

of further autoxidation during fractionation, the oxidized substrates were stabilized by reduction prior to analysis. Each substrate from a Warburg autoxidation was divided into two parts. One part was reduced with Pt/C catalyst to give methyl stearate, oxygenated methyl stearate, and other oxidation products. The second part was reduced with lithium aluminum hydride to give oleyl, oxygenated oleyl, and stearyl alcohols, along with other products. Both reduced fractions were concentrated by TLC to remove unoxidized products that migrated faster, such as methyl stearate in one case and oleyl alcohol in

the other case. Further fractionation can be achieved by TLC with less polar solvents. This research is being continued.

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## CUSTARDS

### Viscous Behavior of Custard Systems

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For rapid cooling of menu items in quantity food service, agitation should be employed unless undesirable changes occur. Fluid properties of custards as affected by ingredients and heating were studied. Systems containing milk, egg, and starch were non-Newtonian and time-dependent. During shearing, viscosity losses increased as rate of shear increased, and were not recovered upon resting. Omitting egg or starch caused marked decreases in viscosity. When sugar was omitted, or when the system was overheated, protein-starch aggregates resulted which produced crossed hysteresis loops. For a custard of typical consistency, milk, egg, starch, and sugar have to be present; a cooking temperature of 185° F. cannot be exceeded. The role of sugar is to suppress protein flocculation during heating. The role of starch is to supply surfaces for protein-starch aggregate formation. Slow speed, short-time agitation of these systems is recommended.

BECAUSE of possible rapid multiplication of bacteria in large batches of warm food, the food should be cooled quickly to shorten the time in the hazardous zone of 140° to 45° F. (7). Previous work (6) has shown that agitating soft custards and puddings during cooling shortened cooling times considerably. However, the rate at which foods were agitated affected their apparent viscosity and, hence, their culinary acceptability.

Information on viscous behavior of food systems should be useful in suggesting speed of agitation during cooling and predicting cooling times. The present study was undertaken to define the viscous behavior of soft custards representing milk-egg-starch-sugar systems as affected by ingredients and heating.

#### Experimental Procedure

The formulas used, except where designated otherwise, are given in Table I.

Throughout the cooking period the heating temperatures were controlled by

the use of a rheostat and recorded by a potentiometer.

**Procedure.** Portion I (Table I), heat in top of double boiler. Portion II (Table I), sift dry ingredients together, add reconstituted egg and milk (when used), and mix 1 minute with electric beater. Add portion II to portion I

at 195° F. within 45 seconds, using 20 strokes with a small wire whip. At 5-minute intervals, agitate for 20 seconds during cooking, using a rubber spatula; keep the mixture covered except when stirred. Total cooking time 30 minutes; average cooking temperature 175° to 185° F. Remove a 150-ml. sample from the cooked mixture, cover, and cool at

Table I. Formulas for Systems and pH of Cooked Systems

System	Abbreviation	Portion I		Portion II				pH of Cooked Systems (Range)
		Milk, <sup>a</sup> g.	Sugar <sup>b</sup> , g.	Milk, <sup>a</sup> g.	Sugar <sup>b</sup> , g.	Starch <sup>c</sup> , g.	Egg <sup>d</sup> , g.	
Milk-egg-starch-sugar	MESSt <sup>e</sup>	2041	113		113	42	184	6.4-6.6
Milk-egg-sugar	MES	2004	113	37	113		184	6.5-6.6
Milk-starch-sugar	MStS	1880	113	161	113	42		6.3-6.4
Milk-sugar	MS	1843	113	198	113			6.4-6.5
Milk-egg-starch	MESSt	1956		85		42	184	6.4-6.6
Milk-egg	ME	1919		122			184	6.5-6.6
Milk-starch	MSt	1797		244		42		6.4-6.6
Milk	M	1760		281				6.4-6.5

<sup>a</sup> Spray-dried, low-heat, nonfat milk solids, reconstituted in the proportion of 227 grams of solids to 1814 ml. of distilled water.

<sup>b</sup> Commercial granulated sucrose.

<sup>c</sup> Commercial cornstarch.

<sup>d</sup> Stabilized dry egg solids, reconstituted 1:3, egg to distilled water, by weight.

<sup>e</sup> System MESSt was a complete soft custard.

room temperature for 1 hour before testing. Determine pH. Transfer the sample to a cylinder of the Haake Rotovisco; allow 10 minutes for temperature equilibration.

