCTION OF FREQUENCY AT 10% STRAIN

The torque measuring range of the instrument (as listed by the manufacturer) is 7.5 to 2,000 gm cm, and recorded output torque values were regularly found between 10 and 150 gm cm when operating at 10% strain. Testing at low strain levels or high MC resulted in unreliable data, torque values below 7.5 gm cm. Low torque response is the reason the 50% MC curves do not go below 10 rad/s in Fig. 1 and 2.

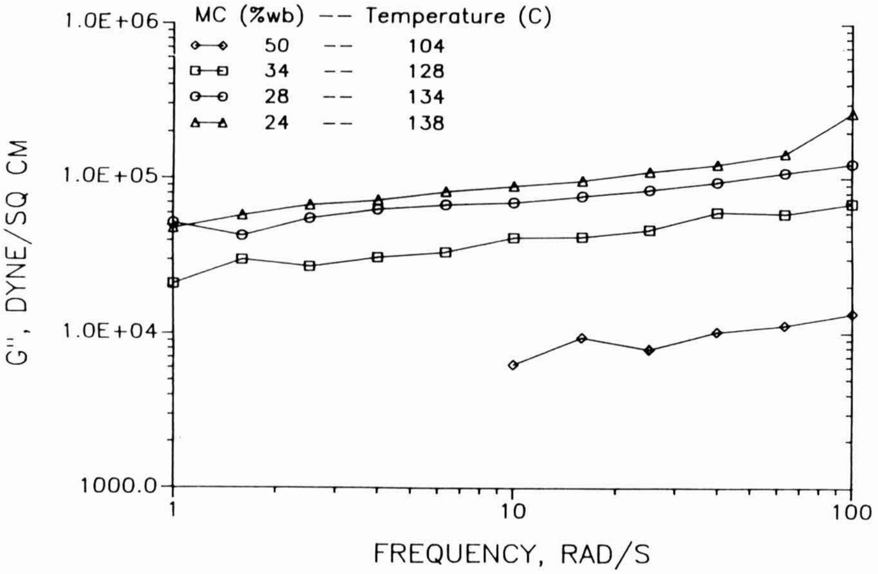

FIG. 3. RELATIONSHIP BETWEEN LOSS MODULI AND MOISTURE CONTENT (OF EXTRUDED RICE DOUGH) AS A FUNCTION OF FREQUENCY AT 10% STRAIN

The lowest MC achieved was 20%. Unfortunately, it was not possible to collect data under this condition because the system would periodically plug, due to sample cooling in the exit pipe, requiring manual removal of the obstructing material. This plugging made it impossible to maintain flow through the system and achieve steady state conditions in the extruder. An attempt was made to collect data at 22%, but erratic and very questionable results were obtained. A complete explanation for this problem has not been found but slip is suspected.

High pressures were required to move material through the ROR: the 50, 34, 28 and 24% MC runs required gauge pressures, measured at the extruder die head, of 1.31, 2.76, 4.14 and 4.96 MPa (190., 400., 600. and 720. psi) respectively. This problem may limit the usefulness of the ROR for many applications due to the difficulty of simultaneously loading the instrument and producing acceptable product. At the 20% MC level, pressures of over 6.89 MPa (1000. psi) were required to maintain product flow. The system configuration required to produce the needed high pressure (Fig. 1) also inhibited the production of commercially viable extrudate.

Disassembling the ROR after grease flushing showed that the instrument has inherent design problems which create serious difficulties in cleaning. An open area behind the test chamber, basically a dead spot referred to as the secondary flow channel, fills with material which cannot be removed between samples or by flushing the unit at the completion of testing. Dead spot material tends to burn on to the test chamber and, if not removed, provides a site for undesirable microbial activity. Dough was also observed to leak past the graphite seal located at the end of the test chamber. In addition, removal and cleaning of the test chamber is laborious: it requires approximately four person-hours and numerous tools.

SUMMARY

The ROR, as currently manufactured, is unacceptable for most food extrusion applications. Instrument design changes can significantly improve this situation. To summarize, the following should be considered in developing a food industry ROR: (1) pressure drop through the ROR should be reduced to a low level (less than .34 MPa or 50 psig) so the presence of the ROR will not interfere with normal operation of the extruder. (2) the test chamber must be redesigned to eliminate dead spots (secondary flow channel, etc.) and facilitate easy disassembly and cleaning. (3) the range of the torque transducer should be reduced, .5 to 500 gm cm would be more realistic, to allow testing at smaller strains. (4) a short pipe, complete with band heaters and a valve to reduce flashing problems, should be installed at the exit to the instrument.

Michigan State University and Rheometrics Inc. are addressing these problems to develop an on-line rheometer suitable for the food industry.

ACKNOWLEDGMENT

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A REVIEW OF PARTICLE BEHAVIOR IN TUBE FLOW: APPLICATIONS TO ASEPTIC PROCESSING[1]

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ABSTRACT

The design of holding tubes for continuous sterilization of foods containing large particulates depends heavily on knowledge of pipe flow behavior of food suspensions. Although directly related literature is limited, a considerable body of potentially useful literature is available on solid-liquid flows. This critical review discusses literature on solid-liquid flow in tubes, including capsule flow, effects on bends, radial migration phenomena, particle-particle interactions, modeling studies and other topics which may have a bearing on particle velocities in holding tubes.

INTRODUCTION

Aseptic processing is a technology of continuous food sterilization and packaging that is of considerable current interest to the U.S. and European food industries. Unlike conventional processing technology, in which food is filled into containers and sterilized by heating, aseptic processing involves separate sterilization of product and package followed by filling and sealing in a sterile area. Product sterilization is accomplished by heating, while the package may be sterilized by various methods including heat, hydrogen peroxide or electron beam irradiation. The U.S. Food and Drug Administration's 1981 approval of the use of hydrogen peroxide as a sterilant for food contact surfaces has permitted the use of metal foil-polymer laminates as packaging materials, thereby

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greatly reducing packaging costs. Additional advantages include potential improvement in product quality via ultra-high temperature, short time treatment, and continuous sterilization capability.

Aseptic processing has been successfully applied to liquid foods, notably fruit juices, and various viscous products. However, at this time the technology remains limited to these products. The next major development foreseen in this area is the continuous sterilization of low-acid foods containing large particulates. A typical system proposed in the industry for this purpose (illustrated in Fig. 1) involves a scraped-surface heat exchanger for heating the product, a holding tube to provide the desired residence time for product sterility, and scraped-surface heat exchangers for product cooling. This system is preferred to various batch processing systems due to lower cost and space requirements as well as continuous processing capability.

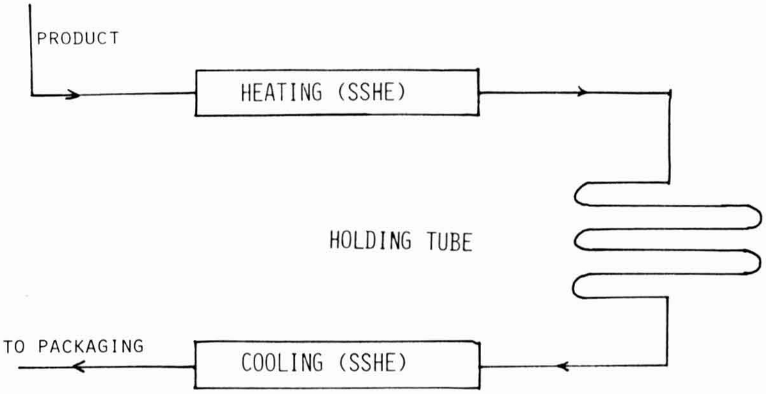

FIG. 1. SCHEMATIC DIAGRAM OF PROCESSING SYSTEM FOR PARTICULATES

A major concern in the processing of low-acid foods (pH > 4.6) is the assurance of microbiological safety of the final product. Since these products can support the growth of *Cl. Botulinum*, the thermal process provided to the food product must be sufficient to render all parts of the product (including the interior of food particles) commercially sterile. No reliable technique presently exists for continuous *in-situ* measurement of temperatures within individual particles flowing through heat exchangers and tubes. Thus mathematical modeling of heat transfer is necessary to identify cold zones within particles and to design heat exchangers and holding tubes of sufficient size to ensure commercial sterility. Early attempts at modeling were made by de Ruyter and Brunet (1973), for spherical particles, and by Manson and Cullen (1974) for cylindrical particles. Both of these studies used major simplifying assumptions in model development; notably that of an infinite convective heat transfer coefficient at the liquid-particle interface. In addition, de Ruyter and Brunet (1973) considered only plug flow velocity profiles (consequently uniform particle residence times) within the system; as assumption that was questioned by Manson and Cullen (1974). Recently, Sastry (1986) modeled the heat transfer into irregular shaped objects in aseptic systems without use of these simplifying assumptions, and determined that the following four parameters were critical to the safe sizing of these systems: (1) particle size, (2) convective heat transfer characteristics of fluid medium (both in heat exchange and holding tube sections), (3) particle residence time distribution in heat exchanger, and (4) particle residence time distribution in holding tube.

It is clear from modeling studies that conduction heat transfer in continuous sterilization systems for particles is relatively well understood, and that such a system may be designed by assigning an arbitrarily conservative value to each of the four parameters listed above. However, such a system is likely to be greatly overdesigned and result in extreme product degradation. Realistic information on these parameters is necessary for optimal design. Additionally, factors such as fluid-to-particle convection and residence time distributions pertain to the broader field of flow phenomena, and are in general, interdependent. For example, a particle moving rapidly through the system has a short residence time, but would likely experience a high relative particle-fluid velocity and consequently a relatively large heat transfer coefficient. Thus, the realistic development of particulate sterilization systems depends largely on an understanding of the flow behavior of fluid-solid particle mixtures in these systems.

The present work is a critical review of literature on solid-liquid flows, with particular emphasis on phenomena which may affect fluid and particle velocities in holding tubes. The limited body of literature directly pertaining to residence time distributions is discussed. The literature on solid-liquid flows is vast, and the discussion herein is restricted to works pertinent to holding tube flows. Topics discussed in this section include pipe flow of particulate suspension, capsule flow, radial migration of particles, particle-particle interactions and mathematical modeling. A summary of cited literature by topic is presented in Table 1.

REVIEW OF LITERATURE

Directly Pertinent Literature on Residence Time Distributions

A slug of fluid or slurry entering a chemical process vessel has been shown to disperse axially, (Danckwerts 1953) and the design of many chemical reactors is based on this important concept. An extensive review of the subject has been presented by Levenspiel and Bischoff (1963) and others. Rao and Loncin (1974) have discussed the role of this parameter in food processing. Studies of residence

TABLE 1.
SUMMARY OF CITED LITERATURE BY TOPIC

Topic	References
1. Residence Time Distributions	Chen and Zahradnik (1967), Danckwerts (1953), Heppell (1985), Levenspiel and Bischoff (1963), Rao and Loncin (1974), Roig et al (1976), Taeymans et al (1985).
2. Pipe Flow of Particulate Suspensions	Furuta et al (1978), Konno et al (1981), Newitt et al (1955), Ohashi et al (1980), Roberts and Kennedy (1971), Toda et al (1972), Zandi (1971).
3. Capsule Flow	Charles (1963), Ellis (1964a,b), Hodgson and Charles (1963), Newton et al (1964).
4. Radial Migration	Ambari et al (1984), Askoy (1973), Auton (1984), Bagnold (1974), Brenner (1966), Brenner and Happel (1958), Bungay and Brenner (1973), Chen and Carstens (1973), Cox and Brenner (1967, 1968), Davies and Samad (1978), Denson (1965), Einstein (1906), Falade and Brenner (1985), Goldsmith and Mason (1962), Hjelmfelt and Mockros (1965), Jeffrey (1922), Jeffrey and Pearson (1965), Karnis et al (1966), Lawler and Lu (1971), Lee (1979), Milne-Thompson (1968), Odar and Hamilton (1964), Oliver (1962), Repetti and Leonard (1964), Rubinow and Keller (1961), Saffman (1965, 1968), Segre and Silberberg (1961), Theodore (1964), Vasseur and Cox (1976, 1977), Willets and Murray (1981), Young (1960).
5. Particle-Particle Interactions	Brady and Bossis (1985), Cox and Brenner (1967), Saffman (1957), Segre (1965), Segre and Silberberg (1961).
6. Mathematical Modeling	Bechteler and Vogel (1983), Durand (1953), Durst et al (1984), Lawler and Lu (1971), Michaelides and Farmer (1984), Newton et al (1964), Patankar and Spalding (1972), Rose and Duckworth (1969), Shook and Daniel (1969).

time distributions in continuous food sterilization and pasteurization systems have been conducted by Chen and Zahradnik (1967), Roig *et al.* (1976), and Heppell (1985). However, these studies apply to liquid foods only, and do not represent the flow of a particulate suspension.

