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**Journal of  
FOOD PROCESS  
ENGINEERING**

**Edited by  
D. R. HELDMAN**

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## JOURNAL OF FOOD PROCESS ENGINEERING

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## CONTENTS

Meetings . . . . .	vii
Temperature Distributions and Liquid-Side Heat Transfer Coefficients in Model Liquid Foods in Cans Undergoing Flame Sterilization Heating R. L. MERSON, S. J. LEONARD, EARNEST MEJIA and J. HEIL, University of California, Davis, California . . . . .	85
Effects of Processing on Adsorption of Off-Flavors Onto Soy Protein ALAN CROWTHER, Celanese Chemical Company, Bishop, Texas, LESTER A. WILSON and CHARLES E. GLATZ, Iowa State Uni- versity, Ames, Iowa . . . . .	99
Effect of Deformation Rate and Moisture Content on Rice Grain Stiff- ness P. D. CHATTOPADHYAY, D. D. HAMANN and J. R. HAMMER- LE, North Carolina State University, Raleigh, North Carolina .	117
High-Temperature Drying-Cum-Parboiling of Paddy S. ZAKI UDDIN ALI and KSHIROD R. BHATTACHARYA, Cen- tral Food Technological Research Institute, Mysore, India . .	123

## MEETINGS

### NOVEMBER 1981

**November 8—11: THE NATIONAL FROZEN FOOD CONVENTION.** San Francisco Hilton, San Francisco, California. Contact: National Frozen Food Convention, Administrative Office, 604 W. Derry Road, PO Box 398, Hersey, Pennsylvania 17033.

**November 15—19: FOOD AND DAIRY EXPO 81.** Georgia World Conference Center, Atlanta, Georgia. Contact: Dairy and Food Industry Supply Association, 5530 Wisconsin Ave., Suite 1050, Washington, D.C. 20015.

### DECEMBER 1981

**December 15—18: ASAE WINTER MEETING: LAND AND WATER RESOURCES — A CRITICAL RESPONSIBILITY.** Palmer House, Chicago, Illinois. Contact: ASAE Headquarters, PO Box 410, St. Joseph, Michigan 49085.



# TEMPERATURE DISTRIBUTIONS AND LIQUID-SIDE HEAT TRANSFER COEFFICIENTS IN MODEL LIQUID FOODS IN CANS UNDERGOING FLAME STERILIZATION HEATING

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## ABSTRACT

*Heat transfer studies were performed with clay suspensions and sugar solutions in 303 × 406 cans heated singly in a flame process simulator. Experimental results show that the can wall temperature does not reach excessive temperatures and that the temperature distribution throughout the can is uniform. The slowest heating point was on the axis near the end of the can but this temperature lagged the center temperature by less than 3°C even with adverse heating conditions (i.e., no head space).*

*The liquid-side heat transfer coefficient in 60% sucrose solution at 30 rpm was measured to be  $466 \pm 34 \text{ W/m}^2 \text{ K}$  based on the cylindrical area of the can. Rotational speed had only a small effect on heating rate in the range of 10 to 120 rpm.*

## INTRODUCTION

Flame sterilization, also known as Steriflamme, is a process in which a gas flame is applied directly to the external surface of rapidly rotating cans filled with convection-heating foods. The process has been applied to many products containing liquids in a wide range of consumer can sizes (Casimir 1975; Leonard *et al.* 1975a). Quality retention in the products is improved to the extent that high temperature, short time processing is achieved (CSIRO 1972; Leonard *et al.* 1975 b,c). Therefore, the process is engineered to take advantage of the high flame temperature, which promotes a high rate of heat transfer to the outside of the can by convection and radiation. The flame temperature is in the range of 1100 to 1650°C (2000 to 3000°F) depending on combustion conditions. However, the can surface temperature does not become excessive because the enhanced liquid side convection quickly transfers the heat to the interior of the can. Thus, even the region near the can wall is not over-heated and the process results in a product of uniform quality.

Flame sterilization machines have been successfully operated commercially in many places throughout the world. Nevertheless, little design information has appeared in the open literature (but see Wu 1971; Ojo 1972; and Fujiwara 1975) and it is possible that performance improvements could still be made through a study of the heat transfer mechanisms of Steriflamme machines. These mechanisms are not simple. For example, the temperature distributions in the food liquid and in the gas space surrounding the cans are difficult to measure or predict, and consequently convective heat transfer coefficients are difficult to determine. Radiation complicates the analysis of heat transfer from the flame.

The present paper begins to document the actual time/temperature treatment that each portion of the canned food receives during flame heating. Internal temperature distributions were mapped experimentally for a typical can size and indicated that a film model with bulk mixing in the interior of the can would be useful to describe the internal heat transfer. Therefore, a film heat transfer coefficient was defined and measured.

## METHOD

Model liquid foods, consisting of a clay suspension or a 60% sucrose solution, were sealed into 303 × 406 cans (7.8 cm diam. × 10.5 cm length, inside dimensions) at a pressure of 0.027 M Pa (22 in. Hg vacuum) and brought to a uniform initial temperature. The cans were then rotated over a propane flame in a process simulator which heated one can at a time (Leonard *et al.* 1975). The burner was 0.4 cm wide and 15 cm long and was oriented with its length at right angles to the axis of the can to approximate the configuration in a commercial machine. Each can was equipped with two thermocouples which were connected with slip rings to strip chart recorders. Temperatures were read from the continuous traces at selected times and plotted as a function of position in the can.

### Temperature Profiles

For mapping temperatures inside the can, insulated support wires were placed on a diameter at the appropriate axial position in the can and the ends were selded to the can walls. Two flexible thermocouple wires were attached to the support wires in each can at selected radial positions. The cans were then filled as full as possible (no headspace) with  $475 \pm 5$  g of 1% Virginia Bentonite clay suspension. After closing,

the cans were brought to 25°C (77°F) and rotated over the flame at 10, 30, 50, 80 and 120 rpm. Fuel gas flow rate was 650 cc/min (converted to standard temperature and pressure, STP) corresponding to a flame temperature of 993°C (1820°F) (Fujiwara and Merson 1976).

#### Heat Transfer Coefficient Experiment

For measuring the internal heat transfer coefficient, one flexible copper-constantan thermocouple for each can was welded directly onto the internal surface of the can at either the midpoint or 1.27, 2.54 or 3.81 cm (1/2, 1, or 1-1/2 in) from the midpoint. The other thermocouple was a rigid stainless steel needle type (Ecklund type CNL) located either at the geometric center of the can or 2.70 cm (1-1/16 in) from the end of the can. The cans were filled about three-quarters full (2.5 cm headspace) with 463 grams of a 60% sucrose solution and closed. They were cooled to a uniform 15.6°C (60°F) before each heating trial. Fuel gas flow rate for this experiment was 1380 cc/min at STP, corresponding to a flame temperature of 1200°C (2190°F) (Fujiwara and Merson 1976). The speed of rotation was held constant at 30 rpm. Heating trials were run in triplicate.

## RESULTS

#### Temperature Distributions

The actual locations of the thermocouples are shown by closed circles in Fig. 1. The open circles in the lower right hand quadrant show where temperatures which were measured on the left hand side were plotted on the graphs in the mirror image position, since symmetry across the center plane may be assumed. As one example of the data, Fig. 2 is a map of temperatures in the Bentonite solution after 8 min of rotation at 50 rpm. The temperature is reasonably uniform throughout the can with the highest temperature close to the wall near the flame and the lowest temperature on the axis near the end of the can.

Figure 3 shows the temperature distribution along the centerline of the can at 50 rpm after 0, 1, 2, 4, 6 and 8 min. The slowest heating point in the can is on the centerline near the end, but there is only a 2.8°C (5°F) difference between this temperature and the temperature at the center of the can. Figure 4 is a similar axial temperature profile at 50 rpm but with the thermocouples located only 0.32 cm (1/8 in) from the wall. The temperature maximum offset from the center plane may suggest that the edge of the flame is burning hotter than the center.

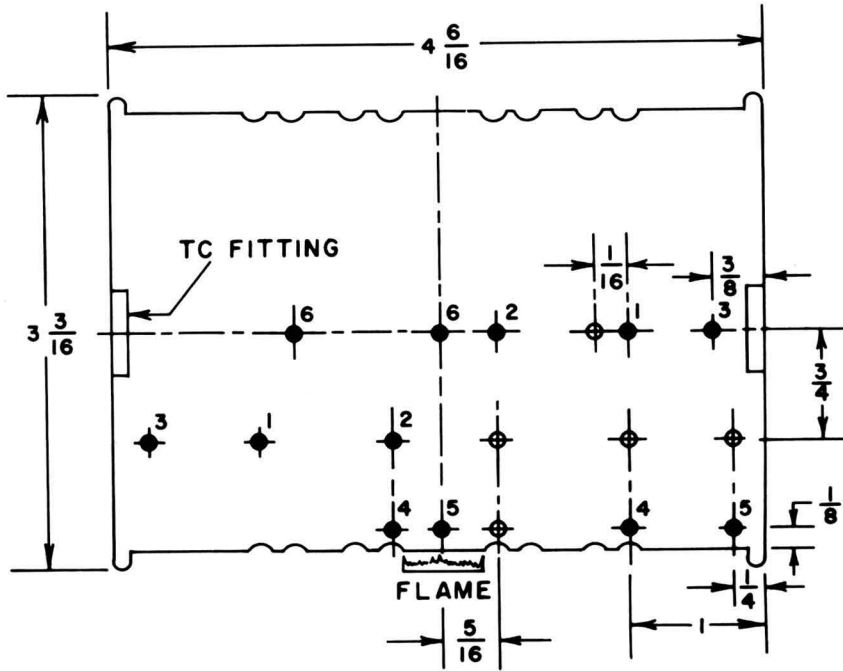


FIG. 1. LOCATION OF THERMOCOUPLES IN 303 X 406 CANS OF BENTONITE CLAY SUSPENSION

Each can (No. 1—6) had 2 thermocouples, ● actual position of TC, ○ image position as plotted in Fig. 2—5. Dimensions are in inches.

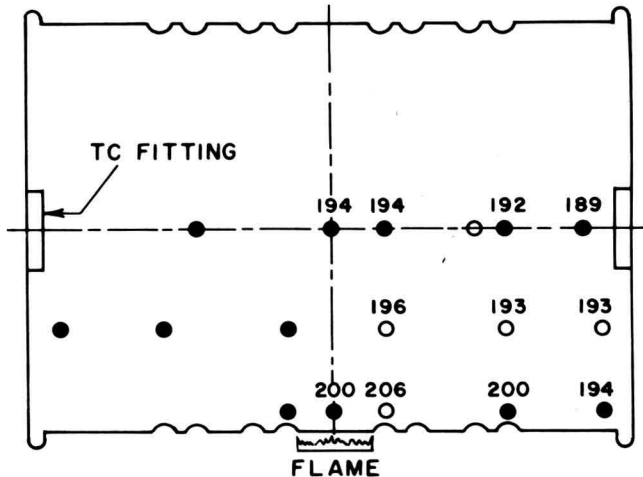


FIG. 2. EXPERIMENTAL TEMPERATURE DISTRIBUTION ( $^{\circ}$ F) IN 303 X 406 CAN OF 1% BENTONITE CLAY SUSPENSION, 50 rpm, BURNER GAS FLOW RATE 650 cc/min

Points correspond to Fig. 1.

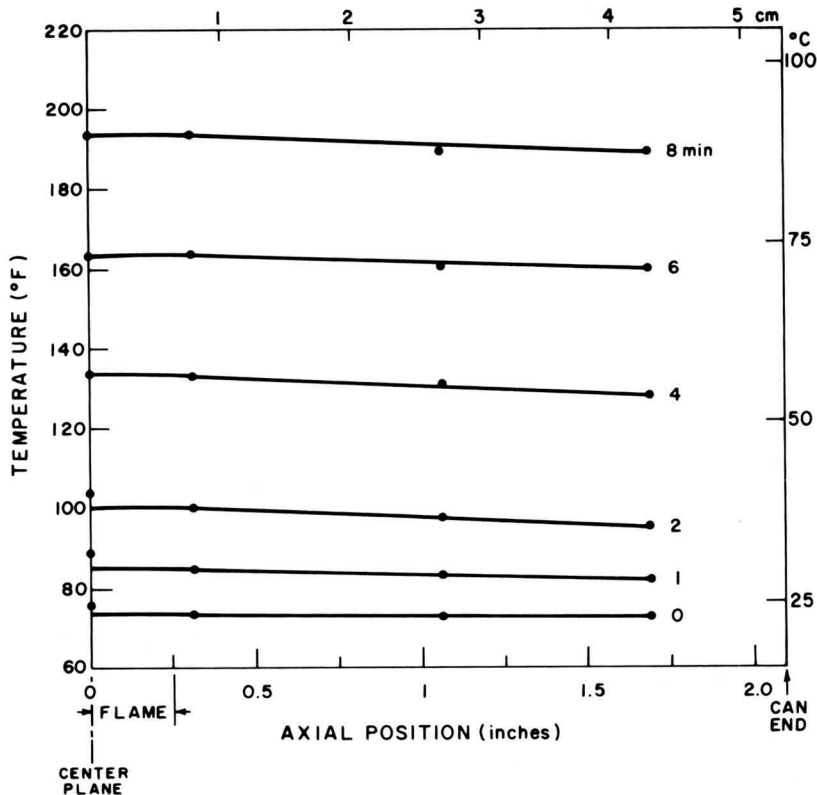


FIG. 3. AXIAL TEMPERATURE PROFILE ALONG CENTERLINE IN 303 X 406 CANS OF BENTONITE CLAY SUSPENSION, 50 rpm BURNER GAS FLOW RATE 650 cc/min

Figure 5 shows readings of thermocouples in cans 2 and 4 located 0.79 cm (5/16 in) from the center plane, over the edge of the flame. The profiles are quite flat except near the wall of the can.