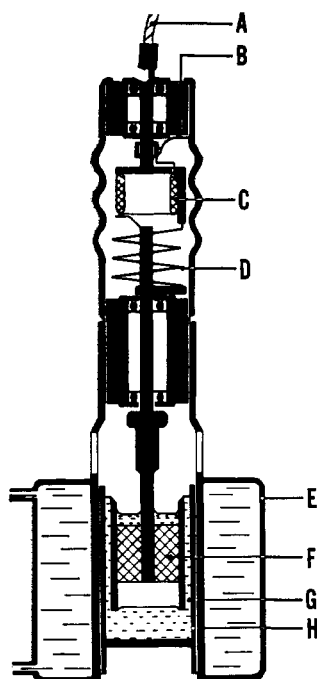


Figure 1. Haake Rotovisco, measuring head with MV attachment

- A. Flexible shaft
- B. Electrical connection
- C. Potentiometer
- D. Dynamometer
- E. Tempering jacket
- F. Rotor
- G. Sample
- H. Sample cup

**Rheological Measurements.** This viscometer measures apparent viscosities in the range of  $5 \times 10^{-3}$  to  $4 \times 10^7$  poises. The apparent rates of shear range from  $10^{-2}$  to  $10^4$  seconds<sup>-1</sup>, and the shear stress ranges from 10 to  $10^6$  dynes per sq. cm. The shear stress-shear rate relationships of the test materials can be determined directly with an expected error of 1 to 2%.

In the present study, the rates of shear used ranged from 8.46 to 685 seconds<sup>-1</sup>. The cylinder-bob combination provided a gap width of 1.92 mm. A recorder was employed in connection with the viscometer. The determinations were made at three temperatures—40°, 90°, and 150° F. The measuring head with cylinder (sample cup) and rotor are shown in Figure 1.

**YIELD STRESS.** To determine yield stress, the lowest shear rate was used and the torque on the bob was increased in small increments to just beyond the yield point. The reading was taken after a 100-second rest period.

**FLOW CURVES.** The lowest shear rate applied to a system was that which resulted in a 5% deflection. At each speed, a 9-second shear period was employed, with a 5-second rest period between shears.

**HYSTERESIS LOOPS.** Hysteresis loops were determined for all systems by shearing at decreasing rates following application of the highest rate of shear.

Since the custard without sugar exhibited an unusual hysteresis loop, the full custard (MESTs) and the sugarless custard (MEST) were studied for structural changes attributable to specific ingredients and heat treatment.

**FLOW BEHAVIOR AT CONSTANT RATE OF SHEAR.** Constant shear was applied

for 2 hours at 16.9, 51, and 152 seconds<sup>-1</sup>.

**REGENERATION OF VISCOSITY.** Regeneration was determined by initially shearing for 20 minutes, resting for specified periods, and again shearing briefly. Three consecutive determinations were made, allowing rest periods of 15, 30, and 60 minutes.

## Results

**Yield Stress.** Yield stress was measurable only in the complete and sugarless custards (MESTs and MEST). The average initial yield values were, respectively: at 150° F., 17.5 and 24.6 dynes per sq. cm.; at 90° F., 50.1 and 63.3; and at 40° F., 142.7 and 176.0.

**Flow Curves.** Representative flow curves, shown by the up portion of the loops (Figures 2 and 3), indicate that the systems were non-Newtonian. For milk and milk-sugar, only two points were obtainable, and their flow behavior could not be characterized.

The degree of non-Newtonian behavior could not be established. The power law equation, shear stress =  $K(D)^n$  (where  $K$  is the fluid consistency index,  $D$  is the rate of shear, and  $n$  is the flow behavior index), could not be fitted to the data of all the systems under all the temperature conditions. This was also true for Casson's (2) formula,  $\sqrt{\tau} = k\sqrt{D} + \sqrt{C}$  (where  $k$  and  $C$  are constants). These formulas have been applied to some nontime-dependent non-Newtonian food materials (3). Harper and El-Sahrigi (4) working with tomato concentrates reported that many times the flow curves were erratic at

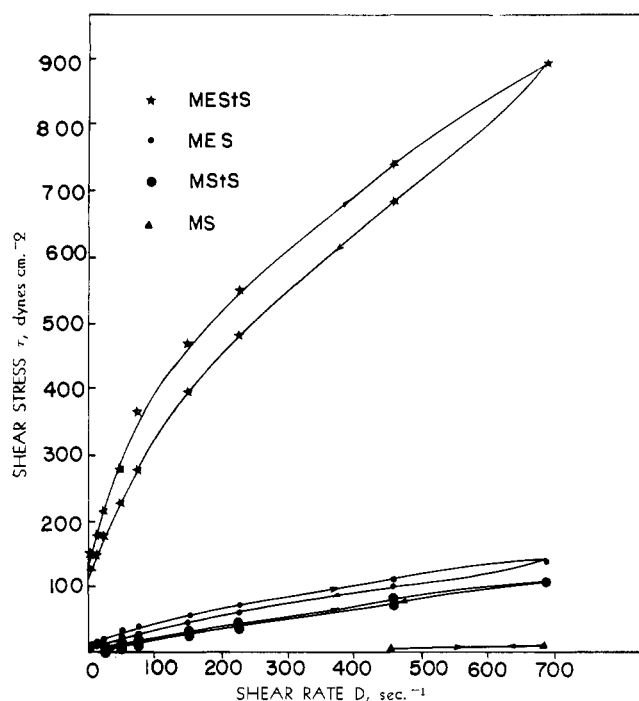


Figure 2. Hysteresis loops of systems at 90° F.