Residence time distribution plays a critically important role in the sizing of holding tubes for particulate slurries. Results of simulations by Sastry (1986) are presented in Fig. 2 and illustrate the strong dependence of holding tube length on residence time dispersions, both in the heat exchanger and holding tube. Experimental data on these parameters is necessary for optimum design.

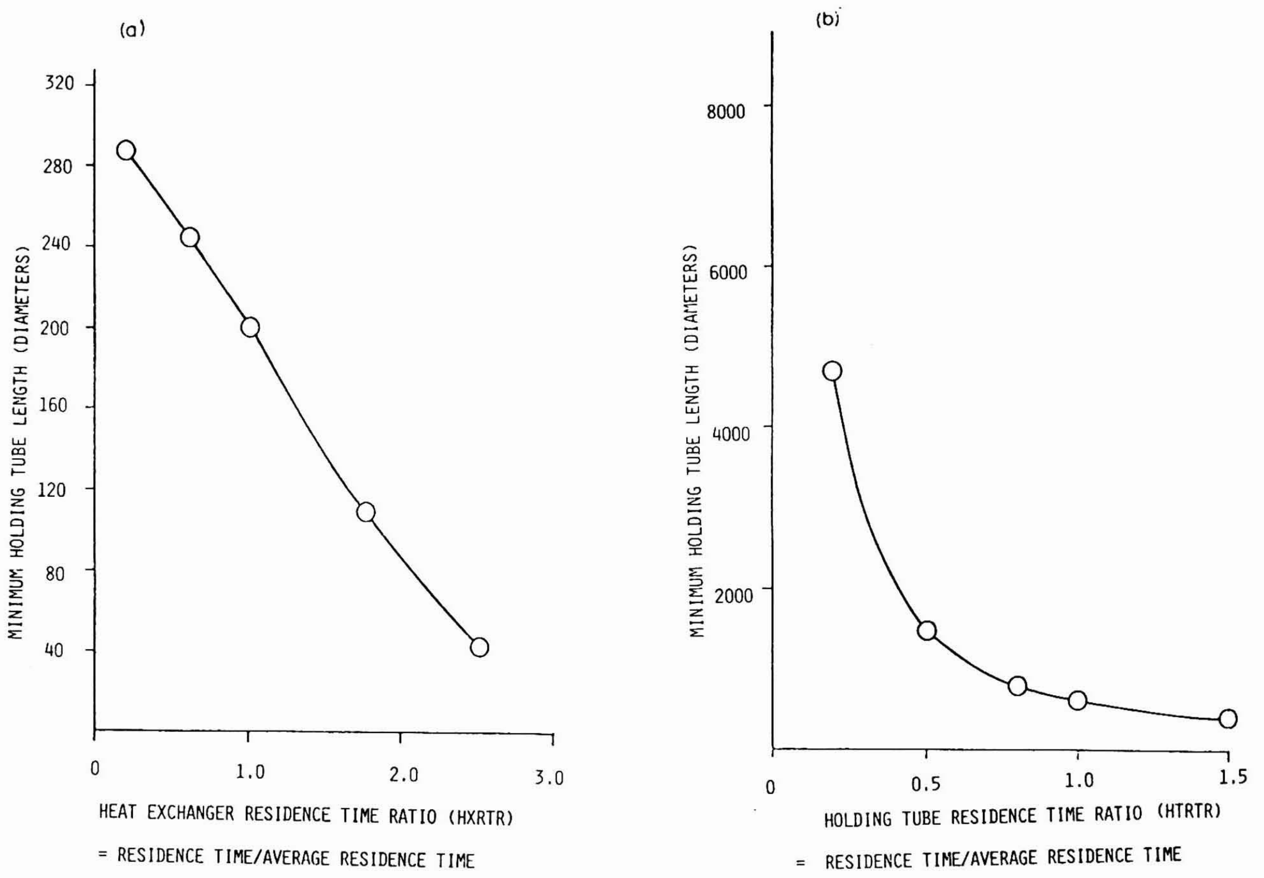

FIG. 2. INFLUENCE OF PARTICLE RESIDENCE TIME IN (a) HEAT EXCHANGER AND (b) HOLDING TUBE ON MINIMUM HOLDING TUBE LENGTH FOR COMMERCIAL STERILITY

A study of residence time distribution of a solid-liquid mixture in a scraped-surface heat exchanger was conducted by Taeymans *et al.* (1985), using a suspension of 6 mm calcium alginate beads in water. They observed that rotational speed of the dasher of the heat exchanger had significant effects on the overall dispersion of residence time. No other variables, (such as fluid rheological properties, particle size, etc.) were investigated. Manson and Cullen (1974) suggest that for a fluid in laminar flow, particle velocities can be determined by knowing the streamline along which the particle is flowing. This is perhaps true when the particles are small in comparison to the tube diameter, but the present situation may well involve particles of dimensions that are of the same order of magnitude as the internal dimensions of the flow vessel.

Although the concept of residence time distribution is an extremely useful one in visualizing the end result of complex phenomena in process vessels, and in the design of process equipment, this approach may be too phenomenological in character for the present situation. It is felt that more meaningful information can be obtained by a study of actual flow patterns as they occur within various sections of the vessel. For example, accurate knowledge of flow profiles of a particle and a fluid would not only provide residence time information, but information to evaluate heat transfer relationships as well. Residence time distribution studies would be more useful when the flow patterns are too complex to be easily visualized in the vessel.

In summary, literature directly pertaining to holding tube residence time distributions is extremely limited. A review of the literature on solid-liquid flows is appropriate.

Literature on Solid-Liquid Flows

The literature on solid-liquid flows in vast, and the discussion presented herein involves mainly selected material of potential relevance to the present situation. A considerable amount of effort has been involved in study of solid-liquid slurry transportion, rheology of suspensions, and the study of an isolated particles in unbounded flows. Before discussing the relevance of specific papers, it may be appropriate to qualitatively characterize the holding tube flow situation for foods containing particulates. The holding tube is inclined at least 0.25 in. per foot of straight run, (Food Processors' Institute, 1982) and possesses 180° bends which may be sloped upwards vertically or at the same angle as the tube itself. The carrier medium is generally a non-Newtonian fluid although Newtonian fluids may be used in certain situations. Flow may be either laminar or turbulent. The particles are close to neutrally buoyant, possess a wide variety of shapes, and their dimensions may be of the same order of magnitude as the holding tube internal diameter. Additionally, fluids lose heat to particles and the air (via tube wall), and the cooling may result in changing rheological properties over the length of the tube. Thus the flow phenomena under consideration are complex in character. It is in this light that we examine the relevance of literature.

Studies on solid-liquid flow in pipes have largely concentrated on economic long-distance transportation of mineral slurries in water, (Gandhi and Snoek 1983; Mih *et al.* 1971). Much effort has been devoted to the determination of pressure drops for pump sizing, and critical velocities for prevention of sedimentation. A considerable portion of this information is not applicable to our situation, because they deal with (1) high-density solids, (2) a Newtonian carrier fluid, (3) small particle-to-pipe diameter ratios (even if particles are large, the pipe diameter is over an order of magnitude larger), and (4) turbulent flow usually predominates in these situations. However, some of the studies on velocity profiles in this field may help provide some insights into the present situation.

Zandi (1971), in a review of the hydraulic transport of bulky materials, classifies solid-liquid flows into five major categories: homogeneous flow, heterogeneous flow, intermediate regime, saltation and capsule flow (Fig. 3). Homogeneous flow is described as occurring when the particles are fine and light, or the mean velocity of flow is sufficient to keep a uniform suspension throughout the cross-section. Heterogeneous flow occurs when particles are coarse, of large density and the flow is such that the solid and liquid phases behave as separate entities, and large density gradients exist in the flow. Intermediate flow is said to occur when conditions for heterogeneous and homogeneous flow exist simultaneously. Saltation flow occurs when the particles form a bed at the bottom of the pipe and proceed in discontinous jumps. Capsule flow is a concept developed in Canada, wherein solids are packed into cylindrical capsules of diameter slightly smaller than the internal pipe dimensions, and are transported in series.

The holding tube flow situation does not comfortably fit any of the categories described by Zandi (1971). The particles in the present case may be large enough to be classified under heterogeneous flow, but they are not of large density, being nearly neutrally buoyant, and are unlikely to exhibit density gradients. Saltation flow may occur in a few instances, but is not likely to be a major occurrence. Capsule flow may, in the extreme instance of large particle sizes, be a moderate descriptor of our flow regime. Nevertheless it is difficult to fit the flow of a food particle slurry through a tube to categories commonly associated with large scale mineral slurry transport.

The literature discussed herein will be divided into five major categories, (1) studies on pipe flows of particle suspensions, (2) studies on capsule flow, (3) studies on radial migration of particles in pipe flow, (4) studies on particle-particle interactions, and (5) modeling studies.

Studies on Pipe Flow of Particulate Suspensions. Mean particle velocities in horizontal and vertical pipes and pipe bends were studied experimentally by Toda *et al.* (1972). Their studies involved a slurry of dense (2.5 g/cc) glass beads of diameter 1.89 mm in water flowing through transparent polyacrylate pipe of 30.2 mm inside diameter. They found that in horizontal pipes, the mean

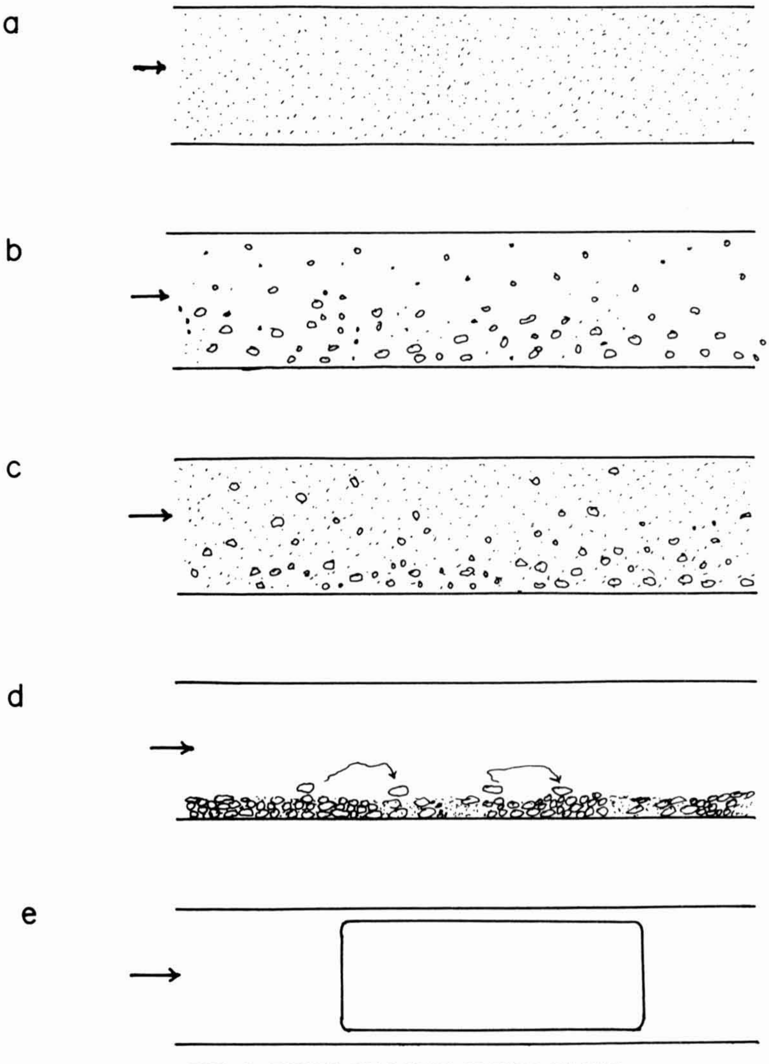

FIG. 3. TYPES OF SOLID-LIQUID FLOW
(after the characterization of Zandi, 1971). (a) homogeneous flow, (b) heterogeneous flow (c) intermediate flow (d) saltation flow and (e) capsule flow.

particle velocity was lower than the mean slurry velocity. This result may be explained by the high density of particles used: for most of the lower velocities they studied in horizontal pipe flow, the particles were largely concentrated near the bottom of the pipe. In a vertical pipe at low flow velocities, the particles were found to lag the fluid. However, as the velocity increased, particles were found to migrate towards the pipe axis and the mean particle velocity was greater than that for the fluid. In horizontal 90° bends, particle velocities were not found to be significantly affected by particle concentration. As radius of curvature was increased, the average particle velocity increased to a value greater than the fluid velocity. At high velocities however, increasing curvature radius beyond a certain point resulted in decreasing relative particle velocities, a result attributed to increasing friction between particles and pipe. Results for vertical 90° bends with horizontal and vertical approach flows were generally similar to those for horizontal bends with certain exceptions. Photography revealed that the behavior of particles in vertical bends was complicated by the effects of gravity, centrifugal force and secondary flow of liquid. At low flow rate, the particles were transported in a state of suspension in the bend, however, at high flow rates, almost all particles were transported along the outside wall of the bend because of inertial and centrifugal forces.