Even for these very full cans, Fig. 2-5 and the other data collected show that the temperature distribution is uniform throughout the can except at the wall. This indicates the mixing within the can is good even under these conditions and suggests that a film model of heat transfer would be useful in calculating heat transfer rates.

The effect of rpm is illustrated in Fig. 6 by plotting the rate of heating of the slowest heating point measured in the can (thermocouple 3 on the axis of the can 0.95 cm [3/8 in] from the end). The rate of heating was calculated from

$$\dot{Q} = mC \frac{dT}{d\theta} \quad (1)$$

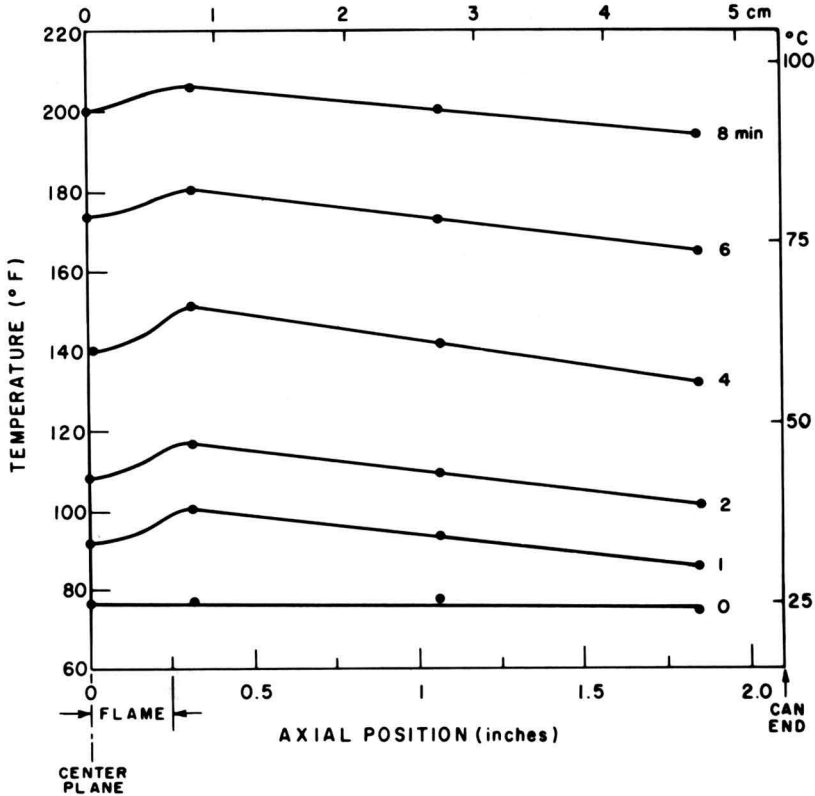


FIG. 4. AXIAL TEMPERATURE PROFILE 0.32 cm (1/8 in) FROM CAN WALL IN 303 X 406 CAN, 1% BENTONITE CLAY SUSPENSION, 50 rpm, BURNER GAS FLOW RATE 650 cc/min

by multiplying the slope of the heating curve for thermocouple 3,  $dT/d\theta$ , by the mass of the solution, 0.475 kg (1.047 lb<sub>m</sub>), and by the heat capacity, assumed to be 4.19 kJ/kg K (1.0 Btu/lb<sub>m</sub>°F). The effect of rotation is small as might be expected for a completely liquid product with no headspace. However, there is a broad maximum in the heating rate between 30 and 40 rpm which is the range of operation of commercial machines. The reason for the decrease in heat transfer rate above 40 rpm has not yet been determined, but it may be caused by enhanced cooling from the lower portions of the can that are not completely enveloped by the combustion gases rising from the narrow burner (Fujiwara and Merson 1976). Another reason may be disturbance of the flame at higher rpm by the momentum boundary layer around the can.

#### Surface Temperature Measurements

Figure 7 indicates the temperature distribution along the inside wall of cans containing 60% sugar solution. The data are plotted at one minute

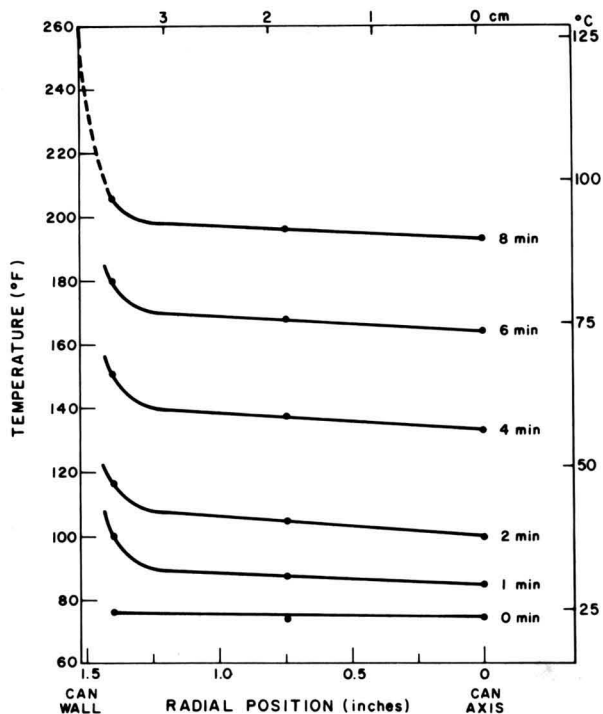


FIG. 5. RADIAL TEMPERATURE PROFILE IN 303 X 406 CAN, 1% BENTONITE CLAY SUSPENSION, 50 rpm, BURNER GAS FLOW RATE 650 cc/min

Thermocouples were located axially 0.79 cm (5/16 in) from center plane and radially 0, 1.9 and 3.5 cm (0 in, 3/4 in, and 1-3/8 in) from can axis.

intervals and each point represents the average of triplicate runs. The complete profile was obtained by making one run with the thermocouple on the right side of the flame and then reversing the can end to end for another trial. The inside surface temperature is fairly uniform except in the immediate vicinity of the flame.

## DISCUSSION

Because of the difficulty in preparing cans with thermocouple support wires it was desirable to use a fluid that could be heated repeatedly without a change in properties. It was also desirable that the fluid be more viscous than water to simulate sugar syrups or other viscous foods. There-

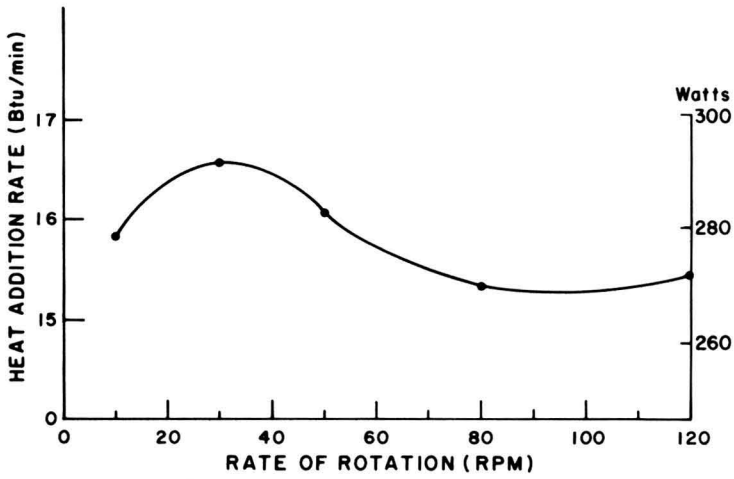


FIG. 6. EFFECT OF RPM ON RATE OF HEATING 1% BENTONITE CLAY SUSPENSION IN 303 X 406 CAN, FUEL GAS FLOW RATE 650 cc/min

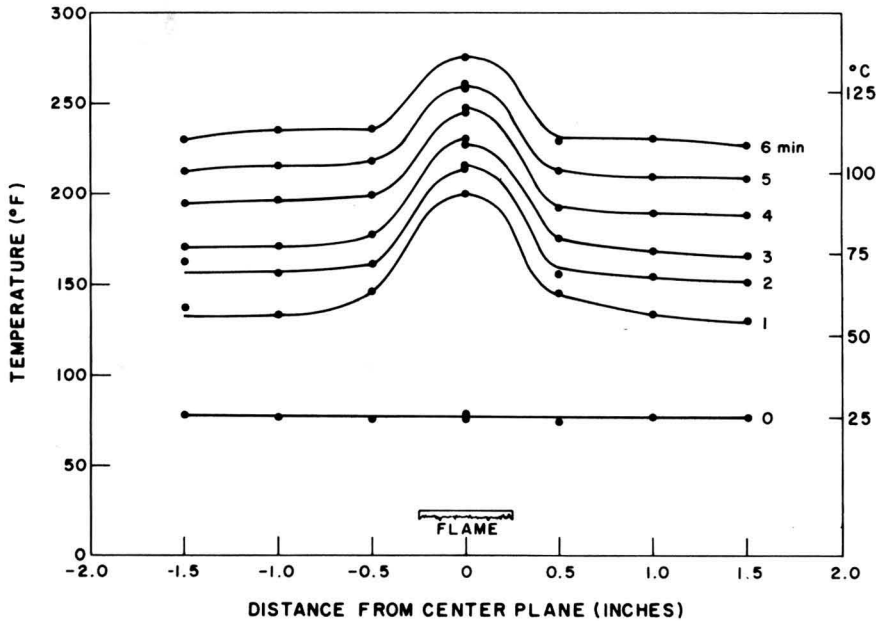


FIG. 7. INSIDE SURFACE TEMPERATURES FOR A 303 X 406 CAN OF 60% SUCROSE SOLUTION ROTATING AT 30 rpm IN A FLAME

Burner gas flow rate 1380 cc/min



fore, a clay suspension was used as the model food in the temperature mapping experiments. Filling conditions were chosen to simulate the most restrictive situation that might be encountered in terms of internal mixing. Thus, the cans were filled completely to eliminate a large headspace bubble that could enhance induced convection. Furthermore, no solid pieces were added to the fluid since limited mapping experiments with particulate foods have shown that food pieces promote mixing, as long as the pack is loose enough to allow the liquid to move through spaces between the pieces.

Thus, the measured temperature profiles show that even under adverse mixing circumstances, sufficient agitation is achieved to give a uniform temperature throughout the can. This is important in terms of achieving bacteriological safety and uniformity of product quality. The addition of normal headspace or food pieces would be expected to enhance this uniformity further.

The can size used in these experiments is typical of the larger sizes (303 × 406; 404 × 502) commonly used in flame processing of fruits and vegetables. Better temperature uniformity would be expected in smaller cans, for example, the 202 × 204 cans used for mushrooms. Even No. 10 sizes (603 × 700) have been shown to heat with good temperature distribution (Leonard *et al.* 1976).

For determining the coefficient of heat transfer to the liquid, it seemed more appropriate to use a typical food liquid and a more normal headspace. Thus, a 60% sucrose solution was used, which simulates a heavy canning syrup.

Taking advantage of the fairly uniform mixing in the interior of the can, let us develop a film model expression for the rate of heat transfer as follows. Divide the can by planes perpendicular to the can axis into  $N$  disc-shaped elements. Since temperature gradients within the can are small except near the flame, we neglect axial heat transfer (including axial transfer through the ends of the can) and assume that the bulk temperature in each element is equal to the centerline temperature  $T_c$ . The rate of heat transfer through the cylindrical surface of the  $n^{\text{th}}$  element is

$$\dot{Q}_n = h_n A_n [(T_{is})_n - T_c] \quad (2)$$

where  $A_n = \pi D \Delta z$  and the total rate to the can is

$$\dot{Q}_T = \sum_{n=1}^N h_n A_n [(T_{is})_n - T_c] \quad (3)$$

Since we were unable to measure individual heat fluxes we defined an average heat transfer coefficient,  $h_i$ , by

$$\dot{Q}_T = h_i \sum_{n=1}^N A_n [(T_{is})_n - T_c] \quad (4)$$

which is equivalent to

$$\dot{Q}_T = h_i A (\langle T_{is} \rangle - T_c) \quad (5)$$

where  $\langle T_{is} \rangle$  is the inside surface temperature averaged over the length of the can.

Experimentally, the surface temperature was measured at seven positions ( $N = 7$ ), and the averaged value plotted as a function of time in Fig. 8. The centerline temperature measured 2.7 cm (1-1/16 in) from the end of the can was also plotted. The heat transfer coefficient was calculated from the straight portions of these curves by combining Eq. (1) and (5) to give

$$h_i = \frac{mC \frac{dT_c}{d\theta}}{A (\langle T_{is} \rangle - T_c)} \quad (6)$$

Based on the 0.463 kg mass, the cylindrical area of the can, 0.0256 m<sup>2</sup> (0.276 ft<sup>2</sup>), and a value of  $C = 2.96$  kJ/kg K (0.708 Btu/lb<sub>m</sub> °F) extrapolated to 60% sucrose from B.F.M.I.R.A. (1967) data,  $h_i$  was found to be  $466 \pm 34$  W/m<sup>2</sup> K ( $82 \pm 6$  Btu/hr ft<sup>2</sup> °F) (see Table 1). The coefficient increased with time as would be expected as the temperature increased and the viscosity of the sucrose solution diminished.

Table 1. Internal film heat transfer coefficient for 60% sucrose solution in 303 X 406 can rotating at 30 rpm

Time min	Temperature, T °C	Temperature, T °F	Viscosity <sup>1</sup> Centipoise	Experimental h		h Calc'd by Eq. (7), W/m <sup>2</sup> K	$\frac{h_{calc}}{h_{expt}}$
				W/m <sup>2</sup> K	Btu/h ft <sup>2</sup> F		
3	58.6	138	10.4	437	77	199	0.454
4	70.6	159	7.0	460	81	212	0.460
5	82.2	180	5.1	477	84	224	0.469
6	98.9	201	3.5	500	88	239	0.478

<sup>1</sup> Handbook of Chemistry and Physics (1955).