No loop formed by MS and M

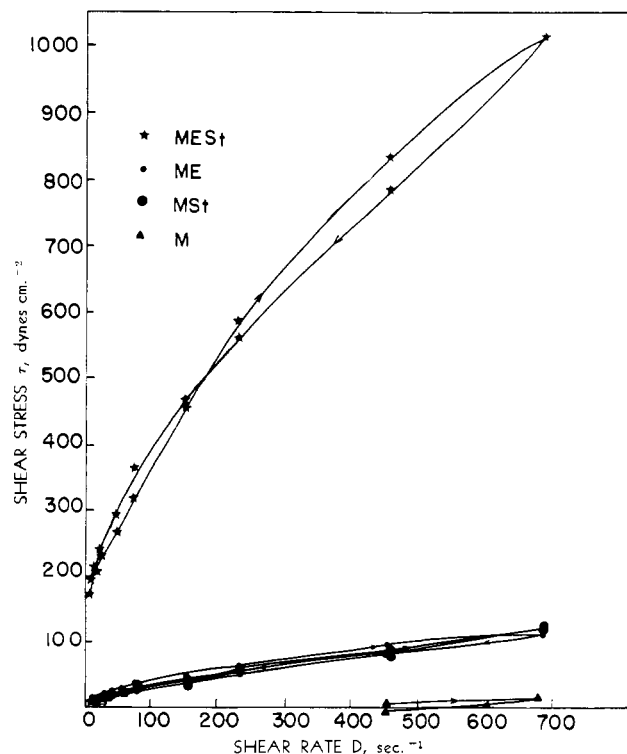


Figure 3. Hysteresis loops of systems at 90° F.

the low-speed end when the more concentrated materials were used; they found the irregularity to be related to yield stress. These authors used down curves as a basis for analysis, which approach linearity over a greater range than the up curves.

Apparent viscosity values (Table II) were calculated from the flow data using the formula  $\tau/D = \eta$ . The systems containing no sugar were of higher apparent viscosity than the comparable sugar-containing systems. The systems containing both egg and starch were considerably higher in apparent viscosity

than the comparable systems containing either egg or starch. In general, as the temperature was decreased from 150° to 90° to 40° F., the values approximately doubled each time.

**Hysteresis Loops.** Hysteresis loops were measurable only in systems containing egg. To obtain a quantitative measure of the effect of ingredients and of test temperature on the hysteresis loops, the absolute areas of the loops were calculated by a planimetric method. Each loop area was expressed as a percentage of the total area (Table III), since systems differed in composition and

temperature during shear. The relationship temperature vs. loop area was a straight-line one for the full custard but not for the sugarless custard. The hysteresis loops of the sugarless custard (MESt) were of an unusual shape. They exhibited a "Figure-Eight" phenomenon which was brought about by two intersections of the up curve with the down curve.

**Effect of Ingredients on Hysteresis Loops.** The role of ingredients was studied to find out the influence of each on the formation of aggregates and the Figure-Eight phenomenon.

**Table II. Apparent Viscosity Values<sup>a</sup> of Systems at Shear Rates 152, 228, 457, and 685 Sec.<sup>-1</sup> at 150°, 90°, and 40° F.**

		Apparent Viscosity Values of Systems, Centipoises								
		150° F.			90° F.			40° F.		
Sec. <sup>-1</sup>	R.P.M.	MESSt <sup>b</sup>	MES <sup>c</sup>	MSSt <sup>d</sup>	MESSt	MES	MSSt	MESSt	MES	MSSt
152	65	146.0	22.1	10.5	309.8	39.0	19.8	567.9	97.6	59.5
228	97	119.0	18.0	10.0	244.8	32.5	18.9	456.1	76.6	53.3
457	194	90.6	13.7	9.1	166.4	25.2	16.9	325.7	54.0	45.0
685	292	72.4	11.5	8.5	135.0	21.0	15.7	257.1	42.5	40.7
		MESSt <sup>e</sup>	ME <sup>f</sup>	MSSt <sup>g</sup>	MESSt	ME	MSSt	MESSt	ME	MSSt
152	65	153	28.3	13.1	302.3	28.9	29.1	599.3	134.1	76.9
228	97	124	24.2	12.0	255.8	23.5	24.5	567.9	103.6	66.4
457	194	92	19.2	10.0	181.5	18.0	19.8	435.2	72.0	53.6
685	292	74.6	17.4	9.2	146.5	14.9	17.8	345.5	55.0	47.6

<sup>a</sup> Three replications. <sup>b</sup> MESSt = milk-egg-starch-sugar. <sup>c</sup> MES = milk-egg-starch. <sup>d</sup> MSSt = milk-starch-sugar. <sup>e</sup> MESSt = milk-egg-starch. <sup>f</sup> ME = milk-egg. <sup>g</sup> MSSt = milk-starch.