The studies of Toda *et al.* (1972) provide useful information on the behavior of particles in various flow situations, particularly in relation to 90° bends, (an area where information is scarce). However, their results pertain primarily to dense, relatively fine particles in water, and cannot be considered directly applicable to the present situation. Even the information on bends is of limited applicability, since the centrifugal and gravitational forces are different in the present situation, and the bends likely to be encountered in aseptic processing are usually close to 180° rather than 90°.

Furuta *et al.* (1978) studied experimentally the concentration distribution of particles in solid-liquid two-phase flow through a vertical transparent pipe of diameter 52 mm. Among the particles they studied were polystyrene particles of density 1.03 g/cc (roughly in our range of applicability), although particle diameters were smaller (1.84 mm) than for the present instance. In vertically upward flow, it was observed that the polystyrene particles increased in concentration toward the pipe axis, while in vertically downward flow, they were found relatively evenly distributed throughout the pipe cross section. These investigators atempted to explain the concentration distribution based on the radial migration force postulated by Saffman (1965), but were unable to make a complete explanation since some of their data apparently showed a radial migration in a direction opposite to Saffman's force. They explained this behavior by anisotropy of the turbulence in the fluid stream. It may be possible to at least partially explain the observations of these researchers in light of the studies of Lawler and Lu (1971); however further discussion of radial migration will be reserved for a later section of the literature review.

Ohashi *et al.* (1980) studied the average particle velocity in solid-liquid flow through vertical and horizontal tubes using photographic methods, and also used laser velocimetry for vertical flow. These researchers also studied polystyrene particles of density 1.05 g/cc; however the suspension was much finer, with particles of diameter of 0.127 mm in tubes of diameters from 1.92 to 5.42 cm. These studies yielded data in general agreement with Furuta *et al.* (1978); in vertical upward flow there was a greater concentration of particles at the pipe axis.

Ohashi *et al.* (1980) also provided dimensionless correlations between average particle velocity and modified Froude number and particle Reynolds number.

Konno *et al.* (1981) attempted to determine pipe-to-slurry heat transfer coefficients for vertical downward flow to water and particle mixtures (particles were ion exchange resin and glass beads). Particle diameters ranged from 0.15 to 1.0 mm and tube diameters from 8 to 19 mm; however, the density of particles was not specified by these researchers. As part of this study, the distribution of particles in the pipe were observed. Particle concentrations were found to be higher at the wall, unlike those found by Furuta *et al.* (1978). It is difficult to interpret the results of these researchers since experimental details were sketchy. Newitt *et al.* (1955) also point to increased migration of particles to the tube axis as the velocity increases. Studies by Roberts and Kennedy (1971) for turbulent flow of neutrally buoyant suspensions indicate that particles generally move faster than the fluid and that the difference increases with concentration. These studies provide useful guidelines to expected flow behavior in holding tubes. Nevertheless the literature in this area remains scant in relation to flow of near-neutrally buoyant, coarse particle suspensions in non-Newtonian media flowing in inclined tubes, with bends as is encountered in our situation.

Studies on Capsule Flow. Capsule flow is a concept in pipelining which involves the enclosure of solids within capsules of diameters comparable and even close to the internal diameter of the pipe. This flow regime may have some relevance to our situation since the particle size are no longer small in comparison to the pipe, although the sizes encountered here may be among the largest possible in the aseptic processing situation.

The concept of capsule pipelining has been discussed by Hodgson and Charles (1963). Capsules were always found to move faster than the fluid medium (usually water), since they usually moved along the axis of the pipe. Smaller diameter capsules flowed along the central rapid moving streamlines, and moved faster than capsules of larger diameter. Depending on the diameter, capsules moved from 5 to 18% faster than the fluid. In addition, for turbulent flow regimes up to Reynolds numbers of 15000, pressure drops in capsule flow were found to be lower than for the fluid alone. However, when the basic water stream was in laminar flow, the addition of capsules was expected to increase the pressure drop (few reliable data points were obtained for these conditions).

In the second paper of a series on capsule pipelining, Charles (1963) attempted a simplified theoretical analysis for the concentric flow of cylindrical forms. The approach involved solution of the one-dimensional Navier-Stokes equation (for an unobstructed fluid medium) in cylindrical coordinates, for a constant pressure drop, obtaining the Poiseuille flow solution. The author then considered an infinitely long cylinder to be located concentrically within the stream, and moving at the average velocity of the stream in the cross section blocked by the capsule. Although the study neglected particle-fluid interactions, it was considered by the author to be a useful basis for the preliminary understanding of the flow of capsules. The analysis indicates the limits of velocities of capsules: an infinitesimally thin capsule would be expected to flow axially at twice the average velocity of the fluid stream, while the largest possible capsule (one nearly the same diameter as the pipe) would be expected to flow at the average velocity of the stream. Models for pressure drop were also developed. One of these models appeared to indicate that for a situation involving turbulent flow in the capsule-free stream, the presence of a capsule of sufficient diameter could suppress turbulence in the annulus between capsule and fluid resulting in decreased pressure drop. This was considered a possible explanation for the observed decrease in pressure drop noticed by Hodgson and Charles (1963).

Ellis (1964a) conducted an experimental investigation on the transport in water of single, neutrally buoyant cylindrical and spherical capsules. His results on effect of diameter of cylindrical capsules generally confirmed qualitatively the theoretical analysis of Charles (1963). Smaller diameter cylinders generally flowed faster than those of larger diameter. The smallest diameter cylinders were observed to follow a wavy, unstable motion, or to migrate radially towards the walls with a nose-up or nose-down configuration, which reduced their overall flow velocity. Shorter cylinders were found to flow slower than longer cylinders due to less balance in their motion; and spheres were found to flow slower than cylinders. As the velocity of the medium increased, the ratio of capsule to fluid velocity decreased only slightly. However, at low velocities, the velocity ratio was found to be highly sensitive to slight changes in fluid medium velocity. At a Reynolds number of about 2×10^4, the velocity ratio showed a sharp rise down to a Reynolds number of about 10^4, followed by a precipitous drop and then a sharp rise. The author discussed this phenomenon in some detail, the indicating that the drop may be due to the breakaway of the laminar boundary layer near the rear of the capsule, and the formation of a turbulent wake behind it. The sharp increase in velocity ratio was considered to be due to the displacement of the point of separation of the boundary layer towards the trailing edge, thereby reducing the drag coefficient. For cylindrical capsules, the effects of a sharp nose or tail on velocity ratio were small. Spherical capsules of the smallest diameters were observed to migrate away from the axis and move at slower velocities than expected. Finally, the data obtained supported the theory of Charles (1963) that turbulence was suppressed in the annular region. This suppression was supposed to arise from a joining of the laminar sublayers adjacent to the capsule and the wall.

Ellis (1964a) also identified several dimensionless parameters influencing capsule flow. These were represented by the relation:

$$V_c/V_{av} = f(V_{av}D\rho/\mu, \sigma\text{-}\rho/\sigma, d/D, L/d, c, e_c/d, e_c/D, \text{end shape}) \qquad (1)$$

and may be helpful in design of experiments.

In another paper, Ellis (1964b) also investigated the transport of spherical capsules of density greater than water. These results are not as applicable in the present case as those of Ellis (1964a), but a few points of interest are worth noting. The zone of instability of velocity ratio of neutrally buoyant capsules was also the zone of great increase in velocity ratio of dense capsules. Notably, these capsules did not attain an axial position, and consequently flowed slower than neutrally buoyant capsules of the same dimensions.

In the last of a series of papers on capsule flow, Newton *et al.* (1964) conducted a numerical analysis of capsule flow. This analysis, although containing many simplifying assumptions, was considerably more sophisticated than that of Charles (1963). The analysis considered a long cylinder in a pipe, and involved a finite difference solution to the Navier-Stokes equation assuming steady, fully developed flow and no vertical forces on the capsule. The simulation indicated that axial displacement of the capsule had an important effect on capsule velocity. Friction was found to decrease capsule velocity uniformly. As friction force was increased, a critical value was reached for larger capsules which resulted in a rapid decrease of the velocity ratio to zero. End-shape effects of capsules was found to be negligible, although the fluid flow field was strongly influenced. Capsule deformation had little effect for 10 to 20% deformations. Beyond this point, the pressure gradient to maintain a given capsule velocity increased almost linearly with deformation. The paper of Newton *et al.* (1964) will be discussed further under the section on mathematical modeling.

In summary, several of the findings for capsule flow may be useful in the aseptic holding tube scenario, although direct application could only be made in few cases of extremely large particles in a Newtonian fluid.

Studies on Radial Migration of Particles in Pipe Flow. One of the most important phenomena affecting particle velocities in pipe flow is that of radial migration away from the pipe axis and wall. A particle moving away from the axis of a pipe moves at a slower velocity than an axially located particle. This phenomenon has received considerable attention in the literature.

Early studies (Einstein 1906; Jeffrey 1922) appeared to indicate that in the pipe flow of a suspension, particles would tend to accumulate near the axis. However, Young (1960) observed accumulation of particles near the pipe wall in certain circumstances. Segre and Silberberg (1961) showed via an experimental study that neutrally buoyant particles would migrate away from both the axis and the wall, reaching an equilibrium position at 0.6 times the pipe radius from the axis. Goldsmith and Mason (1962) found that in Poiseuille flow at low Reynolds numbers, the radial position of a single rigid particle remained constant over prolonged periods of flow, while deformable particles (fluid drops) migrated to the tube axis. The various types of radial migration phenomena are illustrated in Fig. 4.

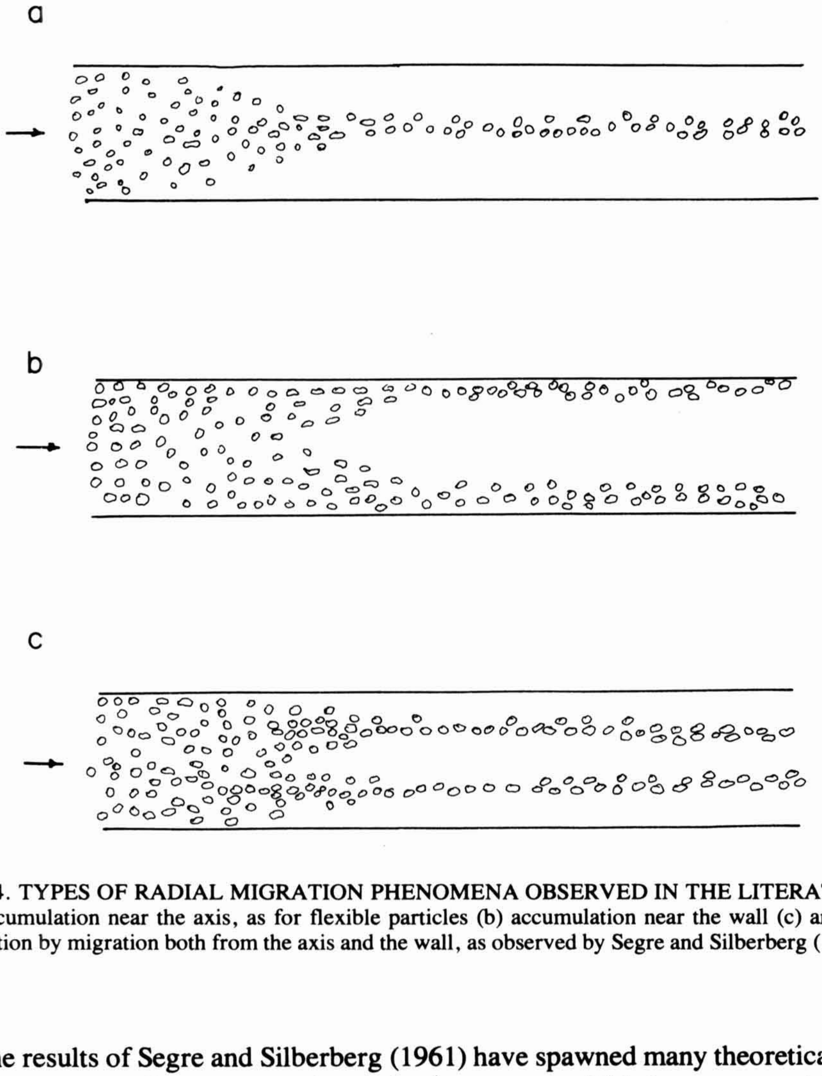

FIG. 4. TYPES OF RADIAL MIGRATION PHENOMENA OBSERVED IN THE LITERATURE (a) accumulation near the axis, as for flexible particles (b) accumulation near the wall (c) annulus formation by migration both from the axis and the wall, as observed by Segre and Silberberg (1962).