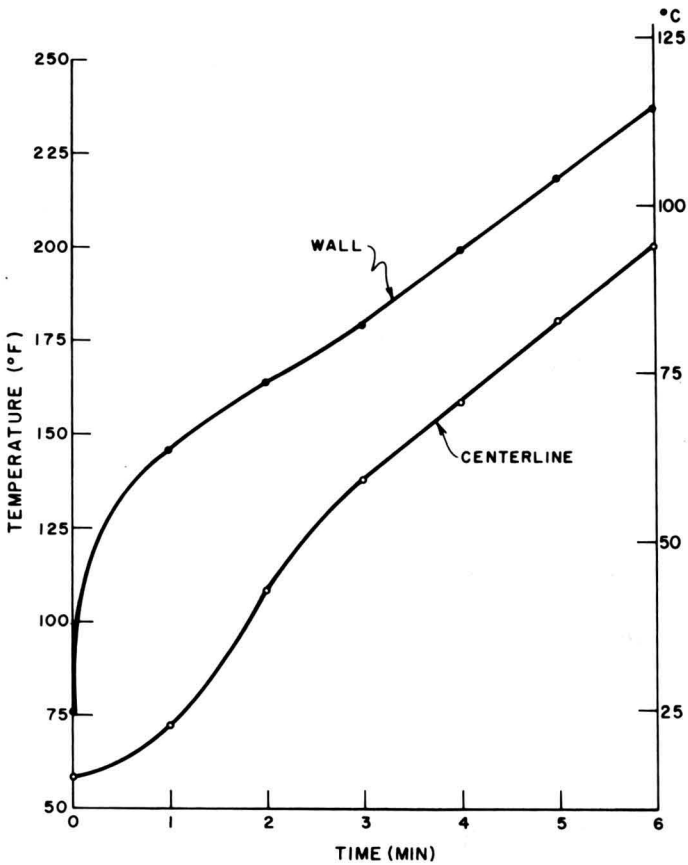


FIG. 8. CENTERLINE TEMPERATURE AND AVERAGE INSIDE SURFACE TEMPERATURE FOR 303 X 406 CAN OF 60% SUCROSE SOLUTION ROTATING AT 30 rpm IN A FLAME

Burner gas flow rate 1380 cc/min.

Quast and Siozawa (1974) have presented data for the overall heat transfer coefficient for cans filled with sucrose solutions rotating in atmospheric steam. Most of their data between 80 and 420 rpm correlated within  $\pm 33\%$  with the Eq. (7) which was obtained by least squares analysis

$$\text{Nu} = 0.17 \text{Re}^{0.52} \text{Pr}^{1/3} (\text{H/E})^{1/3} \quad (7)$$

where  $\text{Nu} = hD/k$  (8)

$$\text{Re} = D_r (\pi D \omega) \rho / \mu \quad (9)$$

$$\text{Pr} = \mu C / k \quad (10)$$

Internal heat transfer coefficients were calculated from Eq. (7) assuming that the can wall and steam side resistances to heat transfer were negligible in the Quast and Siozawa experiments. Numerical values for properties of the 60% sucrose solution were taken as  $k = 0.45 \text{ W/mK}$  (thermal conductivity),  $C = 2960 \text{ J/kgK}$  (heat capacity), and  $\rho = 1287 \text{ kg/m}^3$  (density), all assumed constant, and values of  $\mu$  given in Table 1. Other parameters were  $D_r = 0.0476 \text{ m}$  (diameter of rollers),  $\omega = 0.5 \text{ s}^{-1}$  (rotational speed),  $H = 10.5 \text{ cm}$  (height of can) and  $E = 2.52 \text{ cm}$  (height of headspace).

The calculated coefficients (Table 1) properly correlated the change in viscosity but were uniformly lower than observed experimentally by a factor of 0.47. This may be because the correlation data assume equal coefficients on the ends and body of the can, although heat transfer through the ends is probably lower. The flame heat transfer coefficient assumes all the heat transfers through the sidewall. A better correlation for the present data can be obtained by replacing the coefficient of 0.17 in Eq. (7) by 0.37.

### SUMMARY AND CONCLUSIONS

These experiments with liquid model foods indicate that even under unfavorable processing conditions (i.e., a pure liquid and no initial headspace) the temperature distribution is very uniform within cans of food heated by direct flame. The slowest-heating points in the can were on the axis near the ends of the can; these temperatures lagged behind the temperature at the center of the can by only  $2.8^\circ\text{C}$  ( $5^\circ\text{F}$ ). The temperature distribution is expected to be even more uniform with normal headspace and if the liquid contains food pieces which promote mixing.

Inside surface temperatures were not excessive, not exceeding  $138^\circ\text{C}$  ( $280^\circ\text{F}$ ) in the region near the flame after 6 min of heating when the center temperature was  $93^\circ\text{C}$  ( $200^\circ\text{F}$ ). Thus one would not expect surface burning or damage to can coatings as long as the food material in the can lends itself to adequate mixing.

The temperature profiles indicate a thermal boundary layer near the cylindrical surface of the cans, suggesting that a film model of heat transfer would be applicable. An internal heat transfer coefficient of  $470 \text{ W/m}^2\text{K}$  ( $82 \text{ Btu/h ft}^2 \text{ F}^\circ$ ), based on the entire cylindrical surface of the  $303 \times 406$  can, was measured for 60% sucrose solutions with large head-

space rotating at 30 rpm. The experiments should be extended to liquids containing food pieces; higher heat transfer coefficients would be expected.

#### ACKNOWLEDGMENT

The authors wish to thank R. V. Mehta, B. Patel, and R. Peters for help in making the temperature measurements. Financial support of USDA and Filper Corporation is gratefully acknowledged, as is a fellowship from the Alexander von Humboldt Foundation to R. L. Merson.

#### NOMENCLATURE

$A$	cylindrical area of can
$A_n$	heat transfer area for nth element
$C$	specific heat capacity of can contents
$D$	inside diameter of can
$D_r$	diameter of rollers which turn can
$E$	height of headspace
$h$	film heat transfer coefficient
$h_i$	film heat transfer coefficient inside can based on cylindrical surface area
$h_n$	film heat transfer coefficient for nth element
$H$	inside height of can
$k$	thermal conductivity of liquid in can
$m$	mass of can contents
$n$	index for nth element
$N$	total number of elements
$Nu$	Nusselt number, Eq. (8)
$Pr$	Prandtl number, Eq. (10)
$\dot{Q}$	heat transfer rate
$\dot{Q}_n$	heat transfer rate to nth element
$\dot{Q}_T$	total rate of heat transfer to can
$Re$	rotational Reynolds number, Eq. (9)
$T$	temperature
$T_c$	centerline (bulk) temperature
$T_{is}$	temperature of inside surface of can
$\langle T_{is} \rangle$	temperature of inside surface of can averaged over can length
$\Delta z$	thickness of disc-shaped element of model fluid
$\mu$	viscosity of liquid in can
$\rho$	density of liquid in can
$\theta$	time
$\omega$	rotational speed of can (revolutions/s)

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# EFFECTS OF PROCESSING ON ADSORPTION OF OFF-FLAVORS ONTO SOY PROTEIN<sup>1</sup>

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## ABSTRACT

*Heats of adsorption and adsorption coefficients for the reversible adsorption of several aliphatic alcohols, aldehydes, and ketones on soy protein were measured using gas chromatography. The physical state of protein samples was changed by heating at 100°C and 121°C at moisture contents of 29% and 40%. A comparison was made with values for untreated soy isolate and sheared soy isolate, and the effects of moisture, temperature, and their interaction were examined by analysis of variance. Heats of adsorption were unchanged, but changes in adsorption coefficients of the treated samples demonstrated a significant decrease in binding that can be attributed to protein denaturation. The response depended on the interaction of moisture and temperature effects.*

## INTRODUCTION

To realize the enormous potential of soybeans as a human food source will require processing of the soy to produce a palatable food or food ingredient. Little, however, is known about the effects of processing on

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the interaction of flavor compounds with soy components. In this work, we quantify the effects of thermal processing on the binding of a number of representative flavor compounds to the treated soy protein isolate.

A number of compounds have been associated with soybean off-flavors. These include ketones, aldehydes, alcohols, esters, fatty acids, and amines (Cowan *et al.* 1973; Goosens 1975; Sessa and Rackis 1977; Wolf and Cowan 1977). Interactions of flavors with water-lipid systems (Nelson and Hoff 1968), aqueous sugar solutions (Nawar 1966, 1971), and dilute aqueous solutions (Buttery *et al.* 1969) have been studied by using gas chromatography and headspace analysis.

Some work has been done with proteins. Solms *et al.* (1973) discussed the manner in which volatiles are distributed in food systems containing proteins, carbohydrates and lipids. Gremlı (1974) compared the flavor retention of an aqueous 5% soy protein solution with that of an aqueous buffer solution and concluded that alcohols did not bind, that aldehydes bind both reversibly and irreversibly, and ketones bind reversibly. Franzen and Kinsella (1975) examined the interaction of proteins with other food components, using protein-water, protein-corn oil, protein-endogenous lipid systems, and found that protein reduced headspace concentrations in most instances. Beyeler and Solms (1974) found the extent of binding to protein in aqueous solutions decreased in this order: aldehyde, ketone, alcohol.

The gas chromatographic method used in the present work was used by McMullin *et al.* (1975) to determine heats of adsorption of flavors on lactose. Aspelund (1978) applied the same method, employing soy protein isolate as the column packing. Both studies indicated the involvement of van der Waals forces and hydrogen bonding in the adsorption of volatiles to the lactose and protein substrates.

In this study, adsorption coefficients and heats of adsorption were compared for the adsorption of a number of volatile organics onto soy protein previously subjected to treatments involving different levels of temperature, moisture, and shear.

## MATERIALS AND METHODS

The protein was an isoelectric soy protein isolate (Edi-Pro A, Ralston-Purina Company, St. Louis, Missouri) reported by the company to contain 93.5% protein, 5.5% moisture, and 0.2% fat.

The flavor compounds were n-hexane and 2-pentanone (Matheson, Coleman, and Bell), 2-hexanone, 2-heptanone, n-hexanol, 1-pentanol



(Aldrich Chemical Company), 1-hexanol (Eastman Kodak Company), and 1-heptanol (J. T. Baker Chemical Company).

### Processing Conditions

The major focus was on thermal processing where the four treatments comprised a  $2 \times 2$  factorial design with temperature ( $100^{\circ}\text{C}$ ,  $121^{\circ}\text{C}$ ) and moisture content (29%, 40%) as the variables. A separate treatment incorporated shear. All treatments were repeated twice to provide replicate samples.

Moisture levels were attained by adding sufficient water to the protein to get a homogeneous mixture and then drying to the desired weight in an air circulation drier at  $55^{\circ}\text{C}$  with occasional stirring. The protein was then spread thinly on trays and heated 20 min in a preheated autoclave at the desired temperatures.

Sheared samples were prepared by feeding isolate (33% moisture) to a Wenger X-5 laboratory extruder operated at 700 rpm without a die (and hence, little back pressure). Maximum barrel temperature reached was  $71^{\circ}\text{C}$ .

### Gas Chromatographic Analysis

For use as gas chromatographic column packings, the samples were dried to the initial Edi-Pro A moisture content in an air-circulation drier, ground in a cyclone grinder (UD Corporation), and sized to 230/325 mesh with Tyler sieves. This size was chosen to provide an acceptable balance of surface area and flow rate for the chromatographic studies.

The materials were packed in 3-ft glass columns (2 mm ID), which had been cleaned with 40% NaOH, rinsed with distilled water, then acetone, and dried at  $100^{\circ}\text{C}$ . Approximately 1.4 g of the treated samples and 0.5 g of unprocessed protein could be used while attaining reasonable flow rates at 50 psig head pressure.

Retention times of the flavor compounds were measured at 60, 70, and  $80^{\circ}\text{C}$  on an FID equipped Varian 3700 gas chromatograph with Varian CDS 111 integrator. The compounds injected were drawn from equilibrated reagent headspaces with gas-tight syringes. Where more dilute samples were required, the syringes were flushed several times before injection. Flow rates were measured with a bubble flowmeter at the detector outlet. Column conditions are shown in Table 1.

### Characterization of Packing

Specific surface area measurements were performed for each protein sample by the Micromeritics Instrument Corporation using a modified,

Table 1. Experimental chromatographic conditions for the Varian 3740 gas chromatograph

Injector temperature, °C	150
Detector temperature, °C	150
Column oven temperature, °C	60; 70; 80
Gas flow rates, ml/min	
Nitrogen	23.5-28
Hydrogen	30
Air	300

single-point BET analysis with argon as the adsorbate.

Scanning electron micrographs were taken of selected samples prepared with a 60/40 gold/paladium coating

Disc gel electrophoresis and protein dispersibility index (PDI) (Smith and Circle 1972) measurements were performed on heat-treated, sheared, and unprocessed samples both without pH adjustment and after neutralizing the samples with 40% NaOH.