**Table III. Hysteresis Loop Areas<sup>a</sup> of Egg-Containing Systems at 150°, 90°, and 40° F.**

		Absolute and Relative Loop Areas of Systems			
		Absolute area, dynes cm. <sup>-2</sup> sec. <sup>-1</sup>	Relative area, %	Absolute area, dynes cm. <sup>-2</sup> sec. <sup>-1</sup>	Relative area, %
Temp., ° F.		MESSt <sup>b</sup>		MES <sup>c</sup>	
	150	9,520	2.8	2,000	3.7
	90	35,270	5.9	6,350	6.4
	40	110,800	9.1	16,825	8.5
		MESSt <sup>d</sup>		ME <sup>e</sup>	
150		7,940	2.3	11,190	1.5
90		25,000	3.7	4,625	6.6
40		141,000	8.7	36,300	13.4

<sup>a</sup> Three replications. <sup>b</sup> MESSt = milk-egg-starch-sugar. <sup>c</sup> MES = milk-egg-sugar. <sup>d</sup> MESSt = milk-egg-starch. <sup>e</sup> ME = milk-egg.

MILK. The substitution of distilled water for milk resulted in a system which was very fluid and contained no visible aggregates. Use of fresh skimmed milk when untreated, or heat-treated, or to which sodium caseinate had been added, did not result in a "normal" MESSt system. When nonfat milk solids were reconstituted to an 8% solids level, the resulting system was very similar in appearance to the "normal" MESSt system of 12% solids, in that it contained visible aggregates. The system, however, was of much lower viscosity and did not produce the Figure Eight of the hys-

**Table IV. Average<sup>a</sup> Initial Apparent Viscosity and Average Per Cent Change in Apparent Viscosity of Systems after 30, 60, and 120 Minutes of Shearing at 150°, 90°, and 40° F.**

		150° F.			90° F.			40° F.						
Shear Rate, Sec. <sup>-1</sup>	R.P.M.	System	Av. initial appar. visc., cp.	Av. Decrease (%) in Appar. Visc. after			Av. initial appar. visc., cp.	Av. Decrease (%) in Appar. Visc. after						
				30 min.	60 min.	120 min.		30 min.	60 min.	120 min.				
16.9	7	MESSt <sup>b</sup>	624	27.7	39.3	52.7	1418.5	40.8	54.4	66.4	2724.8	28.9	42.0	53.4
51	22	MESSt	309	35.3	44.7	54.0	601.2	43.8	52.7	62.5	1006.4	48.1	55.5	62.5
152	65	MESSt	162	35.8	45.7	54.9	292.1	50.4	57.0	62.4	598.6	57.2	62.9	66.6
152	65	MES <sup>c</sup>	21.1	39.8	43.6	43.6	23.6	24.6	29.7	33.5	84.9	41.6	45.2	47.9
152	65	MSSt <sup>d</sup>	11.1	22.5	22.5	22.5	21.3	11.3	11.3	11.3	57.6	8.7	10.8	13.9
16.9	7	MESSt <sup>e</sup>	627	14.2	22.8	32.4	1369.3	36.1	35.7	51.2	2443.0	33.4	47.1	58.3
51	22	MESSt	288	32.3	42.7	50.0	748.6	27.6	44.9	51.6	1340.5	41.3	55.1	63.6
152	65	MESSt	148	32.4	42.6	52.0	345.4	45.5	52.4	62.2	641.0	54.2	61.8	67.6
152	65	ME <sup>f</sup>	26.8	39.6	45.5	47.8	49.2	49.6	53.2	55.1	87.3	37.7	38.4	41.9
152	65	MSt <sup>g</sup>	12.5	4.8	...	...	32.7	16.5	20.8	25.4	71.5	11.2	13.8	16.9

<sup>a</sup> Three replications. <sup>b</sup> MESSt = milk-egg-starch-sugar. <sup>c</sup> MES = milk-egg-sugar. <sup>d</sup> MSSt = milk-starch-sugar. <sup>e</sup> MESSt = milk-egg-starch. <sup>f</sup> ME = milk-egg. <sup>g</sup> MSt = milk-starch.

teresis loop. Within the scope of this study, the crossover phenomenon could be produced only by using reconstituted nonfat milk solids at a 12% solids level.

**EGG.** When yolk replaced whole egg, systems were obtained which had low viscosities and broke down rapidly with shear; their hysteresis loops were open (Figure 4). The egg-white system gave crossed hysteresis loops similar to those of a whole-egg system. The difference in shear resistance between the systems made with white or yolk is probably caused by a difference in the toughness of aggregates, the egg-white systems possessing the greater toughness.