The results of Segre and Silberberg (1961) have spawned many theoretical and experimental studies on estimation and measurement of lift forces on particles in shear flows, and in attempting to explain the apparent discrepancy between these results and earlier works. An considerable quantity of literature exists on the subject, including Brenner (1966) and Soo (1967). The apparent discrepancy between the Segre-Silberberg results and earlier works appears to have been the result of slip between fluid and particle. It has been observed that migration occurs towards the wall when particles move faster than the fluid, while the reverse occurs when the particles move slower than the fluid (Oliver 1962; Jeffrey and Pearson 1965; Repetti and Leonard 1964). Oliver (1962) and other researchers have observed that neutrally buoyant particles near the wall migrate inwards, while those near the axis migrate outwards to their equilibrium positions (generally 50 to 70% of pipe radius). Denson (1965) found that for particles lagging the fluid, the radial trajectory of the particle depended on the Reynolds number. At Reynolds numbers above 550, particles showed oscillatory behavior, while at lower Reynolds numbers, these oscillations were damped. Other studies on radial migration have been conducted by Karnis *et al.* (1966) under conditions of Couette and Poiseuille flow, confirming the findings of Goldsmith and Mason (1962) that rigid particles migrated to intermediate positions while deformable particles (e.g., red blood cells) always migrated to the tube axis.

Explanations for the radial migration phenomenon have included particle-particle interactions, radial fluid motion due to entrance effects, but neither of these explanations has proved feasible since single particles also experience radial migration, and this effect occurs beyond the entrance length of a pipe (Lawler and Lu 1971). Lift forces appear to be the major reason, and a number of studies have involved prediction and measurement of these forces. Rubinow and Keller (1961) developed an expression for the lift force experienced by a spinning particle in a quiescent fluid, which was given by:

$$F_1 = \pi \rho_f a^3 \Omega \times V \, (1 + o[Re]) \qquad (2)$$

This is the well-known Magnus force, which is used by tennis players and baseball pitchers to move balls along rising, falling or swerving trajectories. Similarly, a body located on the tube wall would be rotated by moments caused by the fluid shear force, giving rise to migration towards the tube axis (Fig. 5).

The Rubinow-Keller expression is limited in that it depends on the existence of rotation for lift to occur. It has been shown experimentally by Oliver (1962) that particles migrate radially even when constrained from rotation. Theodore (1964) was unable to detect measurable differences in lift forces between rotating and nonrotating particles. Thus the Rubinow-Keller expression has been generally rejected as being the sole explanation of lift in pipe flow. Brenner and Happel (1958) have shown, in their studies of slow flow past a sphere in a tube that the creeping flow equations fail to predict a lift force due to the neglect to inertial terms; thus inertial effects were considered important in radial migration.

Saffman (1965) developed an expression for the lift force acting on a sphere in an unbounded simple shear flow. The relation, subsequently corrected by the author, (Saffman 1968) can be written as:

$$F_1 = 6.46 \mu \, V a^2 (\kappa/\nu)^{1/2} + \text{smaller terms} \qquad (3)$$

Saffman's force does not require rotation for migration to occur, in accord with experimental observations. Saffman also showed that at small values of Reynolds number, unless the rotational speed is much greater than the rate of

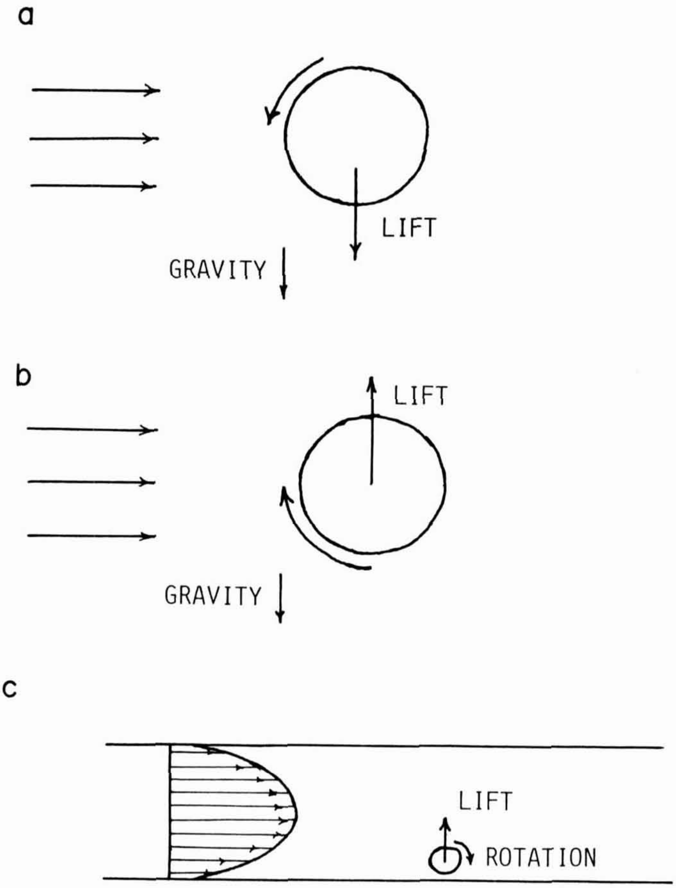

FIG. 5. ILLUSTRATION OF MAGNUS SPIN LIFT FORCE AND ITS INFLUENCE ON THE MOTION OF A SPHERE
(a) downward orientation of "lift" force on a topspun ball (b) upward orientation of lift force for an underspun ball results in a 'sailing' trejectory (c) Magnus effect on a sphere in tube flow -- shear-induced rotation results in lift force towards axis as shown.

shear, the lift force due to particle rotation is less by an order of magnitude than that due to shear. Saffman's expression, however, was derived with some restrictive conditions relative to the particle, shear and rotational Reynolds numbers.

None of the above theories completely explains the two way radial migration phenomenon for neutrally buoyant particles. Brenner (1966) and Karnis *et al.* (1966) indicated that the presence of the wall was a factor of fundamental importance. Odar and Hamilton (1964) and Hjelmfelt and Mockros (1965) studied the motion of accelerating spheres in viscous fluids. Milne-Thompson (1968) obtained a potential flow solution for the lift on a sphere near a plane wall, for large separations between particle and wall relative to particle size. Lawler and Lu (1971) modeled the radial migration of particles in a pipe flow, using a combination of the lift forces of Rubinow and Keller (1961) and Saffman (1965), and assuming that the particle had a negligible influence on the flow profile (Poiseuille flow) of the fluid. Their model was compared to experimental data, and it was indicated that the model was capable of at least qualitatively explaining the various modes of radial migration that had been observed. This model will be discussed further in the section on mathematical modeling.

Wall effects have been the subject of considerable interest, although not all of these studies have been directly concerned with radial migration, the estimation of the magnitudes of forces acting on particles may be useful in the context of studying radial migration. The influence of boundaries has been studied extensively by Cox and Brenner (1967) who solved for the force and couple acting on a body translating and rotating near a wall. Cox and Brenner (1968) analyzed the situation when the sphere was not too close to the wall by solving the Navier-Stokes equation to obtain the lateral force on a sphere, which was then converted to an equivalent radial migration velocity by use of Stokes law. This analysis assumes a creeping flow regime, and particle sizes that are negligibly small in comparison to wall-to-wall distances. Bungay and Brenner (1973) derived an expression for the pressure drop of a relatively small sphere moving near the wall of a tube within which Poiseuille flow occurred. Vasseur and Cox (1976) extended the theory of Cox and Brenner (1968) to fluid flowing between plane parallel vertical walls. They analyzed particle migration for cases involving quiescent fluids, and Couette and Poiseuille flows. They found that for neutrally buoyant particles, the equilibrium position between axis and wall depended on whether or not the particles rotated. Near-neutrally buoyant spheres were found to achieve various equilibrium positions depending on density differences and rotation or nonrotation of particles. These analyses assume that the creeping flow equations apply. Vasseur and Cox (1977) treated the case of spherical particles sedimenting in a stagnant bounded fluid, and found, both theoretically and experimentally, that particles migrated to the axis of the duct. Recently, Ambari *et al.* (1984) presented experimental results on the modified Stokes force acting on a sphere near a wall. Falade and Brenner (1985) derived simple asymptotic formulae for calculating force and torque on a particle moving near a wall, provided the particle center was relatively distant from the wall compared with the particle size.

Much recent attention has been focused on lift measurement in steady flows (Chen and Carstens 1973; Askoy 1973; Bagnold 1974; Davies and Samad 1978; Lee 1979; and Willets and Murray 1981). Bagnold (1974) stated that immersion of a particle in a flow field with a velocity gradient is bound to induce it to spin and consequently experience a lift force. Willets and Murray (1981) found that spheres near a wall experienced lift forces, and for gaps less than a quarter to the sphere diameter, wall effects were found to dominate all dependencies except that on the Reynolds number. Recent studies on developing expressions for lift force have been conducted by Auton (1984), who has performed an inviscid analysis on a sphere in shear flow.

In summary, much has been written on radial migration and lift forces that would be of use in a study of flow of particulate suspensions through holding tube. Many of the theoretical expressions have been developed for low Reynolds numbers verging on creeping flow situations. It is uncertain to what extent these can be extrapolated to a realistic flow regime in the present situation. However, studies on measured lift forces in more rapid flows may provide useful insights to this situation.

Particle-Particle Interactions. It would be expected in general that particulate suspensions flowing through holding tubes would experience particle-particle interactions in addition to hydrodynamic interactions as discussed in the preceding section. Segre and Silberberg (1961) found that in steady flow, particles that became concentrated in a thin cylindrical layer and all moving at the same velocity aligned themselves into regular columns or "necklaces" parallel to the tube axis. Segre (1965) described these interactions as comprising attraction and repulsion with the resulting oscillation but no collisions, until an equilibrium distance was reached. More and more particles would join the group to form "necklaces" (Fig. 6).

Segre (1965) conducted studies to characterize the tendency of particles to group formation. He found that while very large groups or "necklaces" were rare, the tendency to form groups increased with increasing particle dimensions and concentration. Increasing Reynolds number was found increase the grouping tendency upto a maximum, at which point a decrease was observed, the latter phenomenon being attributed to the onset of turbulence. The interparticle gap in each group was found to decrease with increasing Reynolds number and with increasing group population (although some levelling off was observed with large groups). The gap did not appear to be directly related to particle size. A statistical model for group frequency distribution was developed. These studies indicate that in many cases, groups of particles may be expected to possess uniform residence times, although the effect of bends and pipe inclination may alter this tendency.

On a theoretical level, most treatments of particle-particle interactions relate to the two-particle case. Saffman (1957) discussed the interactons of two small spheroidal particles in a flow field. Cox and Brenner (1967) analyzed the motion of two small bodies of arbitrary shape in and arbitrary Stokes flow field, assuming that the two bodies were indentical in shape, that the distance between them was large compared to their size, and that they translated with identical

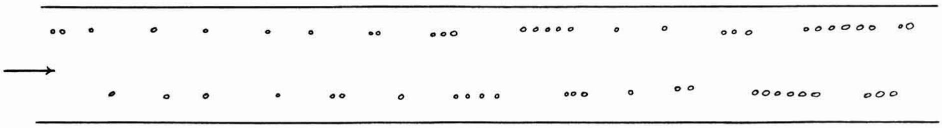

FIG. 6. ILLUSTRATION OF NECKLACE FORMATION IN SUSPENSION FLOW OF PARTICLES
(See Segre, 1965).

velocities in the direction of the line joining their centers. Studies on the rheology of suspensions attempt treatments of two-particle interactions: however, much of this work aims at the prediction of viscosities from a knowledge of microstructure. While many works exist in this area, they are not directly relevant to our study and will not be covered here. Brady and Bossis (1985) state in connection with the rheology of concentrated suspensions, that the computation of many-body hydrodynamic interactions are so severe that analyses involving more than two particles have involved severely restrictive assumptions. They proposed a method (termed Stokesian dynamics) for simulation of these interactions for the situation involving N particles suspended in a Newtonian solvent undergoing bulk linear shear flow at low particle Reynolds numbers. However, their analyses were intended to model the behavior of a homogeneously sheared suspension in regions far removed from any boundaries. The difficulties in modeling many-body interactions within bounded pipe flows appear to be considerable at this time.