The uncorrected retention volume,  $V_R'$ , can be found from the measured values of sample retention time,  $t_R$ , unadsorbed sample (methane) retention time,  $t_D$ , and gas flow rate,  $w$ , as

$$V_R' = w(t_R - t_D) \quad (1)$$

Correcting for pressure drop in the column and differences between the column and flowmeter conditions gives the corrected retention volume,  $V_R$ , as

$$V_R = V_R' j \left( \frac{P_f T}{P_o T_f} \right) \quad (2)$$

where  $j = 3 [(P_i/P_o)^2 - 1] / 2 [(P_i/P_o)^3 - 1]$  is the compressibility correction factor of James and Martin (1952). This volume can be normalized on the basis of mass of the mass of column packing,  $m$ , as

$$V_m = \frac{V_R}{m} \quad (3)$$

Assuming that we are working in the linear portion of the adsorption isotherm (very low gas concentrations) the heat of adsorption,  $\Delta H$ , can be calculated (Kiselev and Yashin 1969) from the slope of a plot of  $\ln(V_m/T)$  versus  $1/T$  and the relation

$$\ln \frac{V_m}{T} = \frac{-\Delta H}{RT} + A \quad (4)$$

An adsorption coefficient independent of packing mass or surface area,  $K$ , can be calculated as

$$K = \frac{V_m}{s}$$

By definition,  $K$  is a Henry's type constant relating the adsorbed surface concentration,  $C_s$ , to the gas phase concentration in equilibrium with it,  $C_g$

$$K = C_s/C_g \quad (5)$$

One could also express the results in terms of free energy and entropy changes by defining (Sawyer and Brookman 1968)

$$\Delta G = -RT \ln K \quad (6)$$

and calculating

$$\Delta S = \frac{\Delta H - \Delta G}{T} \quad (7)$$

Both parameters will be dependent on the units of  $K$ . We consider  $K$  and  $\Delta H$  the more fundamental parameters.

## RESULTS

Representative output peaks are shown in Fig. 1. A heterogeneous absorbent surface, as might be expected for a protein, will tend to cause peak tailing (Kiselev and Yashin 1969). This tailing is minimized by reducing the amount of sample injected but cannot be eliminated for alcohols.

Table 2 gives the physicochemical characteristics of the various samples. Table 3 gives the average adsorption coefficients at 70°C for each treatment while Table 4 gives the heats of adsorption for all treatments and flavor compounds. Adsorption coefficients at 60° and 80°C (not shown) differ in magnitude but qualitatively show the same effects of processing and, hence, serve only to confirm the reported results. The complete data are available (Crowther 1979). The results for hexane do

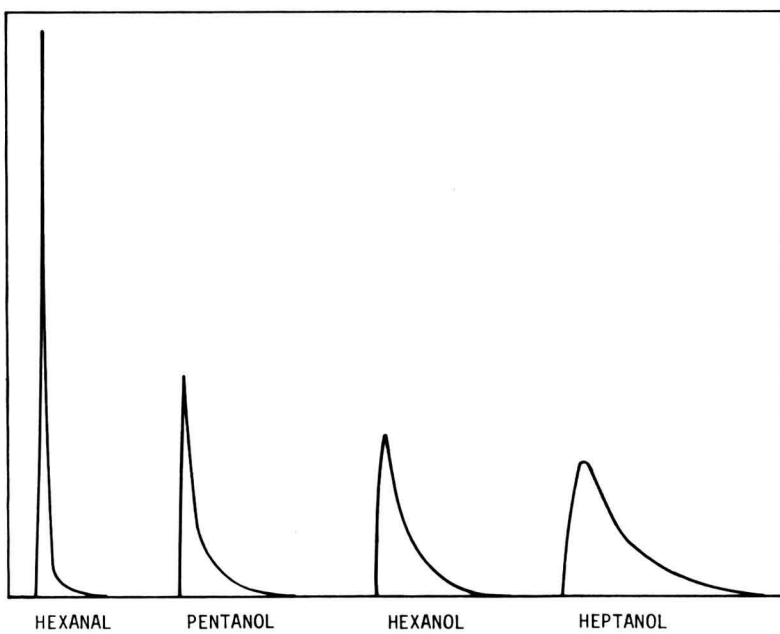
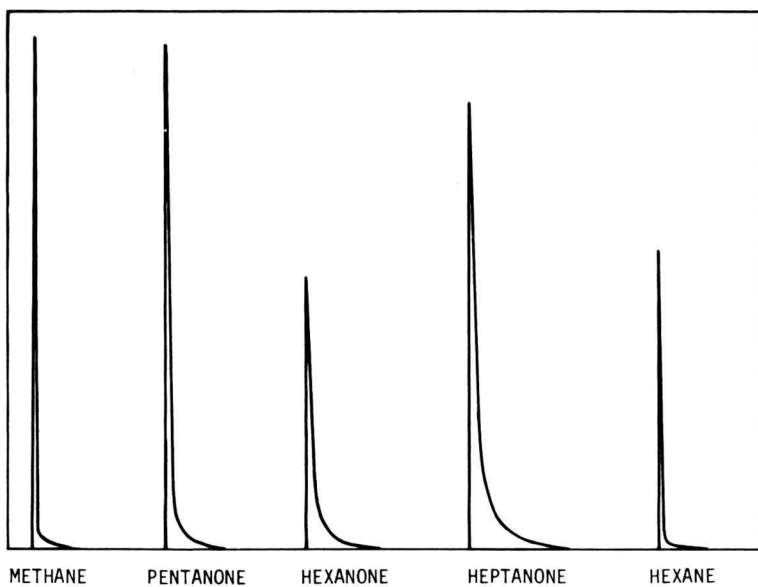


FIG. 1. REPRESENTATIVE PEAKS COLLECTED FROM A  
121°C, 29% H<sub>2</sub>O SAMPLE AT 70°C

The order of the peaks has no significance.

Table 2. Physicochemical properties of the protein samples used as column packings. Temperature and moisture refer to the condition of the sample when autoclaved, not while in the column

Treatment	Specific Surface Area <sup>1</sup> (m <sup>2</sup> /g)	PDI <sup>2</sup>		Electrophoresis <sup>3</sup>
		Isoelectric	Neutralized	
Autoclaved		1.9	7.8	No bands
121°C, 29% H <sub>2</sub> O	0.17, 0.18			
121°C, 40% H <sub>2</sub> O	0.20, 0.24			
100°C, 29% H <sub>2</sub> O	0.23, 0.19			
100°C, 40% H <sub>2</sub> O	0.20, 0.28 <sup>4</sup>			
Untreated	0.20	1.3	88.4	Several strong bands
Sheared	0.28	1.6	48.7	Bands less distinct than Edi-Pro A and some missing

<sup>1</sup> Single-point BET ± 3% accuracy according to Micromeritics Instrument Corporation. The two values for each autoclaved condition are for the two independently prepared samples

<sup>2</sup> Average of two determinations

<sup>3</sup> Neutralized samples

<sup>4</sup> This sample was done with a multiple-point BET analysis (± 1% accuracy) using krypton as the adsorbate

not appear because hexane was not significantly retained. Tables 3 and 4 contain the absolute K and ΔH values, but our discussion will center on the tables which follow. The latter are derived from the results presented in 3 and 4 but compare differences between compounds and treatments.

Figures 2 and 3 illustrate the effect of column temperature on adsorption for one packing and are representative of the plots from which ΔH values were calculated.

### Statistical Analysis

Analysis of variance (using a Statistical Analysis System [SAS] package) for the factorial design showed the moisture-temperature interaction to have the greatest effect on the adsorption coefficient, obscuring the main effects (Table 5). Therefore, the significance of main effects (temperature and moisture) was assessed by using the Student t-test. These results appear in Table 6. This was not done for heats of adsorption since the data clearly indicated that there was no significant change in this parameter.

The major effects of autoclave conditions (Table 6) are seen at 40% moisture, where an increase in temperature results in an increase in K (56–59% for ketones, 64–81% for alcohols, and 64% for hexanal), and at 100°C, where an increase in moisture results in a decrease in K (33–47%). In comparing untreated Edi-Pro A with the autoclaved and sheared

Table 3. Adsorption coefficients for the adsorption at 70°C of various compounds onto Edi-Pro A subjected to three types of treatment

Column Packing Treatments	Adsorption Coefficient ( $\pm$ Standard Error of the Mean)						
	Pentanone	Hexanone	Heptanone	Pentanol	Hexanol	Heptanol	Hexanal
Autoclaved							
121°C, 29% H <sub>2</sub> O <sup>1</sup>	0.822 $\pm 0.090$	1.89 $\pm 0.06$	3.49 $\pm 0.03$	6.66 $\pm 0.80$	13.5 $\pm 1.3$	30.4 $\pm 3.2$	1.54 $\pm 0.11$
121°C, 40% H <sub>2</sub> O <sup>2</sup>	0.879 $\pm 0.062$	1.91 $\pm 0.22$	3.94 $\pm 0.64$	9.04 $\pm 0.71$	17.0 $\pm 2.1$	38.4 $\pm 4.2$	1.79 $\pm 0.27$
100°C, 29% H <sub>2</sub> O <sup>1</sup>	1.046 $\pm 0.023$	2.13 $\pm 0.20$	3.76 $\pm 0.50$	8.39 $\pm 1.65$	16.3 $\pm 2.8$	35.3 $\pm 5.1$	1.90 $\pm 0.30$
100°C, 40% H <sub>2</sub> O <sup>1</sup>	0.556 $\pm 0.023$	1.20 $\pm 0.04$	2.52 $\pm 0.50$	5.51 $\pm 2.10$	9.6 $\pm 2.9$	21.2 $\pm 7.4$	1.09 $\pm 0.02$
Mean	0.825 $\pm 0.102$	1.78 $\pm 0.201$	3.43 $\pm 0.32$	7.40 $\pm 0.81$	14.1 $\pm 1.7$	31.3 $\pm 3.8$	1.58 $\pm 0.18$
Untreated <sup>3</sup>	1.56 $\pm 0.06$	3.48 $\pm 0.01$	6.73 $\pm 0.53$	11.3 $\pm 0.15$	21.7 $\pm 1.2$	52.1 $\pm 1.5$	2.77 $\pm 0.45$
Sheared <sup>3</sup>	1.63 $\pm 0.05$	3.24 $\pm 0.11$	6.50 $\pm 0.26$	16.5 $\pm 0.87$	29.7 $\pm 2.3$	59.4 $\pm 3.2$	3.02 $\pm 0.07$

<sup>1</sup> Average of independently autoclaved replicates<sup>2</sup> Average of four entries. Two independent replicates each run in two columns<sup>3</sup> Average of two columns packed with material from the same batch

Table 4. Heats of adsorption for various compounds onto Edi-Pro A subjected to three types of treatment. Except as noted for heptanol, the  $\Delta H$  values are based on data at 60, 70 and 80°C

Column Packing Treatments	Heat of Adsorption ( $\pm$ Standard Error of the Mean)						
	Pentanone	Hexanone	Heptanone	Pentanol	Hexanol	Heptanol <sup>1</sup>	Hexanol
Autoclaved							
121°C, 29% H <sub>2</sub> O <sup>2</sup>	-6.59 $\pm 1.67$	-9.31 $\pm 0.30$	-9.86 $\pm 0.08$	-14.7 $\pm 0.65$	-16.6 $\pm 0.01$	-16.2 $\pm 1.78$	-7.92 $\pm 1.00$
121°C, 40% H <sub>2</sub> O <sup>3</sup>	-6.55 $\pm 1.15$	-9.20 $\pm 0.79$	-10.9 $\pm 0.14$	-15.1 $\pm 0.16$	-16.9 $\pm 0.17$	-17.6 $\pm 0.99$	-9.36 $\pm 1.41$
100°C, 29% H <sub>2</sub> O <sup>2</sup>	-6.26 $\pm 1.10$	-8.13 $\pm 1.14$	-10.9 $\pm 0.11$	-16.0 $\pm 1.75$	-17.1 $\pm 0.28$	-17.3 $\pm 0.06$	-8.74 $\pm 0.01$
100°C, 40% H <sub>2</sub> O <sup>2</sup>	-10.0 $\pm 0.99$	-10.7 $\pm 3.20$	-12.7 $\pm 1.08$	-13.9 $\pm 1.02$	-16.4 $\pm 0.29$	-17.8 $\pm 0.18$	-11.4 $\pm 0.88$
Mean	-7.35 $\pm 0.89$	-9.34 $\pm 0.53$	-11.2 $\pm 0.6$	-14.9 $\pm 0.4$	-16.8 $\pm 0.2$	-17.2 $\pm 0.4$	-9.36 $\pm 0.74$
Untreated <sup>4</sup>	-10.1	-11.0	-12.7	-13.1	-16.7	-16.8	-11.1
Sheared <sup>4</sup>	-9.11	-10.5	-12.3	-14.2	-15.0	-16.3	-10.8

<sup>1</sup> In three cases, heptanol was not detected at 60°C, and only the 70° and 80°C data were used

<sup>2</sup> Average of two independent replicates

<sup>3</sup> Average of four entries. Two pairs of columns were packed with two independent replicates

<sup>4</sup> Average of two columns with the same packing

Table 5. Analysis of variance for the  $2 \times 2$  factorial design for effects of temperature and moisture on adsorption coefficients (at 70°C) and heats of adsorption (from data at 60, 70, and 80°C). The values are the probability that the independent variable did not affect the dependent variable

Dependent Variable	Independent Variable	Probability						
		Pentanone	Hexanone	Heptanone	Pentanol	Hexanol	Heptanol	Hexanal
K	Moisture	0.107	0.125	0.781	0.819	0.880	0.974	0.509
	Temperature	0.493	0.237	0.280	0.393	0.290	0.220	0.418
	Moisture/ Temperature	0.025	0.047	0.177	0.133	0.102	0.103	0.082
	Interaction	0.414	0.588	0.082	0.556	0.586	0.428	0.197
$\Delta H$	Temperature	0.325	0.929	0.076	0.968	0.901	0.713	0.338
	Moisture/ Temperature	0.218	0.466	0.174	0.285	0.081	0.569	0.368
	Interaction							



Table 6. Comparison of adsorption coefficients for various process variables. The values presented are differences between adsorption coefficients reported in Table 3.