**STARCH.** To prepare a "normal" custard, either complete or sugarless, the timing and sequence of incorporating starch and egg into milk were found to be critical. For the formation of aggregates typical of a normal custard the starch had to be present before the egg proteins coagulated. Because of these findings the role of starch and its components in the formation of custard aggregates was explored. The combination of amylose-amylopectin and the crystalline amylopectin alone resulted in extremely thin products devoid of visible aggregates. However, modified waxy maize produced a system with properties similar to those of the cornstarch system. A hypothesis was proposed that the starch was not acting in chemisorption with the protein to form the aggregates but rather as a physical agent. When sand, ground pumice, graphite, and diatomaceous earth, of similar particle size, were substituted for the starch, the aggregates resulting from these substitutes were very similar in appearance to starch-protein aggregates. When examined microscopically, the inert substances were found to have filled the role played by the starch granules in the system (Figures 5, 6, and 7). Thus, a function of starch was to provide surfaces for aggregate formation.

**SUGAR.** The effect of varying the amount of sugar on the hysteresis loop is shown in Figure 8. When the systems were prepared with equal heat treatment, the proteins of the sugarless custard, MEST, were more denatured than in the complete custard, because sugar raises the denaturation temperature of egg protein (7).

**Effect of Heat Treatment of Systems on Hysteresis Loop.** Previously, we showed that the sugarless custard exhibited unusual hysteresis loops. Now, the effect of heat was studied. By elevating the cooking temperature of the complete custard from 180° to 190° F., a hysteresis loop was obtained which was similar to that of a sugarless system cooked at 180° F. This "overheated" custard exhibited the Figure-Eight phenomenon. On the other hand, on lowering the cooking temperature of the sugarless system to 173° F., the hysteresis loop

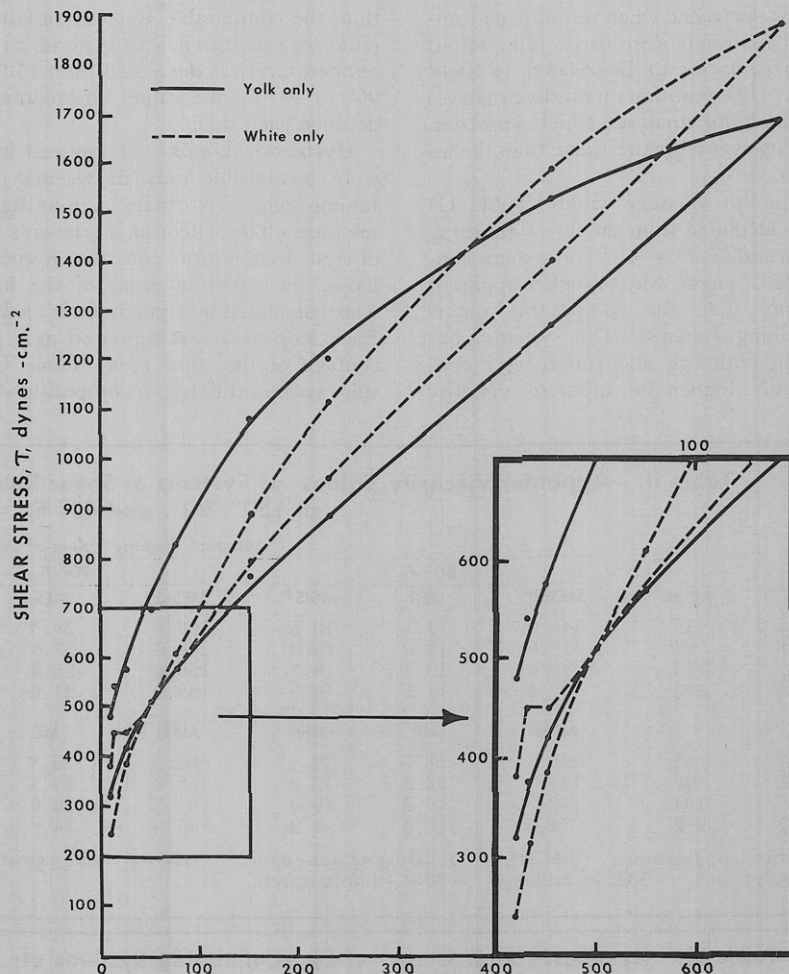


Figure 4. Hysteresis loops of milk-egg-starch systems made with egg yolk and egg white

Determined at 40° F.

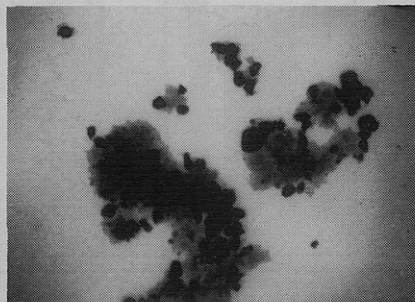


Figure 5. Aggregates of milk-egg-starch system (MEST)

Magnification 100×



Figure 6. Aggregates of milk-egg-sand system

Magnification 100×

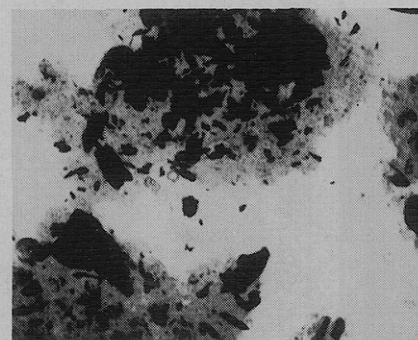


Figure 7. Aggregates of milk-egg-graphite system

Magnification 100×

produced was similar to that of the sugar-containing system cooked at 180° F. (Figure 9). This seems to show that the retardation of protein denaturation, effected by sucrose, was the main cause of the difference in the hysteresis curves of the systems.