Studies on Mathematical Modeling. Adequate mathematical models for solid-liquid flows are necessary for design of holding tubes. This section focuses on some of the numerical solutions to solid-liquid flows in the literature. Although many of the works discussed under radial migration and particle-particle interactions could be classified under the broad category of mathematical modeling, the focus here will be on research pertaining to numerical simulation of fluid flow and particle trajectories and/or velocities, in pipe flow situations rather than specifically on radial migration forces for the creeping flow regime.

The model of Newton *et al.* (1964), discussed earlier considers capsule flow of an infinite cylinder in a Newtonian fluid assuming steady, fully developed flow, the solution being obtained by finite differences. While the study is worthwhile from the standpoint of simulating the motion of a large body through a pipe, the phenomenon of radial migration is not considered, and the assumption of an infinitely long body is a limitation in the model. The solution may be useful in the large particle limit.

The model of Lawler and Lu (1971) is a useful one in that it attempts to integrate the knowledge of the time into a model to describe single particle trajectories in a realistic pipe flow situation. The authors assume that the fluid velocity profile is relatively undisturbed by the presence of particles. Then, using a velocity profile for a Newtonian fluid in fully developed flow, individual particle trajectories are determined based on drag and lift forces on individual particles. The lift forces include both the shear lift of Saffman (1965) and the Magnus lift of Rubinow and Keller (1961). Thus, the problem is simplified from one involving simultaneous solution of fluid and particle dynamics, to one involving particle dynamics alone. The authors report that their simulations yield results that are in qualitative agreement with experimental findings. The authors recognize that the ambitious approach to the problem would be the iterative solution of the Navier Stokes and the particle dynamics equations, but do not use the approach on the grounds that it is not the most expeditious. Further, the model assumes that the expression for radial migration forces of Rubinow and Keller (1961) and Saffman (1965) apply in the situation considered. The model applies to flow in a vertical pipe, and is therefore not directly applicable to holding tubes.

The models discussed above, as well as several other models discussed under radial migration consider the behavior of a single practice. A few studies, (discussion of some of which follows) have involved simulation of slurry velocities instead of the single particle case.

Various empirical correlations for slurry velocities have been presented by Durand (1953). Rose and Duckworth (1969) have developed semiempirical relationships between dimensionless groups using experimental data. Shook and Daniel (1969) develod a theoretical approach by treating the solid-liquid mixture as a single phase fluid with variable density; however, they made major simplifying assumptions in their boundary conditions. Michaelides and Farmer (1984) used a similar approach extended to turbulent flow by modeling the solid-liquid mixture as a single-phase fluid of locally variable time-average density. The solution procedure involved considerable simplification of the Navier-Stokes equations and solution via Runge-Kutta procedures. The results were in good agreement with experimental data for sand-water slurries when the flow could be considered homogeneous; however, when heterogeneous flow conditions prevailed, poor agreement was obtained. The assumption of pseudohomogeneous flow would tend to render this model inapplicable in the present instance. Wood and Kao (1971) studied the transient flow of solid-liquid mixtures in pipes, but their major emphasis was on pressure wave velocities in these pipes.

Bechteler and Vogel (1983) developed a set of five differential equations for solution of one-dimensional transient flows of solid-liquid-gas mixtures in the five unknowns: pressure, solids flow velocity, liquid flow velocity, solids concentration and air concentration. They described two possible methods (finite differences and the implicit method of characteristics) for solving these equations, but presented no solutions. They assumed (1) inviscid flow, (2) gas phase entrapped in the liquid and solid phases, (3) added mass phenomenon occurs within each phase as a result of gas entrappment, thus the flow is approximated as a pseudo-two-phase flow. While the approach may be useful form the standpoint of separate consideration of solid and liquid velocities, the assumption of inviscid flow and the lack of any actual simulation results must be viewed as serious deficiencies.

Durst *et al.* (1984) attempted to numerically simulate particulate two-phase flows using two approaches (1) the Eulerian approach wherein the solids were treated as a continuum, and (2) the Lagrangian approach (more relevant in our case) which predicted particle trajectories in the fluid phase as a result of forces acting on the particles. The particles in this study were considered large enough for significant slip to exist between phases. The Eulerian approach involved use of continuity and momentum equations for both the solid and liquid phases, together with an equation relating the void fraction of phases. The Lagrangian approach involved use of the fluid continuity and momentum equations and particle dynamics equations, and a void fraction relationship. The back-influence of particles on fluid was modeled using source terms in the fluid momentum equations. Solutions were obtained in both cases by means of a numerical code described in detail by Patankar and Spalding (1972). The Eulerian approach is useful in that it describes particle flows in high concentrations, but is limited in that it cannot predict individual particle trajectories. The Lagrangian approach, on the other hand, can provide information on individual particle trajectories, but neglects particle-particle interaction. This model appears to have fewer simplifying assumptions than most, but it does assume the following: (1) small particles (an implicit assumption) from our standpoint even though significant interphase slip may exist, (2) Newtonian fluid, and (3) no particle-particle interactions in the Lagrangian approach. Despite these deficiencies, the model may be fairly useful in the present situation.

In summary, although some potentially useful approaches are identifiable, none of the currently available models are directly applicable to continuous sterilization of particulate food suspensions.

CONCLUSIONS AND RESEARCH NEEDS

The challenges of understanding and accurately predicting multiphase flow phenomena in aseptic processing are considerable. Although much information exists on solid-liquid flows, much of it relates to simplified flow situations involving Newtonian fluids and horizontal or vertical pipes. The influence of inclination and 180° bends in piping has received scant consideration. The radial migration phenomenon has received much attention, and seems to play an important role in determining particle velocities in pipe flow; yet much of the literature has largely focused on individual particles rather than collections of particles, with notable exceptions being Segre and Silberberg (1961) and Segre (1965). The influence of fluid cooling and changing rheological properties has not been considered, while the modeling problem for two-phase flow remains a considerable challenge.

From the standpoint of the industrial designer of aseptic processing systems immediate answers may have to be sought from realistic, residence-time-distribution studies. Such studies have been and are currently being conducted in industry. These studies would provide helpful guidelines on the sizing of holding tubes, but if large dispersions in residence times exist, nonuniformity of processing could still occur.

From the standpoint of the researcher in academia, the challenge is to gain a deeper understanding of flow phenomena in order to help design systems of the future. Is it possible, for example, to design a holding tube which provides minimal residence time distribution and yet achieves high fluid-to-particle heat transfer coefficients? Is it possible to develop generalized flow models to accurately predict trajectories of particle populations? These questions and other related ones can only be answered by long term research.

LIST OF SYMBOLS

Symbol	**Meaning**
a	radius of sphere (m)
c	a factor of friction
d	particle diameter (m)
D	pipe diameter (m)
e_c	capsule surface roughness
F_l	lift forces (N)
L	pipe length (m)
n	power law exponent
V	velocity of particle (m/s)
V_c	capsule velocity (m/s)
V_{av}	average fluid velocity (m/s)

Greek Letters

$\dot{\gamma}$	shear rate (s^{-1})
κ	magnitude of velocity gradient (s^{-1})
μ	viscosity (Pa s)
ν	kinematic viscosity (m^2/s)
ρ, ρ_f	fluid density (kg/m^3)
σ	capsule density (kg/m^3)
τ	shear stress (Pa)
Ω	angular velocity (radians/s)

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ESTIMATION OF HEAT OF MOISTURE SORPTION AND IMPROVED CRITERIA FOR EVALUATING MOISTURE SORPTION ISOTHERM EQUATIONS FOR FOODS

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ABSTRACT

Heat of sorption of moisture on foods can be estimated from sorption isotherm equations having both moisture and temperature as independent variables. Sorption thermodynamics dictate that the heat of moisture sorption should decrease with increased moisture concentration, or increased temperature of food, provided no biological, chemical or other changes occur during sorption. This fact,

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and the Clausius-Clapeyron equation were used to derive two analytical formulae to evaluate the reliability of moisture sorption equations in the calculation of heat of sorption. As examples, Chung and Pfost, modified Harkins and Jura, modified Henderson, and Guggenheim-Anderson-De Boer equations at specified conditions were analyzed.

INTRODUCTION

Estimation of heat of sorption in food processing operations like drying and baking, simultaneous heat and moisture transfer, and moisture changes during storage is important for the quantitative evaluation of these processes. Even for foods of relatively high moisture content, it is not a good approximation to take the total heat of sorption as the heat of evaporation/condensation of pure water for precise calculations. In intermediate and lower moisture foods, the enthalpy of sorption generally increases with a decreasing moisture content, and/or a decreasing temperature (Zuritz and Singh 1985).

Since experimental data are not usually available one needs frequently to estimate the heat of sorption of food products. For many food products this has been done by using the Clausius-Clapeyron equation: wheat flour, starch and gluten (Bushuk and Winkler 1957), corn (Chung and Pfost 1967), wheat (Becker and Sallans 1956), rice (Zuritz and Singh 1985), and rough rice (Wang 1978; Zahed 1982). These methods involve graphical and/or integral forms of the Clausius-Clapeyron equation. Since the moisture sorption equation of most foods already exist in the literature (Iglesias and Chirife 1982) or can be determined experimentally, the method described in this study can provide the estimation of the heat of sorption in an equation form by using the available moisture sorption equation. The equation form is more flexible and easier to use than graphical forms. However, this requires that the sorption equation be a function of both moisture content and temperature. Many sorption equations (such as BET) do not have temperature as an independent variable. However, there are many isotherm equations that satisfy the above requirement. (Iglesias and Chirife 1976; Balaban 1984; Weisser 1985; and Chung and Pfost 1967).

The purpose of the method described here is: (1) To estimate the heat of sorption of foods, if the moisture sorption equation is known. (2) To determine the range of validity of these equations for the purpose of calculating heat of sorption.

MATERIALS AND METHODS

The heat of sorption of water on a substance at any temperature and moisture content can be expressed as (refer to Nomenclature for definition of symbols):

$$\Delta H = \Delta H(M,T) \qquad [1]$$

It is possible to develop an expression for ΔH if the moisture sorption equation is available for a given material, by using the Clausius-Clapeyron equation. For a phase change:

$$\frac{\partial P_v}{\partial T} = \frac{\Delta H}{T\,\Delta V} \qquad [2]$$

For liquid-vapor phase changes one can neglect the volume of the liquid phase compared with the volume of vapor (Singh and Zuritz 1985b):

$$\Delta V \approx V_{vapor} \qquad [3]$$

The critical pressure of water is 218.2 atm and its reduced pressure is 0.0045 at atmospheric pressure. The compressibility factor at this reduced pressure is very close to one for normal temperatures (Wark 1977). Therefore one can apply the ideal gas law for one mol of vapor with negligible error (Zuritz and Singh 1985b):

$$V_{vapor} = \frac{R'\,T}{P_{vapor}} \qquad [4]$$

Substituting into [1]:

$$\Delta H = R'\,T^2 \frac{\partial}{\partial T}(\ln P_{vapor}) \qquad [5]$$

Since the value of the fugacity coefficient is generally unity at a pressure of 1 atm (Balzhiser 1972), one can define:

$$a_w = \frac{P_{vapor}}{P_{sat}} \qquad [6]$$

Then:

$$\Delta H = R'\,T^2 \frac{\partial}{\partial T}(\ln P_{sat} + \ln a_w) \qquad [7]$$

For pure water, $a_w = 1$ and Eq. [7] becomes:

$$\lambda = R'\,T^2 \frac{\partial}{\partial T}(\ln P_{sat}) \qquad [8]$$

Therefore, Eq. [7] can be expressed as:

$$\Delta H = \lambda + R'\,T^2 \frac{\partial}{\partial T}(\ln a_w) \qquad [9]$$

The saturation vapor pressure P_{sat} of water can be expressed in several ways. Two equations were evaluated for their suitability to calculate λ. For 0–200 °C range P_{sat} is expressed in terms of T as (Anon. 1985):

$$\ln P_{sat} = \frac{C_8}{T} + C_9 + C_{10} T + C_{11} T^2 + C_{12} T^3 + C_{13} \ln T \qquad [10]$$

Therefore:

$$\frac{\partial}{\partial T}(\ln P_{sat}) = -\frac{C_8}{T^2} + \frac{C_{13}}{T} + C_{10} + 2 C_{11} T + 3 C_{12} T^2 \qquad [11]$$

$$\lambda = R'(- C_8 + C_{13} T + C_{10} T^2 + 2 C_{11} T^3 + 3 C_{12} T^4) \qquad [12]$$

Another equation describing the change of P_{sat} with T is given by Kiefer (1941):

$$\ln P_{sat} = 54.51 - \frac{6887}{T} - 5.31 \ln T \qquad [13]$$

Therefore:

$$\frac{\partial}{\partial T} (\ln P_{sat}) = \frac{6887}{T^2} - \frac{5.31}{T} \qquad [14]$$

and:

$$\lambda = R' \left[6887 - 5.31 T \right] \qquad [15]$$

The plot of Eq. [12] and [15] for λ vs. T together with the λ values for pure water taken from Perry (1973) is shown in Fig. 1. It can be seen that the three parameter Kiefer equation gives a better estimate of the λ value for the range considered than the six parameter ASHRAE equation. One should note that Eq. [10] and Eq. [13], and their constants are for the purpose of calculating P_{sat}, and not △H. Therefore, although a six parameter equation may describe P_{sat} better than a three parameter model, this is not necessarily true for △H.