Comparison	$\Delta K$ at 70°						
	Pentanone	Hexanone	Heptanone	Pentanol	Hexanol	Heptanol	Hexanal
Autoclaved <sup>1</sup>							
29% H <sub>2</sub> O	-0.224***	-0.24	-0.27	-1.73	-2.8	-4.9	-0.36
40% H <sub>2</sub> O	0.323***	0.71**	1.42****	3.53****	7.4***	17.2***	0.7***
100°C	-0.49**	-0.93**	-1.24***	-2.88	-6.7***	-14.1***	-0.81***
121°C	0.057	0.02	0.45	2.38	3.5	8.0	0.25
Untreated vs. Sheared <sup>2</sup>	0.07	-0.24****	-0.23	5.2**	8.0**	7.3***	0.25
Untreated vs. Autoclaved <sup>3</sup>	-0.735**	-1.7*	-3.3*	-3.9***	-7.6***	-20.8**	-1.19***

\*P (probability of no significant difference) < 0.01; \*\* P < 0.05; \*\*\* P < 0.1; \*\*\*\* P < 0.2  
<sup>1</sup> (K<sub>121</sub> - K<sub>100</sub>) at constant % H<sub>2</sub>O or (K<sub>40</sub> % - K<sub>29</sub> %) at constant T; df = 4 (using an estimate of the standard deviation by pooling the estimates at the four conditions)

<sup>2</sup> (K<sub>sheared</sub> - K<sub>untreated</sub>); df = 2

<sup>3</sup> (K̄ - K<sub>untreated</sub>) where K̄ is the mean of the four autoclaved samples; df = 4

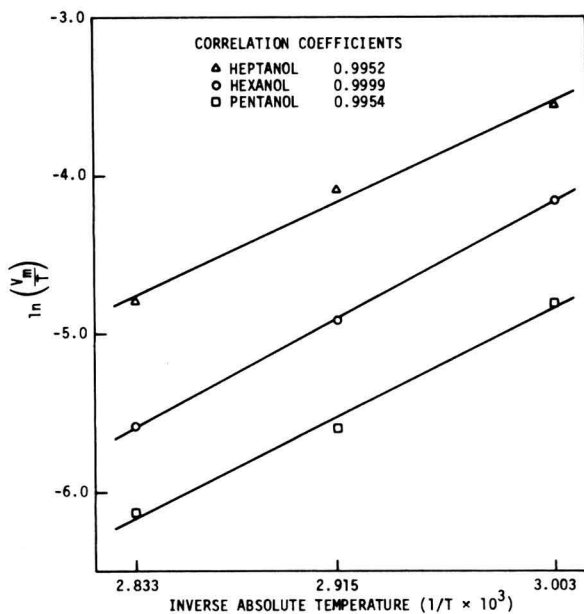


FIG. 2. LINEAR REGRESSION LINES USED TO CALCULATE  $\Delta H$  FOR ALCOHOLS RUN ON ONE OF THE 121°C, 29% H<sub>2</sub>O SAMPLES

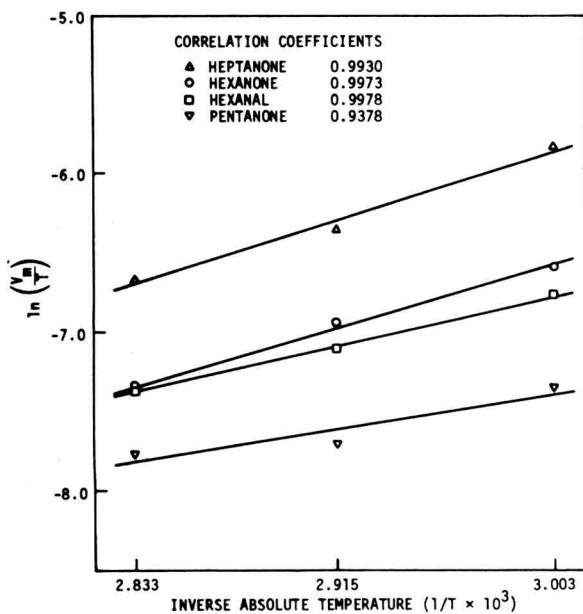


FIG. 3. LINEAR REGRESSION LINES USED TO CALCULATE  $\Delta H$  FOR CARBONYL COMPOUNDS RUN ON ONE OF THE 121°C, 29% H<sub>2</sub>O PACKINGS

samples (Table 6), autoclaving greatly reduces adsorption (47–49% for ketones, 35–40% for alcohols, and 43% for hexanal) while shear has a significant effect only on the alcohols where values are increased from 14–46%.

For the last two comparisons, the values for the four moist-heat treatments were averaged. The lowest K values occurred at 121°C, 29% H<sub>2</sub>O (see Table 3 for K values).

## DISCUSSION

### Heats of Adsorption

The values for heats of adsorption ( $\Delta H$ , Table 4) obtained in this study seem to be reasonable. That is, all the values are negative and increase in magnitude as chain lengths increase within an homologous series. Table 7 compares  $\Delta H$  values with those obtained by Aspelund (1978), with Edi-Pro A, and McMullin *et al.* (1975), with lactose. Values calculated in our work are generally higher (i.e. more negative) than Aspelund's and lower than those of McMullin *et al.*

Table 7. Comparison of heats of adsorption on three solid substrates. The present values are averages for all treatments of Edi-Pro A.

Flavor	$-\Delta H$ (kcal/mole) <sup>1</sup>		
	Aspelund, 1978 Edi-Pro A	Current Study Heat-Treated Edi-Pro A	McMullin et al., 1975 Lactose
Pentanone	2.92	8.10	10.81
Hexanone	6.04	9.81	12.03
Heptanone	8.11	11.56	13.09
Hexanal	8.89	9.90	11.66
Pentanol	11.25	14.50	15.70
Hexanol	13.89	16.45	17.57
Heptanol	18.06	17.80	17.18

<sup>1</sup> All values for each work were averaged

The energy difference was 6.2–6.6 kcal/mole between the alcohols and corresponding ketones and aldehyde. These observations can be used to determine the nature of the binding. McMullin *et al.* found that the heat of adsorption of n-heptane was 6.38 kcal/mole. This may be used as a measure of nonspecific (VanderWaals) interaction energy. Since our calculated  $\Delta H$ 's are somewhat less than those of McMullin *et al.*, it may be presumed that  $\Delta H$  for heptane in our system would be the same or slightly less than 6.38 kcal/mole. This value also agrees with

Aspelund's value of 6.48. The resulting difference in  $\Delta H$  between heptane and heptanone and heptanone and heptanol is then 5–6 kcal/mole (McMullin *et al.* 1975), which makes it reasonable to assume that adsorption involves nonspecific interaction plus one hydrogen bond for carbonyls or two hydrogen bonds for alcohols. This conclusion necessitates that the adsorbent (soy protein) have numerous polar binding sites that would facilitate formation of two hydrogen bonds.

#### Flavor Binding and Molecular Structure

Less information is available on amounts of flavor binding. Arai *et al.* (1970) studied flavor retention by mixing n-hexanal and n-hexanol with native, partly denatured, and denatured soy protein. The mixtures were subjected to a vacuum and in each case more n-hexanal than n-hexanol was retained. Native soy protein retained the least amounts of these flavors while denatured protein retained the most with partly denatured protein retaining an intermediate amount. Our work shows lower adsorption coefficients for hexanal on heated protein indicating less retention. However, the type of binding measured by Arai *et al.* probably was chemical (irreversible) bonding. In this study, the only molecules detected were those that traversed the column and thus were only physically (reversibly) adsorbed. Hexanal is likely to be more reactive than hexanol (Arya and Parihar 1977) while hexanol is adsorbed more strongly than hexanal. This would explain why more hexanal relative to hexanol was retained in the study by Arai *et al.* while the opposite was true in this study. Gremlı (1974) also found aldehydes to adsorb irreversibly.

The adsorption coefficient,  $K$ , reflects both the strength of adsorption, indicated by  $\Delta H$ , and the number of binding sites. Consistent with this, the measured values of  $K$  and  $\Delta H$  are found to increase with chain length (increased VanderWaals interaction). Likewise, alcohols show higher  $\Delta H$ 's and  $K$ 's than do carbonyl compounds. Since  $\Delta H$  values were unaffected by processing, the observed changes in  $K$  are postulated to be the result of changes in the number of binding sites.

The comparative extents of binding (as indicated by  $K$ ) in this dry system are predominantly the reverse of the order found in aqueous solutions of soy proteins (Beyeler and Solms 1974; Gremlı 1974). Here, alcohols clearly had the highest  $K$ 's while the carbonyls were quite close (compare hexanol, hexanone, and hexanal in Table 3).

Scanning electron microscopy, electrophoresis, and PDI measurements were performed to look for further indication of molecular rearrangements that might be responsible for changes in the number of binding sites. There is no evident visual difference between the sheared and heat-treated protein (see Fig. 4). However, both electrophoresis and PDI

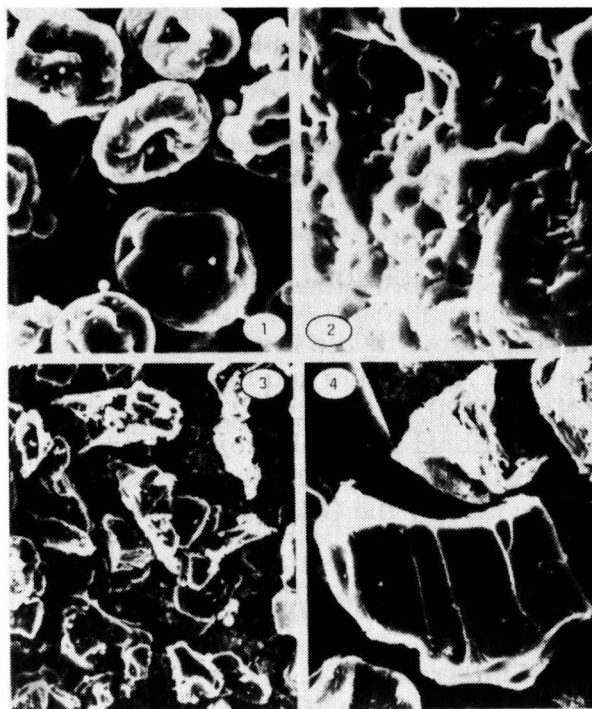


FIG. 4. SCANNING ELECTRON MICROGRAPHS OF COLUMN PACKINGS

1. Unprocessed Edi-Pro A (930x), 2. Heat-treated Edi-Pro A before grinding (225x), 3. Sheared Edi-Pro A (290x), 4. Heat-treated Edi-Pro A (700x).

results changed most drastically for the moist-heat treated samples, as would be expected. This, in turn, coincides with the reduction in adsorption coefficients for the heat-treated samples.

There are at least two types of denaturation that may have occurred. Solms *et al.* (1973) describe the denaturation in the study by Arai *et al.* (1970) as an unfolding type reaction. Burgess and Stanley (1976) indicate that a heat aggregation also is possible in which intermolecular peptide bonds are formed.

The present work is consistent with a picture of a chain unfolding of the globular proteins, which exposes the more nonpolar regions of the protein, decreasing both solubility and the availability of polar hydrogen bonding sites for flavor compounds. Those treatments that reduce solubility must also cause the greatest decrease in binding sites as indicated by the decrease in adsorption coefficient. The changes seem to

involve an interaction between moisture and temperature levels during processing. This suggests that we ought to consider water activity as the important process variable. However, such measurements were not made.

The net effect of processing on the flavor of a food will depend, of course, on the interaction of the nonprotein food components as well.

#### ACKNOWLEDGMENT

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#### NOMENCLATURE

A	Constant of integration, Eq. (4), $A = \ln \frac{V_m}{T}$ at $T = \infty$
$C_g$	Gas phase concentration, gmole/ml
$C_s$	Surface concentration, gmole/m <sup>2</sup>
$\Delta G$	Change in free energy defined by Eq. (6)
$\Delta H$	Heat of adsorption, kcal/gmole
j	Compressibility correction factor, dimensionless
K	Adsorption coefficient, ml/m <sup>2</sup>
m	Mass of column packing, g
$P_i$	Pressure at column inlet, psia
$P_t$	Pressure at flow meter, psia
$P_o$	Pressure at column outlet, psia
R	Gas constant
s	Specific surface area of packing, m <sup>2</sup> /g
$\Delta S$	Change in entropy defined by Eq. (7)
$t_D$	Retention time of unadsorbed gas, s
$t_R$	Retention time of sample, s
T	Column temperature, K
$T_f$	Flow meter temperature, K
$V_m$	Normalized retention volume, Eq. (3), ml/g
$V_R$	Corrected retention volume, Eq. (2), ml
w	Gas flow rate, ml/s

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# EFFECT OF DEFORMATION RATE AND MOISTURE CONTENT ON RICE GRAIN STIFFNESS<sup>1</sup>

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## ABSTRACT

*Grains of brown rice, paddy variety IR-8, were subjected to two types of uniaxial compressive loading, constant displacement rate and sinusoidally varying stress, to determine behavior over a wide range of time scale. Grains from four different moisture level groups were used.*

*With a time decrease from 120 to  $1.6 \times 10^{-4}$  s the relaxation modulus increased 15, 25, 16 and 20 fold at respective moisture contents of 12, 17, 22 and 27%. A master relaxation curve for each moisture content is presented. Understanding the viscoelastic behavior of brown rice as a function at time and moisture content is basic to the solution of stress-strain analysis problems.*

## INTRODUCTION

Rice is consumed mostly in the form of whole unbroken grains. Rice breakage which occurs during harvesting, drying, milling and storage frequently results from applied mechanical forces. By varying the force or deformation rate over a range from relatively low values representing storage conditions to the extremely high values experienced under impact conditions an assessment of load-deformation characteristics can be made.

The effect of loading rate on the uniaxial compression modulus of grains was investigated by Shpolyanskaya (1952) for wheat, by Zoerb

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and Hall (1960) for beans, maize and wheat, and at high deformation rates by Arnold (1969). Wen and Mohsenin (1970) determined complex tensile moduli for corn horny endosperm at low frequencies. Chattopadhyay *et al.* (1978) reported high sinusoidal frequency (100 to 1000 Hz) modulus values for brown rice. In these cited investigations the range of strain rates was not great enough to present a modulus master curve over several decades of time.