Through experiments in which the particles were size-graded, it was learned that the size of the aggregates alone was not responsible for the appearance of the crossover phenomenon; therefore, toughness of the aggregates must also play a role. From what is known about the effect of heat on protein, toughness is

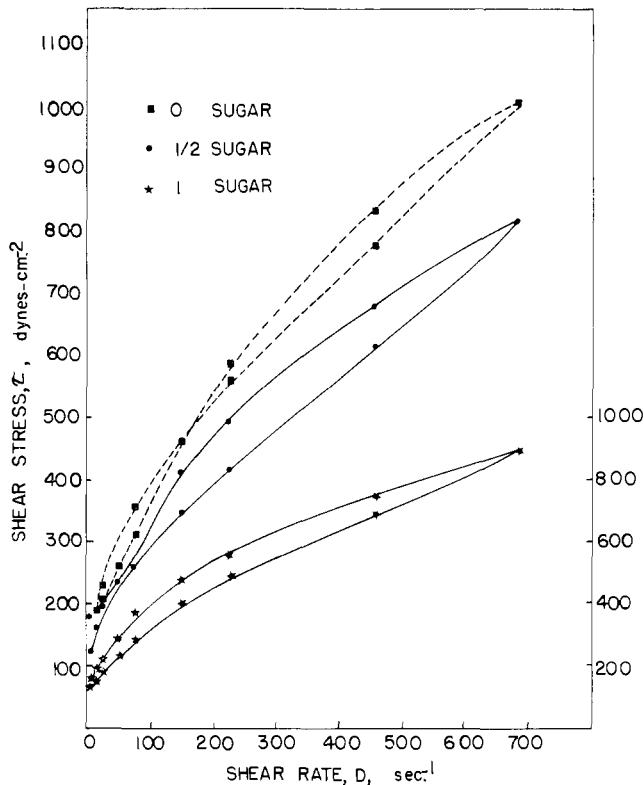


Figure 8. Hysteresis loops at 90° F. of three milk-egg-starch systems varying in amount of sugar

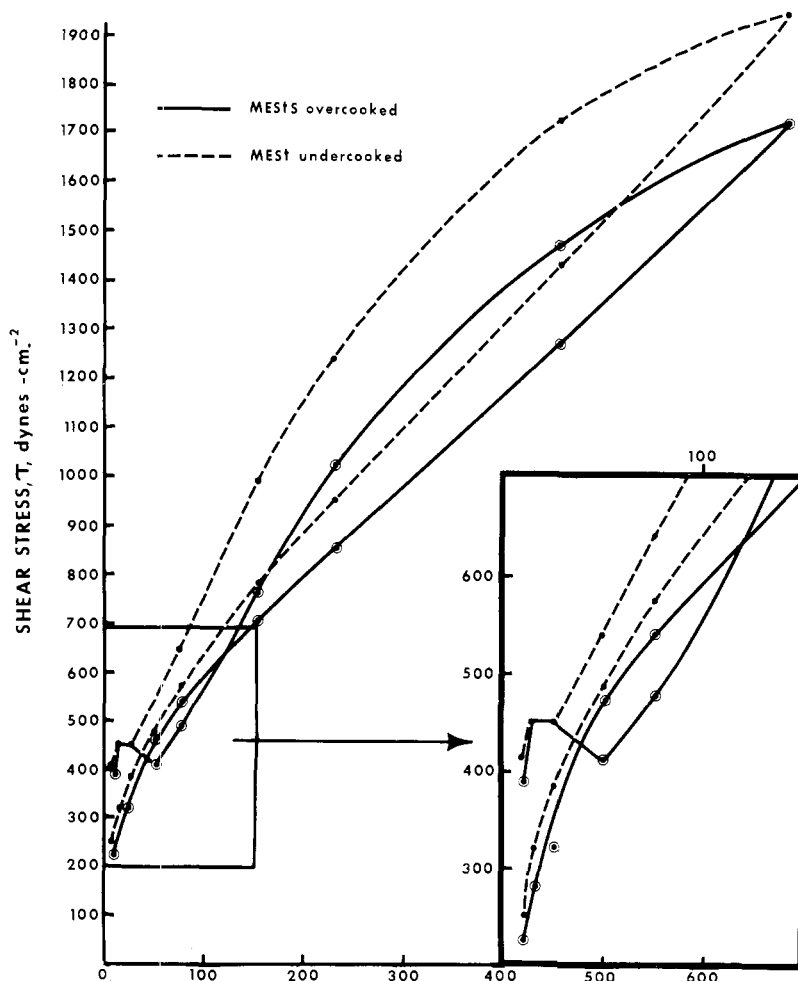


Figure 9. Hysteresis loops of overcooked MESSt and undercooked MESSt systems

Determined at 40° F.

affected mainly by the degree of denaturation and aggregation. The two main factors shown in these protein systems to influence the degree of denaturation and aggregation are: the cooking temperature, and the presence or absence of sucrose. The two systems, the sugarless and the overcooked sugar-containing custards, which were expected to be composed of relatively tough aggregates, produced the Figure-Eight phenomenon.