Since Eq. [13] was more accurate in the calculation of △H, it was used from this point on.

It is known that △H usually increases with decreasing T and with decreasing water content. Therefore:

$$\frac{\partial}{\partial T} (\Delta H) < 0 \qquad [16]$$

and

$$\frac{\partial}{\partial M} (\Delta H) < 0 \qquad [17]$$

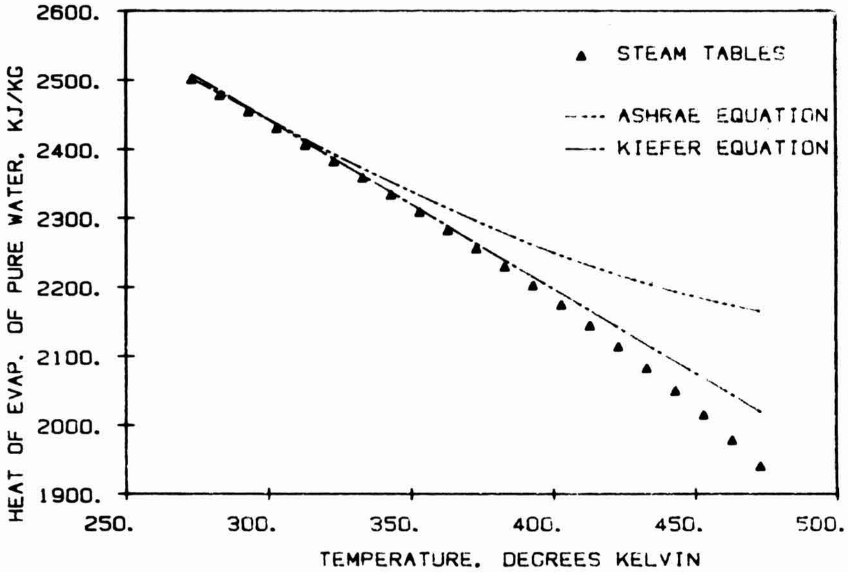

FIG. 1. COMPARISON OF λ VALUES FOR PURE WATER, CALCULATED FROM THE ASHRAE AND KIEFER VAPOR PRESSURE EQUATIONS

Equations [16] and [17] constitute the criteria by which the validity of using a moisture sorption equation to calculate heat of sorption is measured. Substituting:

$$\frac{\partial}{\partial T}(\Delta H) = \frac{\partial}{\partial T}\left\{ R'T^2 \frac{\partial}{\partial T}(\ln a_w) + \lambda \right\} < 0 \qquad [18]$$

and since λ is a function of temperature only:

$$\frac{\partial}{\partial M}(\Delta H) = \frac{\partial}{\partial M}\left\{ R'T^2 \frac{\partial}{\partial T}(\ln a_w) \right\} < 0 . \qquad [19]$$

And from Eq. [15]:

$$\frac{\partial}{\partial T}(\lambda) = -\ 5.31\ R' \qquad [20]$$

It can be seen that the derivative of λ in Eq. [20] is always negative. Since the heat of evaporation of water decreases with an increase in temperature, theoretically Eq. [15] can be used regardless of temperature range to calculate λ.

Only moisture sorption equations having both T and M (or W) as variables can be evaluated by using Eq. [18] and [19], and their range of validity in the calculation of heat of sorption can be found.

RESULTS AND DISCUSSION

Four isotherm equations having both moisture content and temperature as variables were analyzed for their suitability to calculate △H, and their validity range investigated. These equations are:

(1) Chung and Pfost equation. (Zuritz and Singh 1985)
(2) Modified Harkins and Jura equation. (Balaban 1984)
(3) Day and Nelson's modified Henderson equation. (Zuritz and Singh 1985)
(4) Guggenheim-Anderson-De Boer equation (GAB). (Weisser 1985).

Chung and Pfost Equation:

$$a_w = \exp\left[c_o\, T^{c_1} \exp\left(c_2\, T^{c_3}\, W\right)\right] \qquad [21]$$

$$\Delta H = R'\left\{c_o\, T^{c_1+1} \exp(c_2\, T^{c_3}\, W)\left[c_1 + c_2\, c_3\, W\, T^{c_3}\right]\right\} + \lambda \qquad [22]$$

Also:

$$\frac{\partial}{\partial T}(\Delta H) = c_o\, R'\left\{\exp(c_2\, T^{c_3}\, w)\, T^{c_1+1}\left\{(c_1 + 1)\frac{1}{T}(c_1 + c_2\, c_3\, w\, T^{c_3}) + (c_1 + c_2\, c_3\, w\, T^{c_3})\, c_2\, w\, c_3\, T^{c_3-1} + c_2\,(c_3)^2\, w\, T^{c_3-1}\right\}\right. - 5.31\, R' \qquad [23]$$

$$\frac{\partial}{\partial W}(\Delta H) = R'\, c_o\, T^{c_1+1} \exp(c_2\, T^{c_3}\, W)\, c_2\, T^{c_3}\left\{c_1 + c_2\, c_3\, W\, T^{c_3} + c_3\right\} \qquad [24]$$

Figure 2 is the plot of △H versus T, using the Chung and Pfost isotherm at different moisture contents for ceral grains(Chung and Pfost 1967). The numerical values of constants c_0, c_1, c_2, and c_3 are taken from Zuritz and Singh (1985), and are shown in Table 1. Using these, and Eq. [23] and [24] one can evaluate if the estimation of the △H of sorption based on the Chung and Pfost equation in given conditions is appropriate. The slope of the curves in Fig. 2 represents $\partial(\Delta H)/\partial T$ as expressed by Eq. [23]. For all moisture contents considered, the slope is negative. Therefore, Chung and Pfost equation passes the criterion of Eq. [16].

Fig. 3 is the plot of △H versus W at different temperatures. The slope $\partial(\Delta H)/\partial W$ as expressed by Eq. [24] is negative for all temperature ranges considered and conforms to the criterion of Eq. [17].

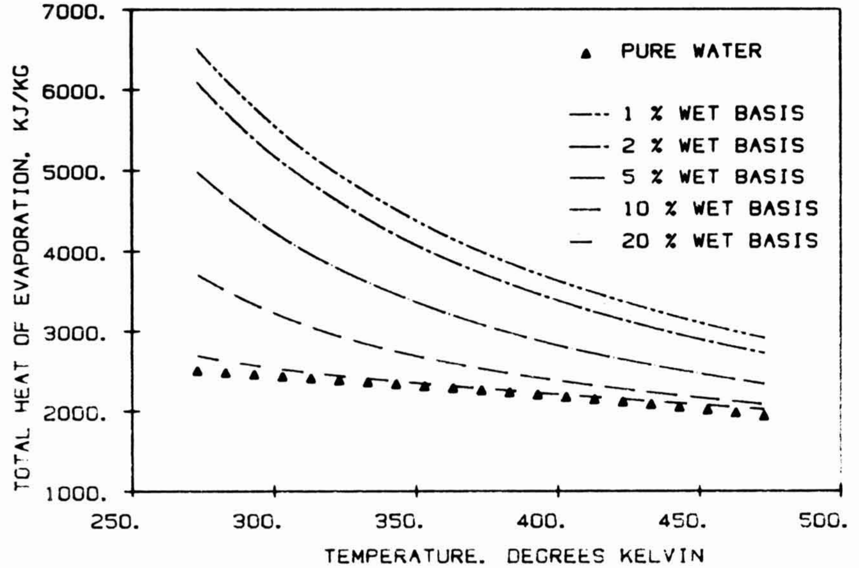

FIG. 2. TOTAL HEAT OF EVAPORATION VERSUS TEMPERATURE, CALCULATED FROM THE CHUNG AND PFOST ISOTHERM EQUATION

TABLE 1.
VALUES OF CONSTANTS USED IN EQUATIONS

Chung and Pfost — Ref: Zuritz and Singh 1985a

$c_0 = -3.883686\ 10^9$ $c_1 = -3.52486$
$c_2 = -1.1205\ 10^{-4}$ $c_3 = 1.30047$

Modified Harkins and Jura — Ref: Balaban 1984

$b_1 = 9.5018\ 10^{-5}$ $b_2 = 3.114242\ 10^{-2}$ $b_3 = 1.311031\ 10^{-4}$

Day & Nelson's modified Henderson — Ref: Zuritz and Singh 1985a

$a_0 = -5.9672\ 10^{-57}$ $a_1 = 21.596937$
$a_2 = 2.75096\ 10^6$ $a_3 = -2.453454$

Guggenheim-Anderson-De Boer — Ref: Weisser 1985

	Least squares	Nonlinear fit
$C' =$	$2.18\ 10^{-2}$	$7.56\ 10^{-4}$
$K' =$	1.292	0.927
$H_1 - H_m =$	16.40	24.29
$H_\ell - H_m =$	−0.69	0.11
$W'_m =$	1.095	4.027
$W''_m =$	332.	−46.7

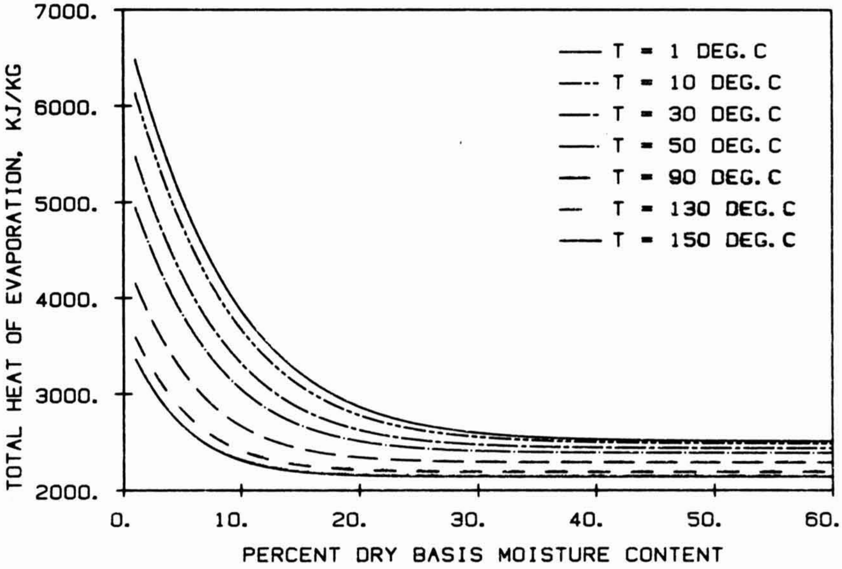

FIG. 3. TOTAL HEAT OF EVAPORATION VERSUS MOISTURE CONTENT, CALCULATED FROM THE CHUNG AND PFOST ISOTHERM EQUATION

Since both criteria are met, one can conclude that the Chung and Pfost equation can be used to estimate the heat of sorption of water on rough rice within the temperature and moisture ranges considered. It should be emphasized that these predicted values of △H are only estimates.

Modified Harkins Jura Equation:

$$a_w = \exp\left[\frac{\frac{1}{M^2} - b_1}{b_2 - b_3 T} \right] \qquad [25]$$

$$\Delta H = R' \left[\frac{T^2 b_3 \left(1/M^2 - b_1 \right)}{\left(b_2 - b_3 T \right)^2} \right] + \lambda \qquad [26]$$

$$\frac{\partial}{\partial T}(\Delta H) = R' \left\{ 2 T b_3 b_2 \frac{\left(\frac{1}{M^2} - b_1 \right)}{(b_2 - b_3 T)^3} - 5.31 \right\} \qquad [27]$$

$$\frac{\partial}{\partial M}(\Delta H) = \frac{2 R' T^2 b_3}{(b_2 - b_3 T)^2 M^3} \qquad [28]$$

Figure 4 is the plot of △H versus T, using the modified Harkins Jura isotherm at different moisture contents in Ocean Perch. The numerical values of constants b_1, b_2 and b_3 are taken from Balaban (1984), as shown in Table 1. The slopes of the curves in Fig. 4 represent $\partial(\triangle H)/\partial T$ and are negative for all moisture content ranges considered for Ocean Perch.