The purpose of this paper is to present data over a sufficient number of time decades to adequately demonstrate the modulus master curve necessary for solution of time dependent stress-strain analysis problems for rice kernels.

### PROCEDURE

Paddy rice of the IR-8 cultivar was hand shelled to obtain brown rice kernels. A specially designed kernel holder was used to shape each kernel into a cylindrical specimen as described by Chattopadhyay *et al.* (1978). The length of each specimen was measured with a micrometer. Cross-section dimensions were determined by measuring projection magnifications of microscope views. Specimens were held in glass jars containing predetermined sulfuric acid concentrations for 25 days to condition groups to 12, 17, 22 or 29% moisture content, dry basis.

Selection of deformation or forcing rates was critical to the research but could only be done within limits dictated by the equipment. Constant deformation rates were  $2.12 \times 10^{-6}$ ,  $8.47 \times 10^{-6}$  and  $4.23 \times 10^{-5}$  m/s corresponding to the relaxation time range of 0.6 to 120 s. Sinusoidal forcing was done at 100 Hz increments from 100 to 1000 Hz corresponding to relaxation times of  $1.6 \times 10^{-3}$  to  $1.6 \times 10^{-4}$  s.

The constant deformation rate tests were done on a Model TM Instron Universal Testing Machine equipped with an environmental chamber. Temperature inside the chamber was held at 25°C and various relative humidities corresponding to prescribed rice kernel equilibrium moisture contents were maintained. Three specimens from each moisture content group were tested at each deformation rate. The technique described by Hammerle and Mohsenin (1970) was used to analyze results. For each deformation rate five strain values of the specimen were divided by the strain rate to obtain a series of time intervals. At each time the moduli of the replications were averaged. Details are given in Chattopadhyay 1973.

Five specimens were dynamically tested at each test condition. The specimen was placed between two rigid flat surfaces and subjected to a sinusoidally varying force. Force and resulting displacement were read

from an oscilloscope equipped with Polaroid camera for making a permanent record of each screen trace. Procedural details and experimental results have previously been reported by Chattopadhyay *et al.* 1978.

A necessary assumption made when analyzing the data was that the rice grain responded to the forcing as a linearly viscoelastic material. Specifically, the compressive static and dynamic moduli were not dependent on the magnitude of stress or strain applied. This was necessary for dynamic tests since in order to hold the rice grains in place, they were preloaded statically and the static stress and resulting strain were not sensed by the dynamic transducers. Numerically the total force on a specimen was always kept less than 15% of the minimum force causing failure. In the case of the constant displacement rate tests, results are transformed to what are equivalent to relaxation moduli at different strain levels so the assumption is also necessary.

## RESULTS

The mean values of the compressive relaxation moduli for rice grain at various moisture levels obtained from the two types of experiments are shown in Fig. 1. Coefficients of variation for the data used to calculate the means ranged from 4 to 10% for the constant displacement rate tests. For the dynamic tests the coefficients of variation were higher, especially at the 27% mc. Specific ranges are: 12% mc (2–11%), 17% mc (10–15%), 22% mc (5–17%), and 27% mc (15–30%). Quadratic equations representing the data at each moisture content are given in Table 1 and shown as lines in Fig. 1. All equation terms were significant, the quadratic term for 17% mc at the 5% level and all others at the 1% level.

Table 1. Equations relating modulus values and relaxation times for each moisture content

<u>12% mc</u>
$\text{Log } E(t) = 9.031 - 0.1941 \log t + 0.00432 (\log t)^2$
<u>17% mc</u>
$\text{Log } E(t) = 8.725 - 0.1914 \log t + 0.01721 (\log t)^2$
<u>22% mc</u>
$\text{Log } E(t) = 8.528 - 0.1546 \log t + 0.02813 (\log t)^2$
<u>29% mc</u>
$\text{Log } E(t) = 8.331 - 0.1817 \log t + 0.01487 (\log t)^2$

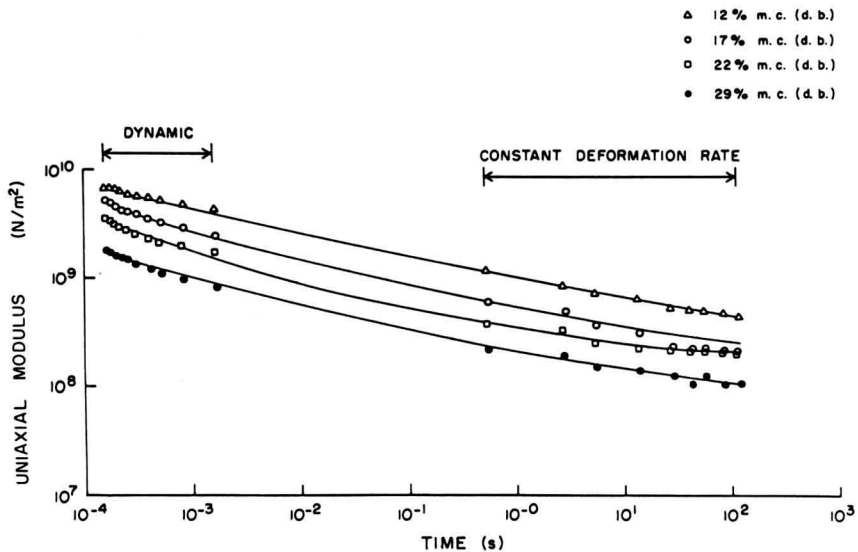


FIG. 1. MEAN VALUES OF RELAXATION MODULI AS DEPENDENT ON TIME AND MOISTURE CONTENT

Moisture content effects and modulus values are in general agreement with data for other cultivars at a single strain rate (e.g., Hoki 1979). Examination of Fig. 1 reveals that the modulus increased respectively, by factors of 15, 25, 16 and 20 for 12, 17, 22 and 29 (d.b.) percent moisture contents with a time decrease from 120 to  $1.6 \times 10^{-4}$  s.

Interpolating the range covering  $1.6 \times 10^{-3}$  to 0.6 s where data are not available, fairly uniform curves having typical shapes for linear viscoelastic materials are achieved thus confirming again the viscoelastic nature of the rice grain specimens. The spacing between lines for the different moisture contents shows the large influence of moisture content on the properties of this high starch grain.

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# HIGH-TEMPERATURE DRYING-CUM-PARBOILING OF PADDY

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## ABSTRACT

*Alternate methods of parboiling of paddy, in place of conventional steam-parboiling, for obtaining improved hydration ability in the parboiled rice were explored. Ratnachudi variety, a medium-grained, tall indica, high-amylose rice, was used. The paddy was soaked in warm water to a saturation moisture level (ca 30%, w.b.). The soaked paddy was dried by either exposing it to very hot air (150°–200°C) or by roasting it in hot sand (250°–300°C) for a few minutes. It was observed that under appropriate high-temperature conditions paddy got simultaneously dried and parboiled by both the above treatments. Parboiled rice so produced was examined for appearance (translucence, color), milling breakage, alkali degradation pattern and starch retrogradation. It was found to be quite comparable to normal steam-parboiled rice in all respects, except that it showed the desirable property of improved hydration ability due to lowering of starch retrogradation. Possible development of the technique into commercial process is indicated.*

## INTRODUCTION

Parboiled rice is prepared by soaking paddy in water until it is saturated, steaming to gelatinize the starch, then drying it and milling. Flaked rice is prepared in India by a somewhat similar process. Here, the soaked paddy, instead of being steamed, is rapidly roasted in hot sand and then flaked by passing it repeatedly under an edge-runner. During passage under the edge-runner, the husk and the bran, which have been rendered dry and brittle by the roasting, get pulverized and removed and the endosperm gets flattened. Flaked rice is used extensively for making snack and quick-cooking foods in south Asia.

In a study of the comparative properties of the two products in this laboratory, two points were established (Ali and Bhattacharya 1976a, 1976b). First, the high-temperature roasting step not only dried but

also gelatinized the rice. In fact, when the roasted paddy was not flaked but finish-dried in air and milled, it yielded rice that was typically parboiled both in appearance and in properties. Second, although superficially similar, the two processes yielded rice with certain markedly dissimilar properties. While conventional steam-parboiled rice cooked slower than ordinary white rice, roasting-parboiled (or flaked) rice had better cooking (i.e. hydration) quality. This difference was due to the fact that the gelatinized starch got partially reassociated, or retrograded, in the moist conventionally parboiled rice, while such retrogradation was hindered in roasting-parboiled rice due to the simultaneous rapid dehydration during gelatinization by roasting.

It was envisaged on the basis of these studies that it might be possible to develop a new process of parboiling of rice based on the principle of rapid drying of soaked (or otherwise wet) paddy, using either hot sand or hot air. Indeed a somewhat similar process of parboiling, called the 'Sella' process, has been long prevalent as a small-scale process in the hill districts of Uttar Pradesh and Punjab states of India, and another one called the 'Chatti' process in the Sind province of Pakistan. In these, 2–3 kg batches of soaked paddy are manually roasted in an iron pan with or without sand over a fire. But no scientific study of these processes is available. Certain other studies too had shown earlier that when high-moisture harvest paddy was dried either by conduction heating using sand (Iengar *et al.* 1971, Khan *et al.* 1974) or with very hot air (Ban 1971), it seemed to partially parboil the rice. Iengar *et al.* (1971) went further and suggested hot-sand roasting of soaked paddy as a possible method of parboiling; but no detailed study of the process or of the product quality was carried out.

Exploratory studies on the process of drying-cum-parboiling of soaked paddy was therefore proposed. The feasibility of parboiling by sand roasting was demonstrated with a very small laboratory set up in the earlier work (Ali and Bhattacharya 1976b). Further studies on the process using (1) hot sand in somewhat larger scale and (2) hot air are reported below.

## MATERIALS AND METHODS

Ratnachudi paddy (a medium-grained, high-amylose variety), procured from the local market, was used. It was prepared for the drying-parboiling studies by soaking in warm water overnight (Bhattacharya and Indudhara Swamy 1967), which gave a saturation moisture content of about 30% (wet basis).



## Hot-air Drying and Parboiling

A very small laboratory set up was used (Fig. 1). It consisted of a Wolf hand air-blower connected through a 1 kw electrical heater to a cylindrical upright glass drying chamber (73 mm i.d.  $\times$  170 mm high) partially closed at the top. Both the blower and the heater were connected to a variac each to regulate the volume and temperature of drying air. A wire-mesh horizontal platform placed about 20 mm from the bottom in the drying cylinder served to hold the paddy to be dried. After preheating the set up by passing hot air under the desired conditions for 10–15 min, 50 g of soaked paddy (giving a bed thickness of 15–18 mm) was placed in the cylinder and dried for the desired period.

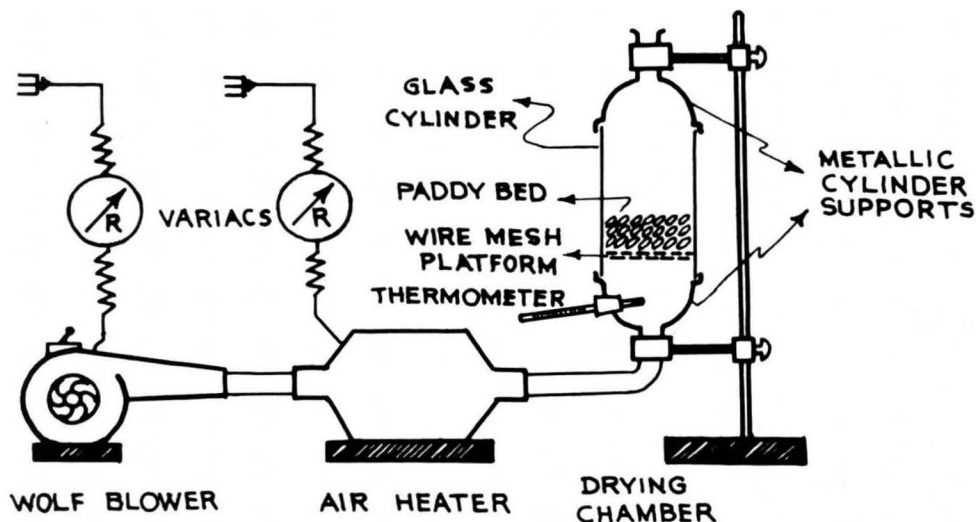


FIG. 1. SCHEMATIC DIAGRAM OF LABORATORY-SCALE SET UP FOR HOT-AIR DRYING-CUM-PARBOILING OF PADDY

## Laboratory-Scale Sand Roasting

The equipment and the process were similar to those used earlier (Ali and Bhattacharya 1976b), but a somewhat larger (holding capacity about 5 kg) and power-driven unit was used (Fig. 2). It was an electrically heated, cylindrical, rotary (18 rpm) batch roaster that could be vertically tilted, and was provided with appropriate mixing baffles and a thermostatic control. The temperature was noted from a thermometer inserted through the hopper-shaped open inlet-cum-discharge end. The roaster, along with 1200 g sand ( $-22+60$  mesh, washed and dried), was put into motion and heated at the desired initial temperature for a few minutes.