#### Flow Behavior at Constant Shear.

At constant shear, the change in apparent viscosity with time is an important criterion of the time-dependent property of a system. At each testing temperature, the per cent change in apparent viscosity was greatest during the initial shear period of 20 to 30 minutes, especially in the complete custard (Table IV).

"Humping" was observed in the sugarless custard (Figure 10) at low test temperature and at slow and intermediate rates of shear. Humping may be due to entanglement of large aggregates observed in the sugarless custard.

The dramatic changes in apparent viscosity at continuous shear over a 2-hour period cannot be attributed to the variable, time; they are the result of shear, as shown in a separate experiment. Holding samples of custard for 2 hours without shear for various time intervals, up to 2 hours, did not change initial viscosity nor behavior during shear at a constant rate after this 2-hour hold.

Since it would be desirable to predict the viscosity at any time during continuous shear at a constant shear rate, attempts were made to fit the data to three mathematical models. A three-constant model

$$\eta = A + \frac{B}{C + t} \quad (1)$$

was examined. This model, as well as the following ones, implies that after an infinite time of shear,  $t$ , the viscosity is constant and equal to  $A$ . A second attempt to predict the experimental viscosity data was made with the equation

$$\frac{1}{\eta - \eta_{t=\infty}} = Ct \quad (2)$$

A third model was based upon the equation

$$\ln(\eta - \eta_{t=\infty}) = Ct \quad (3)$$

Equation 3 gave a good fit for the portion of the curve between 20- and 100-minute shear, although not for the initial 20-minute period. For the sugarless custards the fit was, in general, poor (Figures 11 and 12).

**Regeneration.** Regeneration during the 2-hour period of rest was very slight to nil. The systems described here cannot be classified as thixotropic, since thixotropic systems will regenerate upon resting. Nonrecoverable breakdown brought about by shear has been referred

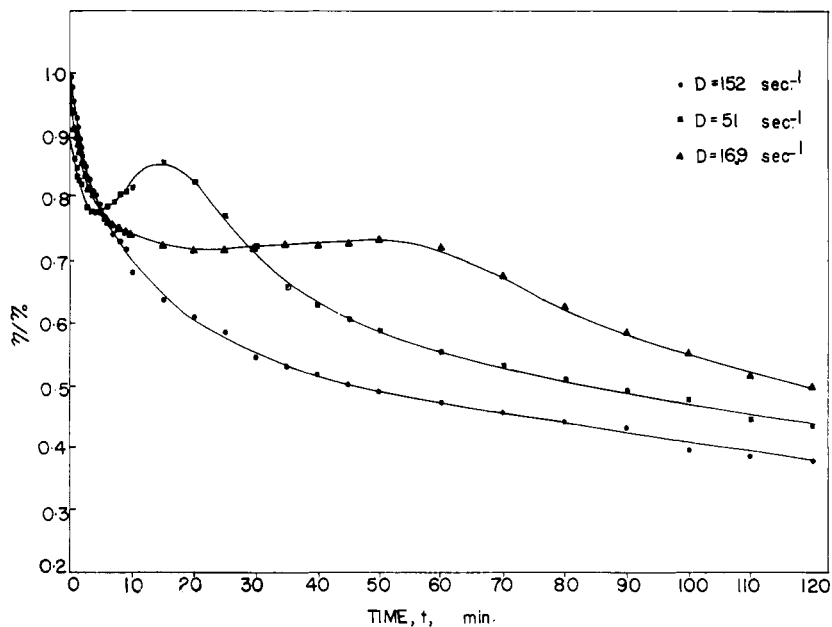


Figure 10. Time-dependence curves illustrating humping of sugarless custard at three rates of shear

Tested at 90° F.