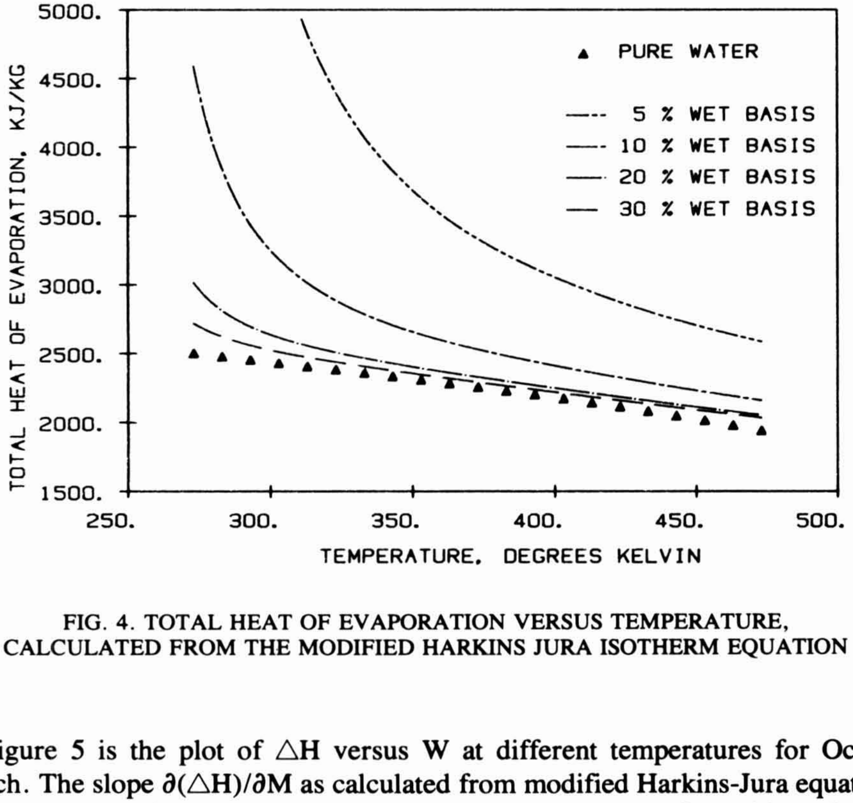

FIG. 4. TOTAL HEAT OF EVAPORATION VERSUS TEMPERATURE, CALCULATED FROM THE MODIFIED HARKINS JURA ISOTHERM EQUATION

Figure 5 is the plot of △H versus W at different temperatures for Ocean Perch. The slope $\partial(\triangle H)/\partial M$ as calculated from modified Harkins-Jura equation is also negative for all temperature ranges considered. Therefore, this moisture sorption equation can be used to estimate the heat of sorption of water on Ocean Perch, within the temperature and moisture content ranges considered.

Day and Nelson's modified Henderson Equation:

$$a_w = 1 - \exp\left[a_o \, T^{a_1} \, W^{a_2 T^{a_3}} \right] \quad [29]$$

$$\ln(a_w) = \ln\left\{ 1 - \exp\left[a_o \, T^{a_1} \, W^{a_2 T^{a_3}} \right] \right\} \quad [30]$$

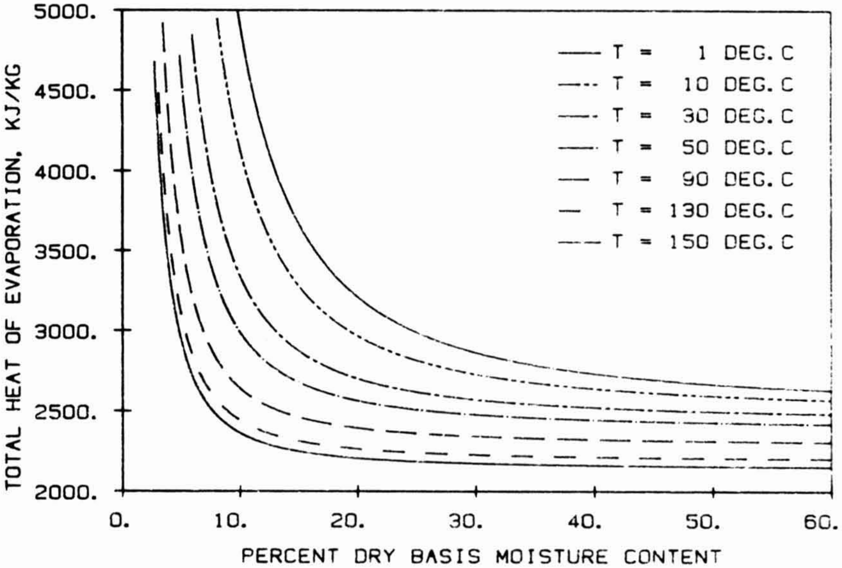

FIG. 5. TOTAL HEAT OF EVAPORATION VERSUS MOISTURE CONTENT, CALCULATED FROM THE MODIFIED HARKINS JURA ISOTHERM EQUATION

Let:

$$z_1 = W^{a_2 T^{a_3}} \qquad [31]$$

$$z_2 = a_0 T^{a_1} \qquad [32]$$

Then:

$$\Delta H = R' \left[\frac{-\exp(z_1 z_2)}{1 - \exp(z_1 z_2)} z_1 z_2 T \left\{ a_1 + \ln(W) a_2 a_3 T^{a_3} \right\} \right] + \lambda \qquad [33]$$

Figure 6 is the plot of △H versus T, using Day and Nelson's modified Henderson isotherm at different moisture contents for rough rice. The numerical values of constants a_0, a_1, a_2, a_3, are taken from Zuritz and Singh (1985a), as shown in Table 1. The slope of the curves in Fig. 6 represents $\partial(\triangle H)/\partial T$. It can be seen that for some range of temperature below 350°K, the slope becomes positive, and thus violates the criterion of Eq. [16].

$$\frac{\partial}{\partial T}(\Delta H) = R'\left\{\frac{-\exp(z_1\ z_2)\ z_1\left[\ a_0\ a_1\ T^{a_1-1} + \ln(W)\ a_2\ a_3\ T^{a_3-1}\ z_2\ \right]}{\left[\ 1 - \exp(z_1\ z_2)\ \right]^2}\right.$$

$$z_1\ z_2\ T\ (\ a_1 + \ln(W)\ a_2\ a_3\ T^{a_3}) + \left[\ z_1\ (\ a_0\ a_1\ T^{a_1-1} + \right.$$

$$\left.\ln(W)\ a_2\ a_3\ T^{a_3-1}\ z_2)\right]\ T\ \frac{-\exp(\ z_1\ z_2)}{1 - \exp(\ z_1\ z_2)}\ (\ a_1 + \ln(W)\ a_2\ a_3\ T^{a_3})$$

$$+ (z_1\ z_2)\ \frac{-\exp(\ z_1\ z_2)}{1 - \exp(\ z_1\ z_2)}\ (a_1 + \ln(W)\ a_2\ a_3\ T^{a_3}) + \ln(w)\ a_2\ (a_3)^2$$

$$\left. T^{a_3-1}\ \frac{-\exp(\ z_1\ z_2)}{1 - \exp(\ z_1\ z_2)}\ z_1\ z_2\ T - 5.31\ \right\} \qquad [34]$$

$$\frac{\partial}{\partial w}(\Delta H) = R'\ z_2\ T\left\{ - \frac{\exp(z_1\ z_2)(a_2\ T^{a_3}\ \frac{z_1\ z_2}{w}}{\left[\ 1 - \exp(z_1\ z_2)\right]^2}\ z_1\left[\ a_1 + \ln(W)\ a_2\right.\right.$$

$$\left. a_3\ T^{a_3}\right] + (\ a_2\ T^{a_3}\ \frac{z_1}{w})(\frac{-\exp(\ z_1\ z_2)}{1 - \exp(\ z_1\ z_2)})\left[\ a_1 + \ln(W)\ a_2\ a_3\right.$$

$$\left.\left. T^{a_3}\right] + z_1\ (\frac{-\exp(\ z_1\ z_2)}{1 - \exp(\ z_1\ z_2)})\ \frac{a_2\ a_3\ T^{a_3}}{w})\ \right\} \qquad [35]$$

Figure 7 is the plot of △H versus W at different temperatures. It can be seen that for 1 °C and for 10 °C, the slope is slightly positive, thus violating the criterion of Eq. [17]. Therefore, this equation should not be used within the temperature and moisture ranged indicated in Fig. 6 and 7 to estimate the heat of sorption of rough rice.

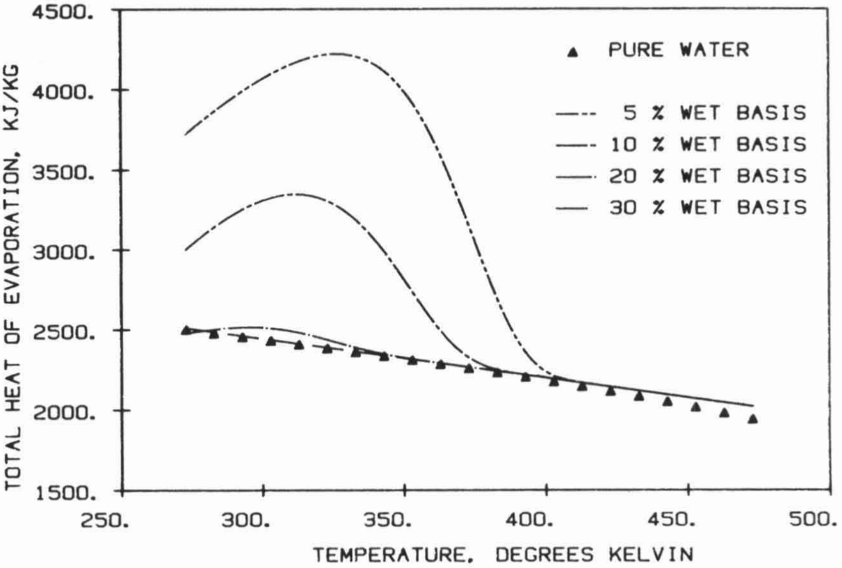

FIG. 6. TOTAL HEAT OF EVAPORATION VERSUS TEMPERATURE, CALCULATEI FROM THE DAY AND NELSON'S MODIFIED HENDERSON ISOTHERM EQUATIOI

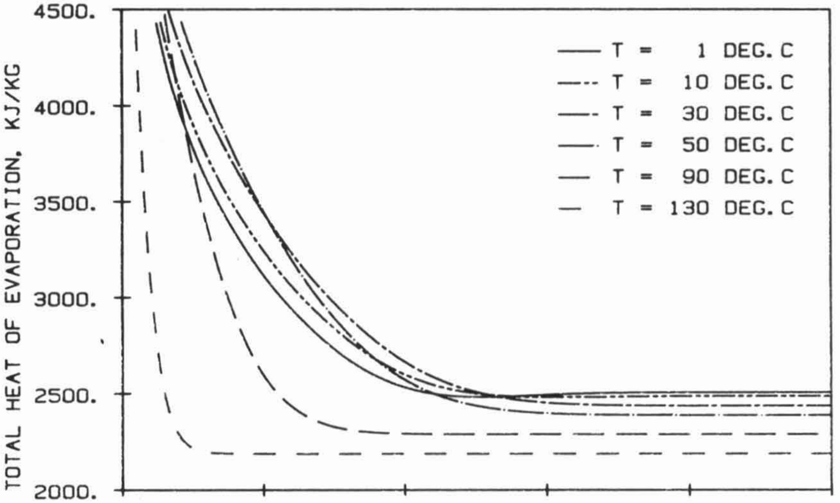

FIG. 7. TOTAL HEAT OF EVAPORATION VERSUS MOISTURE CONTENT, CALCULATED FROM THE DAY AND NELSON'S MODIFIED HENDERSON ISOTHERM EQUATION

Guggenheim-Anderson-De Boer Equation (GAB):

$$\frac{W}{W_m} = \frac{C\ K\ a_w}{(1 - K\ a_w)(1 - K\ a_w + C\ K\ a_w)} \qquad [36]$$

Where :

$$C = C(T) = C'\ \exp\left[(H_1 - H_m)/R''T \right] \qquad [37]$$

$$K = K(T) = K'\ \exp\left[(H_\ell - H_m)/R''T \right] \qquad [38]$$

$$W_m = W_m(T) = W'_m\ \exp\left[W''_m/T \right] \qquad [39]$$

$$a_w = \frac{-\ BB\ -\ (BB^2 - 4\ AA\ CC)^{1/2}}{2\ AA} \qquad [40]$$

Where:

$$AA = W\ K^2\ (1 - C) \qquad [41]$$

$$BB = K\ \left[C\ (W - W_m) - 2\ W \right] \qquad [42]$$

$$CC = W$$

With the restriction:

$$BB^2 - 4\ AA\ CC \geq 0 \qquad [43]$$

Then:

$$\Delta H = R'\ T^2\ \frac{\partial}{\partial T}(\ln a_w) + \lambda \qquad [44]$$

The $\triangle H$ equation is too complex to attempt the $\partial(\triangle H)/\partial T$ and $\partial(\triangle H)/\partial W$ derivatives for practical purposes.