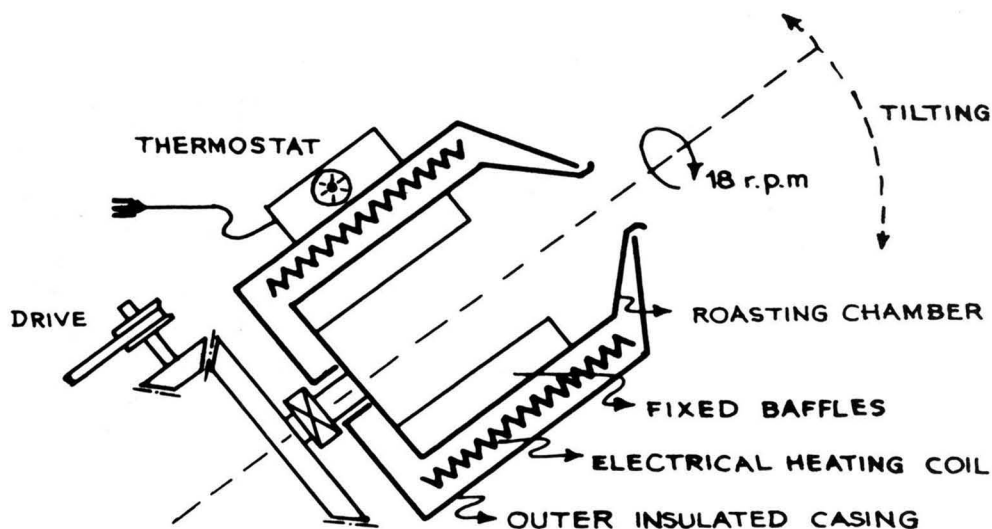


FIG. 2. SCHEMATIC DIAGRAM OF LABORATORY-SCALE ROASTER

The inner roasting chamber rotates (18 rpm), but the outer jacketed casing remains stationary. The whole equipment can be tilted by over  $90^\circ$  for charging, roasting and discharging.

Soaked paddy (600 g) was quickly added and the mixture was heated for the desired period, after which the paddy-sand mixture was discharged on to a sieve to remove the sand.

The actual temperature attained by the mixture was much lower than the initial sand temperature. For instance, for an initial sand temperature of  $250^\circ\text{C}$ , the mixing temperature dropped to  $120^\circ\text{C}$  after 1.5 min of adding soaked paddy and then gradually rose to  $145^\circ\text{C}$  at 5 min. Since this temperature was variable, only the initial sand temperature has been reported in the following description. The paddy after roasting attained a temperature of approximately  $15\text{--}20^\circ$  less than the corresponding mixture temperature. For an initial sand temperature of  $250^\circ$  and  $300^\circ\text{C}$ , respectively, the final paddy temperature lay in the range of about  $90^\circ\text{--}130^\circ\text{C}$  for a roasting period of 1.75–4.25 min and 0.5–2.75 min, respectively.

#### Large-Scale Roasting

Large-scale roasting trials were conducted using a high-capacity (500–700 kg/h) continuous 'gram roaster' (manufactured by Shanmugham Pillai and Sons, Dindigal, Tamil Nadu), normally used for commercial puffing of bengal gram (chick pea). As shown schematically in Fig. 3, it consisted of two concentric metallic tubes (outer tube  $55 \times 235$  cm), both provided with helical baffles to propel the material. The outer tube

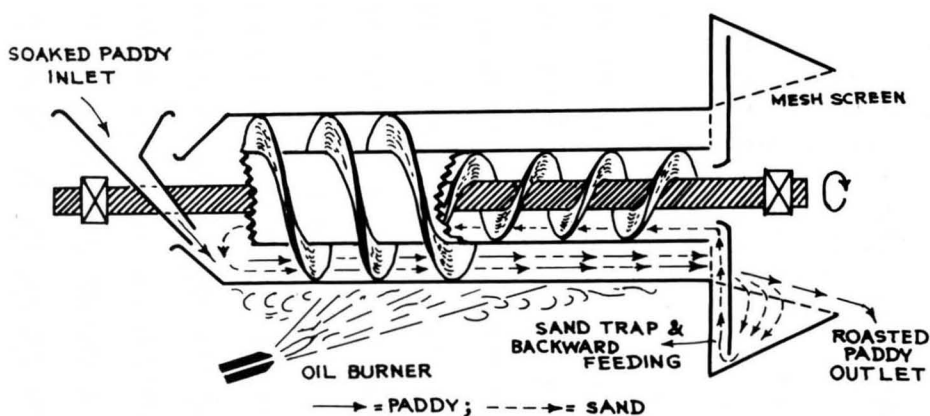


FIG. 3. SCHEMATIC DIAGRAM OF LARGE-SCALE OIL-FIRED GRAM ROASTER

carried the sand and grain mixture forward, while the inner tube carried the sand, separated through a screen and automatically retrieved at the exit point, backwards. Grain was fed continuously through an adjustable opening from a hopper at the rear end, and came out (after screening out the sand) through a chute at the exit end. The whole equipment was mounted on bearings in a fire-brick chamber; it was rotated (10 rpm) by an electrical motor and was heated by an oil-fired burner from below. The unit, along with the prescribed quantity of sand, was initially pre-heated to about  $175^{\circ}\text{C}$ , after which feeding of paddy was started. The steady temperature reached during the actual roasting was  $130\text{--}140^{\circ}\text{C}$ . The residence time of paddy was about 80 s.

#### Post-Drying Handling and Analysis

For study of starch retrogradation, treated paddy (or a sample thereof) was held in a closed test tube or bottle, completely filled, either at room temperature (RT,  $25^{\circ}\text{--}30^{\circ}\text{C}$ ) (put in after cooling by spreading) for 24 h or at  $70^{\circ}\text{C}$  for 4–6 h (Ali and Bhattacharya 1976b). The paddy was then dried in the shade.

For milling, when required in small quantities for EMC-S studies (see below), 10–15 g paddy was manually milled using a small hand polisher (P. Colombini) after dehusking in a laboratory McGill sheller. For study of milling quality, the experimentally dried paddy was finish-dried in shade and then milled using a McGill sheller and a McGill miller (Bhattacharya and Sowbhagya 1972, Ali and Bhattacharya 1972a).

Moisture content of paddy or rice was determined by oven drying (Indudhara Swamy *et al.* 1971). The equilibrium moisture content at

tained by rice when soaked in water at RT (EMC-S) was determined by the methods described earlier (Indudhara Swamy *et al.* 1971, Ali and Bhattacharya 1976b). Briefly, about 2 g of the sample of whole milled rice or cut brown rice (see below) was put under distilled water in a beaker for overnight (about 18 h), by which time all samples reached moisture equilibrium. The grains were then strained and wiped with filter paper, and their moisture content, i.e. EMC-S, was determined by drying in an oven as usual. When using relatively wet paddy, where milling was not possible, the procedure was as follows: About 3 g of paddy was put under water in a Petri dish, each grain was cut open longitudinally into two halves by a sharp blade one by one, and the husk coverings were later removed manually — all as far as possible under water. The cut brown rice was then left overnight under water to attain equilibrium and treated next day as above. Relatively dry paddy, which was difficult to cut but easy to mill, was hand milled and the milled grains were soaked etc. as above for determining the EMC-S. It was known from earlier experiments (Ali and Bhattacharya 1976b) that the two procedures gave identical results. When EMC-S was thus determined in a sample immediately after steaming or roasting, it was called the 'immediate EMC-S' of the sample. EMC-S was usually again determined after storage and/or drying of the sample, which value was the stable EMC-S of the material.

The moisture contents of all samples are expressed on wet basis (w.b.) but the EMC-S is expressed on dry basis (d.b.) to better bring out its changes during drying, holding etc.

Alkali degradation of the milled kernels in 1% KOH was determined as described in an earlier paper (Ali and Bhattacharya 1972b). As discussed in that paper, nonparboiled rice in this alkali concentration would remain unaffected, while parboiled grains would show more and more degradation with increasing severity of parboiling. The test thus gave a confirmatory evidence of parboiling and its extent.

## RESULTS AND DISCUSSION

The general approach to the investigation was the same as adopted before (Ali and Bhattacharya 1976b), viz. to dry the soaked paddy under various high-temperature conditions and to determine the equilibrium moisture content attained by rice when soaked in water at room temperature (EMC-S). The extent of rise, if any, in EMC-S of the sample determined immediately after the treatment (designated as 'immediate EMC-S') as compared to the value of the untreated sample gave an index of the extent of gelatinization, i.e. parboiling. For example, the untreated

paddy gave an EMC-S of about 45%. Any treatment which left the EMC-S of the product essentially unchanged would indicate mere drying without parboiling. On the other hand a value of immediate EMC-S of 100% and over 150%, say, would indicate that the treatment led to mild and relatively severe parboiling, respectively. General appearance of milled rice (color, translucence, white core) and its alkali degradation were noted as confirmatory evidence of parboiling. The EMC-S of the sample was again determined after holding at RT or at 70°C; the fall in this value as compared to the immediate EMC-S value indicated the extent of starch retrogradation if any.

#### Parboiling by Hot-Air Drying

The rate of drying of soaked paddy at various air temperatures at an air-flow rate of 0.23 m<sup>3</sup>/min — i.e. the maximum that permitted an undisturbed bed of paddy — is shown in Fig. 4A. That a sufficiently high air temperature not only dried but also simultaneously parboiled the paddy is shown by the rise in its immediate EMC-S (Fig. 4B). Air at 120°C merely dried but hardly parboiled the paddy, while that at 180–200° gave a severe parboiling effect. These results were substantiated by the degradation shown by the milled rice grains in dilute alkali; a few selected results are shown in Fig. 5. Further, samples showing increased EMC-S showed typical translucent kernels characteristic of parboiled rice. However, the alkali test revealed that the extent of parboiling was not uniform among all the grains in a sample (Fig. 5) — probably a result of a stationary bed of paddy being exposed to hot air from one side.

When soaked paddy was first partially dried with 180°C air for 60 s (moisture content about 24%), it produced mild parboiled rice (immediate EMC-S, about 110% d.b.). When drying was continued at this stage at lower temperatures (150°, 120° and 100°C) with a view to reduce the cost of the process, the paddy merely dried with little or no further increase in the severity of parboiling (results not shown). On the contrary, there was a slight decrease in the EMC-S value on prolonged drying with 100°C air, indicating partial retrogradation of starch in dry paddy at high temperatures as already observed earlier (Ali and Bhattacharya 1976b).

A study of different air-flow rates at an air temperature of 180°C showed that the rate of drying (Fig. 6A) and the extent of parboiling (Fig. 6B) were dependent on the flow rate also. Apparently it was a question of total heat input. The effect was nearly as good at a flow rate of 0.18 m<sup>3</sup>/min as at 0.23 m<sup>3</sup>/min.

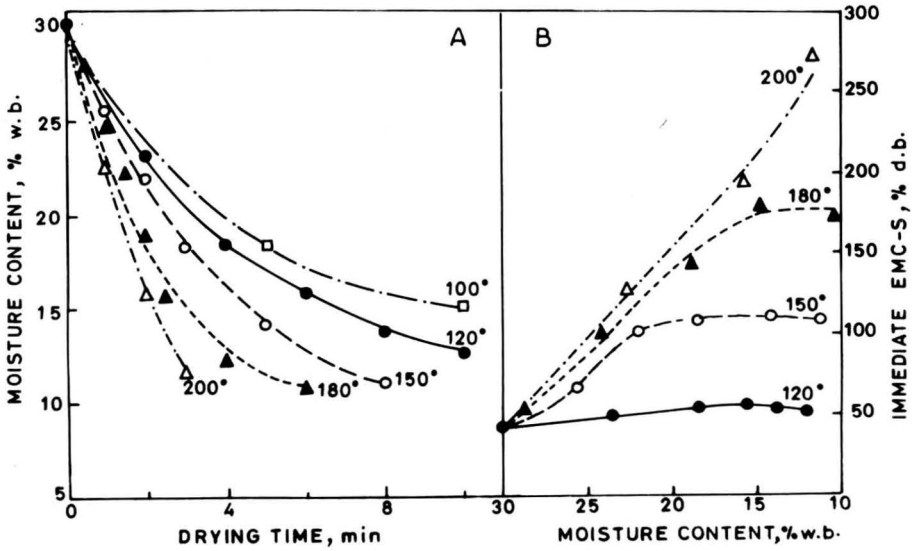


FIG. 4. CHANGES IN MOISTURE CONTENT (A) AND IMMEDIATE EMC-S VALUE (B) OF SOAKED PADDY WHEN DRIED WITH AIR AT VARIOUS TEMPERATURE ( $^{\circ}$ C, INDICATED)

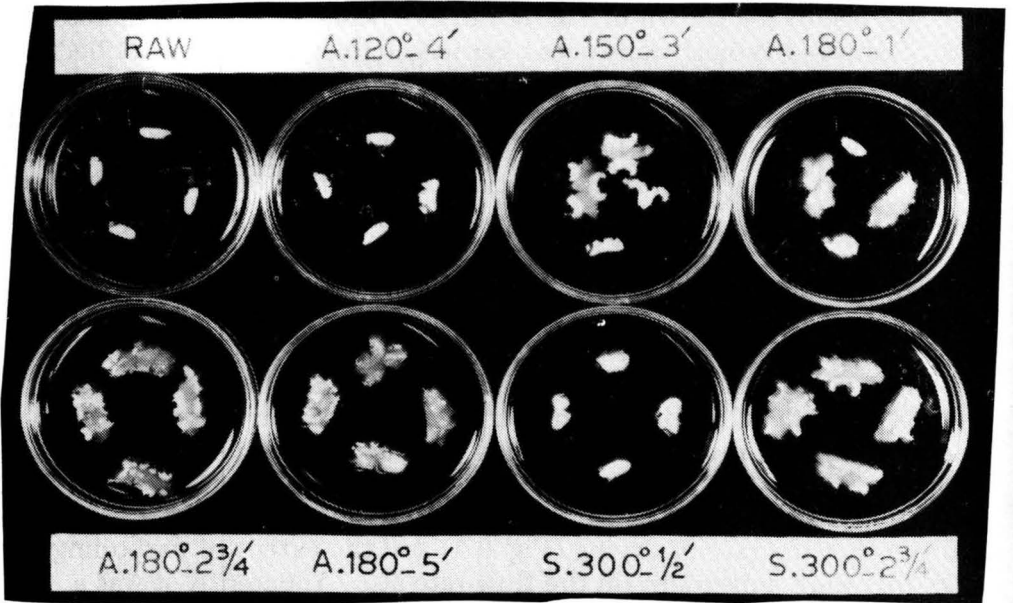


FIG. 5. ALKALI DEGRADATION OF PARBOILED RICE KERNELS UPON SOAKING IN 1.0% KOH OVERNIGHT

Samples dried with hot: A= air and S= sand; the two numerals stand for the temperature ( $^{\circ}$ C) and the duration (min) of the treatment, respectively.

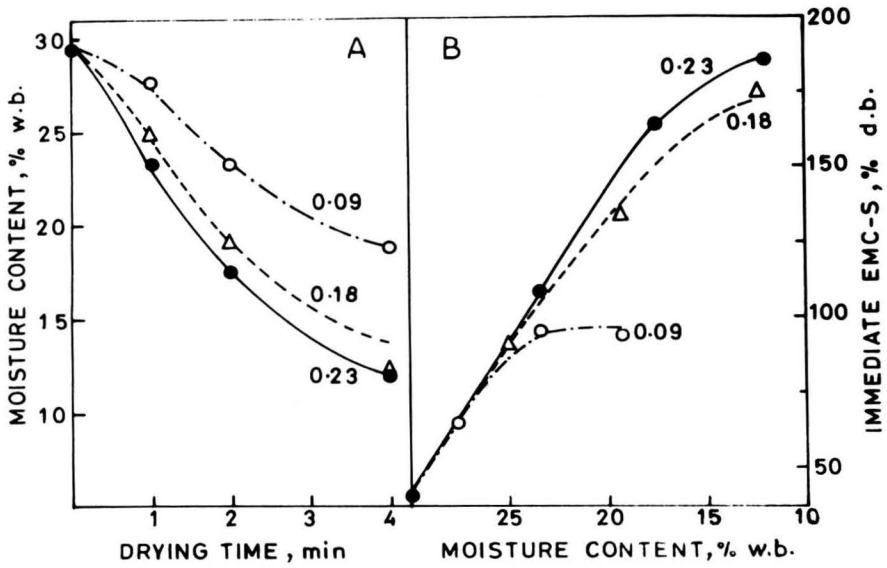


FIG. 6. EFFECT OF AIR-FLOW RATE ( $m^3/min$ , INDICATED) ON DRYING (A) AND PARBOILING (B) OF SOAKED PADDY

Air temperature 180°C.

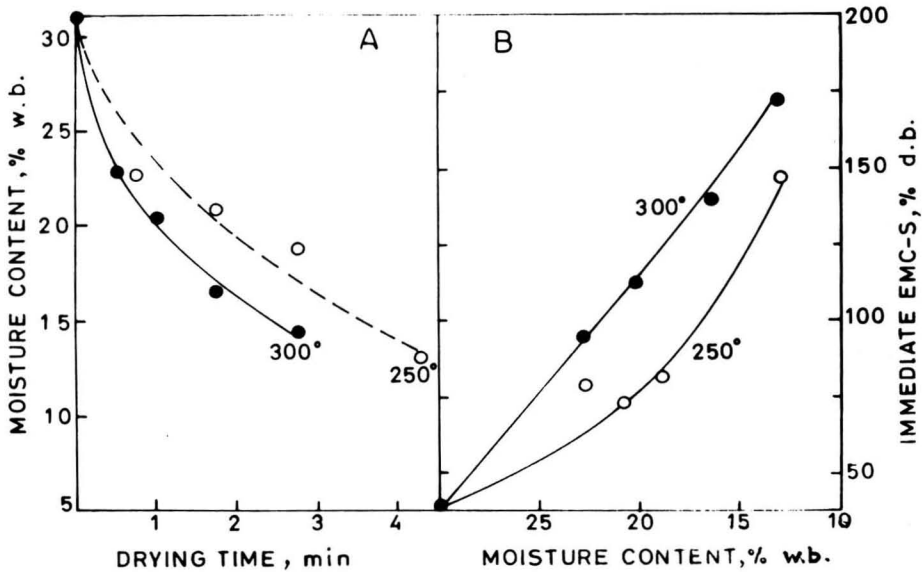


FIG. 7. EFFECT OF ROASTING SOAKED PADDY WITH SAND HAVING DIFFERENT INITIAL TEMPERATURES ( $^{\circ}C$ , INDICATED) ON ITS DRYING (A) AND SEVERITY OF PARBOILING (B)

### Parboiling by Sand Roasting

Based on the earlier observations (Ali and Bhattacharya 1976b), initial sand temperatures of 250° and 300°C were selected for the laboratory-scale trials. As shown in Fig. 7, mild to severe parboiled rice could be produced at both the temperatures by drying for the appropriate time. This was confirmed by alkali test (Fig. 5).

A semilarge-scale trial of this process was conducted by roasting about 150 kg paddy using the oil-fired continuous gram puffing unit. At the roasting temperature of 130°–140°C, the roasted paddy at the discharge end attained a temperature of 97°–98°C and a moisture content of about 21% (w.b.). The moisture content could be quickly reduced to below 20% by cooling and aerating the hot paddy by spreading it on the floor. The immediate EMC-S (93%) and alkali test (not shown) showed the rice was mildly parboiled, and the rice obtained after shade-drying and milling had typical parboiled-rice appearance.

### Starch Retrogradation

Starch retrogradation (with consequent loss in hydration ability) was studied in the above samples by determining their EMC-S again after holding and noting the drop in the value in comparison to the immediate EMC-S. Little or no retrogradation occurred in any of the samples upon holding at room temperature when its moisture content was 20% or less, higher-moisture samples giving progressively greater retrogradation. Quick shade drying prevented retrogradation even in slightly wetter samples. But holding at 70°C gave clear retrogradation even in samples in the moisture range of 15–20%. A few selected results are shown in Table 1. These results are in accord with earlier observations (Ali and Bhattacharya 1976b).

The above results confirmed one advantageous aspect of the process of drying-cum-parboiling as opposed to that of normal steam-parboiling, namely the hydration power of the rice was not unduly reduced by starch retrogradation (provided the moisture content of the paddy had been brought down during the process to 20% or below). However, the results also emphasized that the treated paddy had to be quickly cooled to room temperature if this advantage had to be retained (cf. difference between RT and 70° holding in Table 1); this could prove to be a somewhat difficult but crucial step in commercial-scale operation.

### Appearance of the Parboiled Rice

Mild to moderately parboiled rice obtained from any of the above processes had the characteristic color, translucence and appearance of



Table 1. Effect of high-temperature drying and of post-drying storage on change in hydration ability of soaked paddy

Medium	Drying Temperature °C	Moisture to Which Dried % w.b.	EMC-S <sup>1</sup> , % d.b.			
			Immediate	After Holding at RT	70°C	
Original soaked paddy		30.1	43	—	—	
Air	150	25.6	66	51	—	
		22.0	101	75	—	
		18.3	108	98	—	
		14.2	109	104	—	
		180	28.3	53	45	—
		24.0	100	62	—	
		18.8	144	136	—	
		14.8	178	169	—	
Sand	250	22.7	75	59	54	
		20.9	79	63	54	
		18.8	82	77	58	
		13.8	147	141	136	
		300	22.9	94	60	54
		20.3	112	109	61	
		16.5	140	137	116	
		14.6	159	156	146	

<sup>1</sup>Equilibrium moisture content attained by rice when soaked in water at room temperature

normal steam-parboiled rice. But severely parboiled rice produced by these methods (1) had a slight roasted, though acceptable, odor (caramelized odor in very severely parboiled rice), (2) appeared rather malleable when bitten by the teeth rather than brittle, even when fully dry, and (3) had grains that appeared rather flat and compressed and with somewhat pronounced ridges (Fig. 8). The last property apparently resulted from the sudden lateral collapse of the kernel due to the rapid drying, and the collapsed state was frozen in the dry kernel. For, when the same grains were remoistened and then slowly dried, they quickly regained their normal plump and smooth appearance. These ridges could be a disadvantage for milling, which would indicate that the process should be so controlled as not to lead to very severe parboiling.

#### Milling Breakage

Parboiling is well known to dramatically improve the milling quality of paddy (Bhattacharya 1969), which is in fact one of the main reasons that paddy is parboiled. The present processes proved on the whole satisfactory in this respect also. The control untreated paddy gave 35–40% grain breakage upon milling (g broken grains/100 g milled rice). In con-

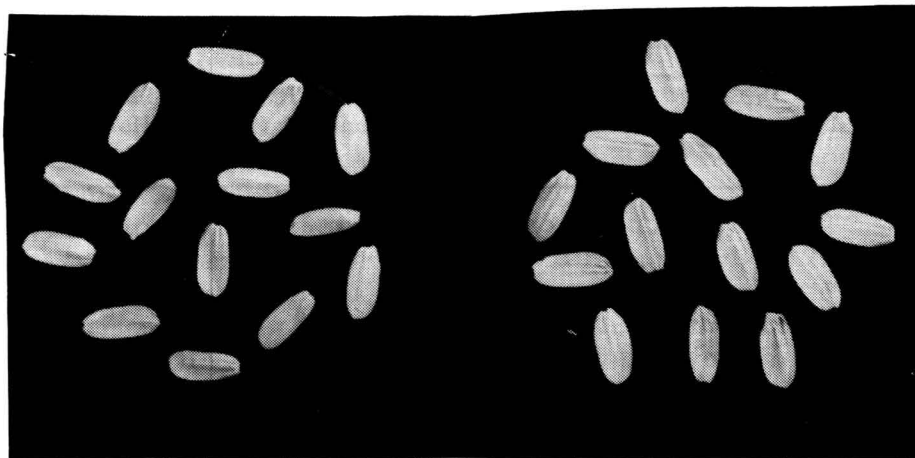


FIG. 8. APPEARANCE OF BROWN RICE OBTAINED FROM NORMAL STEAM-PARBOILING (LEFT) AND VERY HOT-AIR PARBOILING (RIGHT)

Note the flat appearance of and pronounced ridges in the latter.

trast, milling breakage was generally less than 5% in all the above cases (hence data not presented). However, samples processed with hot air at low temperatures ( $100^{\circ}$  or  $120^{\circ}\text{C}$ ) were exceptions; these samples, particularly those dried to a very low moisture content (15% or less), gave very heavy breakage (30–90%). This is evidently related to the low or negligible gelatinization caused at these low temperatures.

### CONCLUSIONS

These studies have demonstrated that soaked paddy can be parboiled by drying at high temperatures for a short time. The product so obtained has the advantage of easier hydration as compared to conventional steam-parboiled rice. Another likely value of the process is that it can possibly be applied directly to high-moisture harvest paddy without soaking (cf. Iengar *et al.* 1971, Khan *et al.* 1974). This could be one way of protecting paddy when harvested in rainy weather, such as the Kuruvai crop in Tamil Nadu.

The hot-air parboiling system would not appear from the present data to be economically feasible unless the air could be recycled to prevent the excessive loss of energy.

The roasting-parboiling system seems on the other hand to hold good promise for developing it into a commercial process, for which a suitable

continuous roasting system needs to be designed. The 'Sella' process is similar in principle but uses a rather low temperature. The partially moist roasted paddy is also often stored warm in a sealed container for a few days for unknown reasons before being finally dried in the sun. Without a knowledge of the effects of the temperature and moisture level during roasting and of storage of the moist product, a prime advantage of the process (viz., reduced retrogradation) may be partially or fully nullified. Detailed study of the process and its improvement as needed may be called for.

Based on the observations of the present and the earlier work, attention has to be given to the following factors for a successful operation of any system of drying-cum-parboiling of paddy:

(1) The paddy should be rapidly dried at a temperature proportional to the desired severity of the parboiling. However, very severe parboiling would be undesirable due to possible milling difficulties.

(2) The final moisture content during high-temperature drying should be about 20% (w.b.) for the following reasons: (a) A moisture content much greater than the above could cause two difficulties. First, there could be substantial to severe starch retrogradation upon cooling of the paddy (cooling would occur, for instance when the paddy was being terminally dried). Secondly, the milling quality of excessively moist parboiled paddy would be damaged during the terminal drying unless it was done in two stages (Bhattacharya and Indudhara Swamy 1967). (b) On the other hand if high-temperature drying was continued below 20% moisture, it could either lead to excessive breakage (temperature too high) or to substantial starch retrogradation in the warm semi-moist rice (temperature rather too low).

(3) The processed paddy should be immediately cooled to room temperature to prevent starch retrogradation.

(4) It should be finish-dried, after a suitable period of tempering, to about 14% moisture content (w.b.) for safe storage and/or milling.

#### ACKNOWLEDGMENT

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CONTENTS

Meetings . . . . . vii

Temperature Distributions and Liquid-Side Heat Transfer Coefficients  
in Model Liquid Foods in Cans Undergoing Flame Sterilization Heating  
R. L. MERSON, S. J. LEONARD, EARNEST MEJIA and J.  
HEIL, University of California, Davis, California . . . . . 85

Effects of Processing on Adsorption of Off-Flavors Onto Soy Protein  
ALAN CROWTHER, Celanese Chemical Company, Bishop, Texas,  
LESTER A. WILSON and CHARLES E. GLATZ, Iowa State Uni-  
versity, Ames, Iowa . . . . . 99

Effect of Deformation Rate and Moisture Content on Rice Grain Stiff-  
ness  
P. K. CHATTOPADHYAY, D. D. HAMANN and J. R. HAMMER-  
LE, North Carolina State University, Raleigh, North Carolina . 117

High-Temperature Drying-Cum-Parboiling of Paddy  
S. ZAKIUDDIN ALI and KSHIROD R. BHATTACHARYA, Cen-  
tral Food Technological Research Institute, Mysore, India . . 123