The numerical values of constants C', K', H_1, H_ℓ, H_m, W'_m, W''_m are taken from Weisser (1985), as seen from Table 1. The constants were calculated from experimental data by using both a least squares method and a nonlinear regression method (Weisser 1985). Figure 8 is the plot of $\triangle H$ versus T, using GAB isotherm at different moisture contents for ground coffee by using the constants calculated by the least squares method. The slope of the curves in Fig. 8 represents $\partial(\triangle H)/\partial T$, and is negative for the moisture contents considered.

Figure 9 represents the $\triangle H$ versus moisture content at different temperatures. The slope $\partial(\triangle H)/\partial W$ is negative for all temperatures considered except for 1 °C and below 3% moisture. Therefore, the GAB isotherm equation can be used to estimate heat of sorption values within the ranges of temperatures and moisture contents where the slopes are negative.

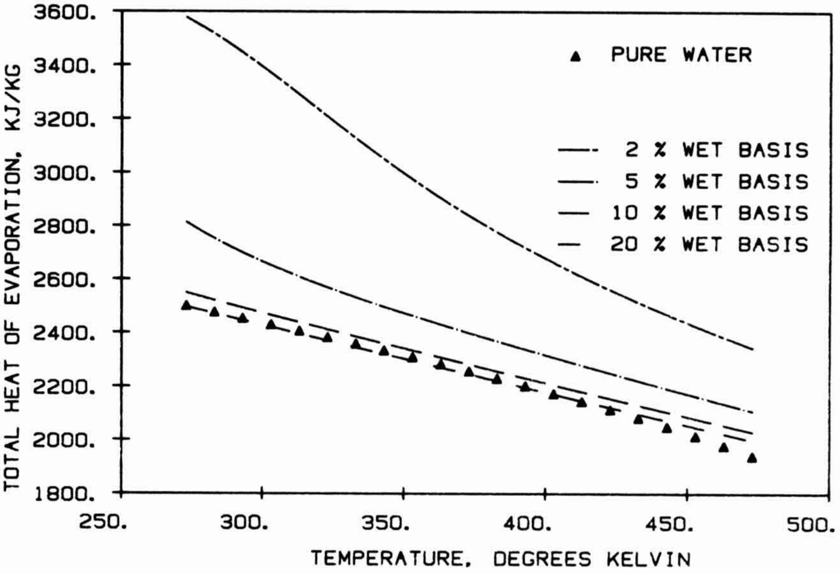

FIG. 8. TOTAL HEAT OF EVAPORATION VERSUS TEMPERATURE, CALCULATED FROM THE GUGGENHEIM-ANDERSON-DE BOER ISOTHERM EQUATION
Parameters calculated by a least squares method.

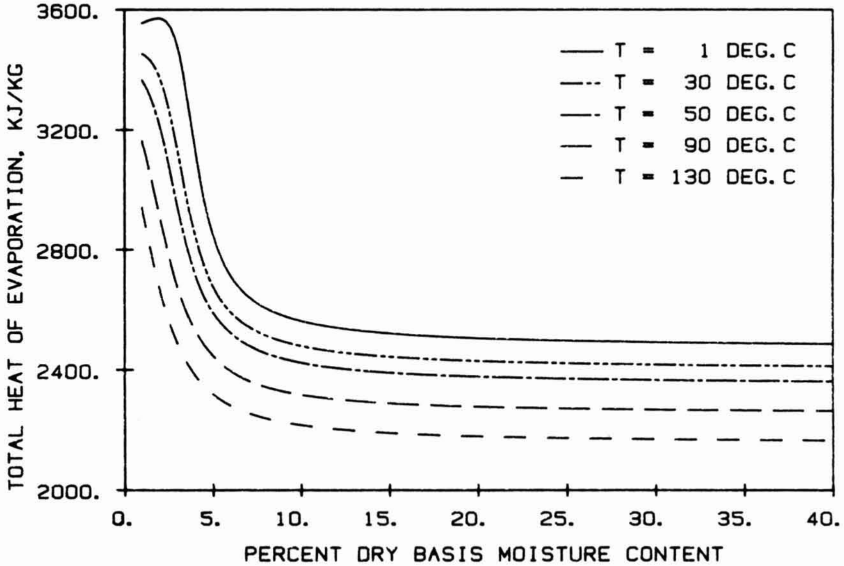

FIG. 9. TOTAL HEAT OF EVAPORATION VERSUS MOISTURE CONTENT, CALCULATED FROM THE GUGGENHEIM-ANDERSON-DE BOER ISOTHERM EQUATION
Parameters calculated by a least squares method.

Figure 10 is the plot of △H versus T at different moisture contents for the GAB equation, but for which the constants were calculated by a nonlinear regression method from the same experimental data used in Fig. 8 and 9 (Weisser 1985). The spike and dip at various moisture contents around 406 °K is unusual. Around this temperature, the equation has a disturbance. Since all calculations were in double-precision, this should not be due to numerical error enhancements. It would not be safe to estimate heat of sorption using this equation with these constants and within the specified T and W ranges.

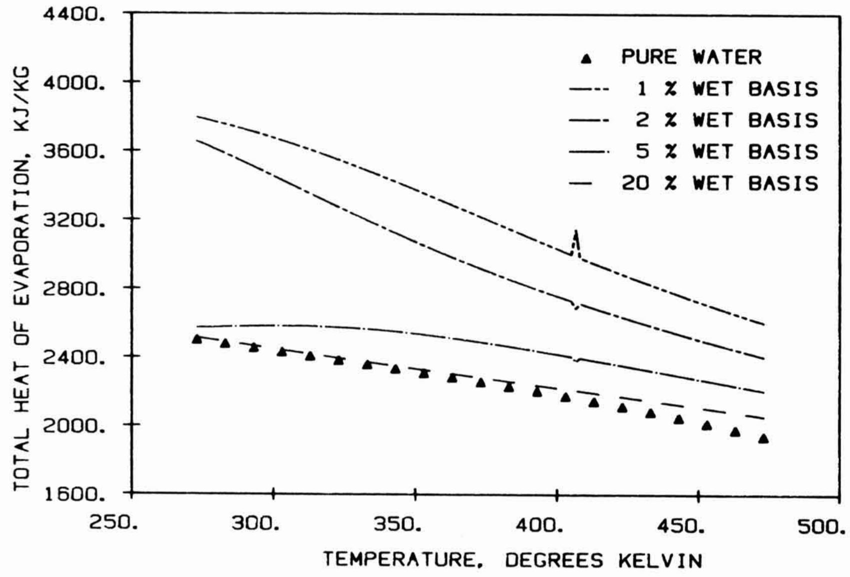

FIG. 10. TOTAL HEAT OF EVAPORATION VERSUS TEMPERATURE, CALCULATED FROM THE GUGGENHEIM-ANDERSON-DE BOER ISOTHERM EQUATION
Parameters calculated by a nonlinear fit method.

Figure 11 is the plot of △H versus moisture content at different temperatures for the same equation with constants calculated by nonlinear regression. Although the slopes of the curves are negative, one can observe a disturbance around 5% moisture, for temperatures of 1, 30 and 50 °C where all curves come together. This means that around 5% moisture temperature has no effect on enthalpy of sorption. Since temperature has an effect on the enthalpy of sorption both lower and higher than 5% moisture content, this behavior has no obvious explanation.

It should be noted that for the same GAB equation, and for the same experimental data, there can be differences in the estimated △H values depending on the method of calculation of GAB constants.

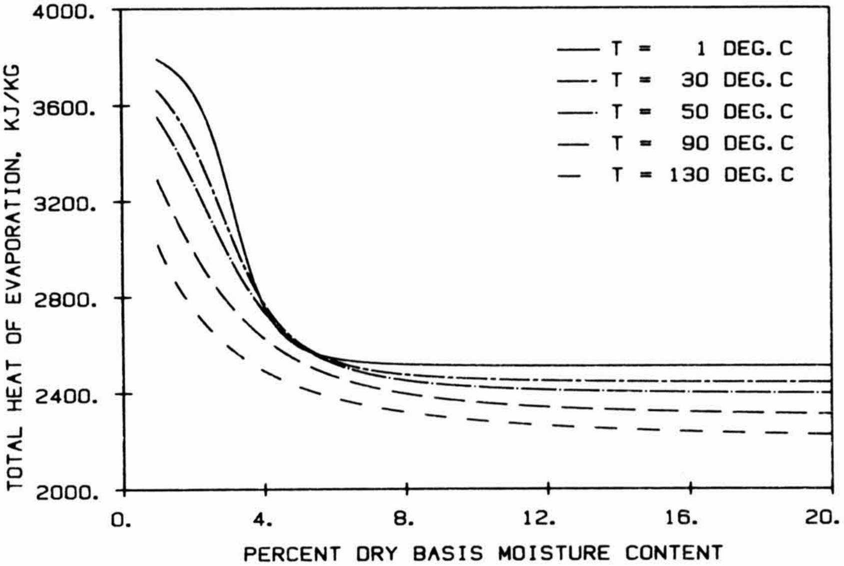

FIG. 11. TOTAL HEAT OF EVAPORATION VERSUS MOISTURE CONTENT, CALCULATED FROM THE GUGGENHEIM-ANDERSON-DE BOER ISOTHERM EQUATION
Parameters calculated by a nonlinear fit method.

As a summary, it was shown that enthalpy of sorption can be estimated by this method when: (1) the experimental $\triangle H$ data is not available for a given food material, in a given range of T and W, (2) The moisture sorption equation is available for the food considered in that particular T and W range, and has both T and W as independent variables, and (3) The moisture sorption equation conforms to the criteria discussed in this work, within the given range of T and W. The accuracy of these estimations should be confirmed by experimental data, when possible. Even if both criteria are met for a given moisture sorption equation, this does not mean that the predicted heat of sorption will be close to the "real" value. But if the criteria are not met, the equation can not be used within that particular range of T and W. Therefore, the criteria are necessary, but not sufficient for an acceptable prediction of the heat of sorption.

NOMENCLATURE

a_0, a_1, a_2, a_3: Experimentally determined constants for Day and Nelson equation
b_1, b_2, b_3: Experimentally determined constants for modified Harkins Jura equation
c_0, c_1, c_2, c_3: Experimentally determined constants for Chung and Pfost equation
C' = Entropic accomodation factor
$C_8 = -5800.2206$
$C_9 = 1.3914993$
$C_{10} = -0.04860239$
$C_{11} = 0.41764768\ 10^{-4}$
$C_{12} = -0.14452093\ 10^{-7}$
$C_{13} = 6.5459673$
H_1 = Total heat of sorption of the first layer on primary sites KJ/kg
H_ℓ = Heat of condensation of pure water, KJ/kg
H_m = Total heat of sorption of the multilayer which differs from the heat of condensation of pure liquid water, KJ/kg
K' = Entropic accomodation factor
M = Wet basis moisture content, kg H_2O/kg total
P_v ^{5} Partial pressure of water vapor, Pa
P_{sat} = Vapor pressure of pure water, Pa
R' = Gas constant, 0.44614 KJ/kg °K
R'' = Gas constant, $8.3144\ 10^{-3}$ KJ/g-mol °K
T = Temperature °K
W = Dry basis moisture content, kg H_2O/kg dry material
W_m = Water content on dry basis at fully occupied active sorption sites with one molecule of water. (monolayer in BET theory)
W'_m = Pre-exponential constant
W''_m = Temperature coefficient
$\triangle H$ = Total heat of sorption, KJ/kg
$\triangle V$ = Volume, m^3
λ = Heat of evaporation of pure water, KJ/kg

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