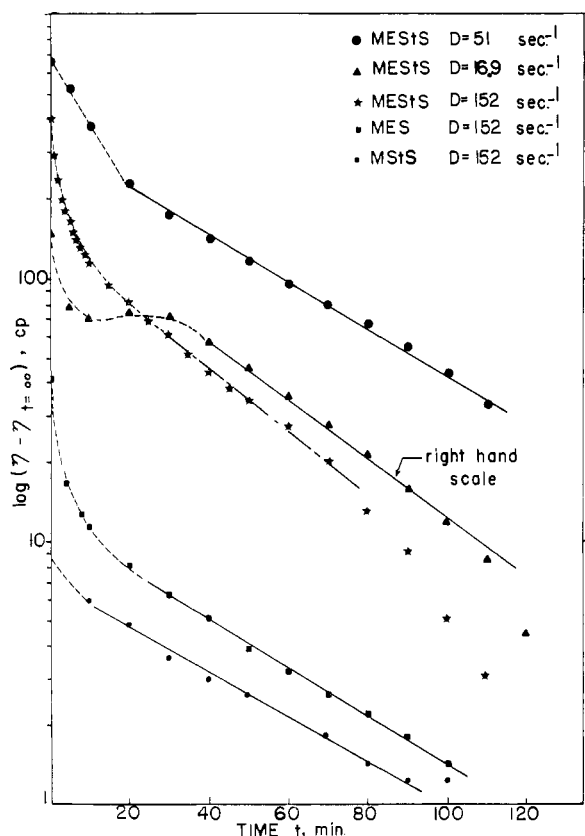


Figure 11. Viscosity-time relation of sugar-containing systems tested at 90° F.

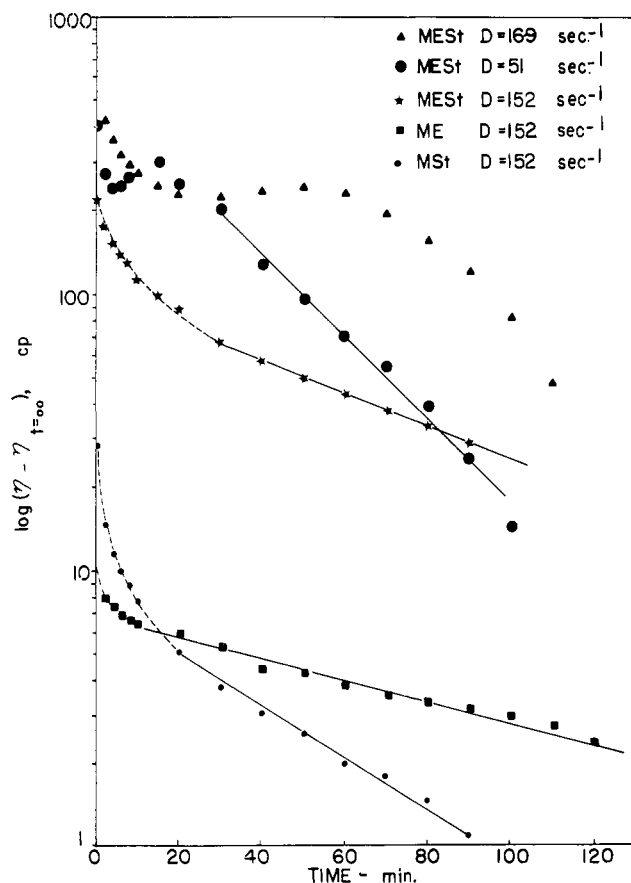


Figure 12. Viscosity-time relation of sugarless systems tested at 90° F.

to as "Rheodestruction" by Höppler (5). From observation of the samples before and after shearing, the unsheared custards did not appear homogeneous, although not curdled, whereas the sheared samples were invariably smooth

and homogeneous. When resting periods of 24 hours were allowed, the original viscosity was not restored.

#### Significance

The purpose of the study was to define

the viscous behavior of custard systems as affected by ingredients and heating. The most significant points are: For a custard of typical consistency, milk, egg, starch, and sugar had to be present and a cooking temperature of 185° F. could

not be exceeded; the role of sugar was to suppress protein flocculation during heating and the role of starch was to supply surfaces for protein-starch aggregate formation. Vulnerability of the systems to shear was demonstrated.

#### Acknowledgment

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## HICKORY SMOKE

### Composition of Hickory Sawdust Smoke. Furans and Phenols

Eighteen components derived from hickory wood smoke have been separated by gas-liquid chromatography and identified by comparing the retention times and infrared spectra with known authentic samples. Three additional components have been given tentative identifications and three remain unidentified. Five components of wood smoke—acetol, 2-cyclopentenone, cyclotene, 4-vinylguaiacol, and eugenol—have not been reported, previously.

FOOD PRODUCTS have been processed for countless years by exposure to wood smoke. This treatment contributes to desirable flavor and color and exerts a preservative effect. With the development of other methods of preservation, foods now are smoked mainly for their sensory qualities. In recent years, beginning with the work of Pettet and Lane in 1940 (10), intensive studies have been made on the parameters of formation and the composition of smoke for the purpose of improvement in the quality of smoked meat and food products. Phenols have been reported primarily responsible for the smoky aroma and taste of food products (7, 17) although other major classes—such as acids and carbonyls—have been identified in smoke (4, 5, 12, 16).

Early investigations of the phenolic fraction of smoke preparations reported determination of phenols on the basis of total phenols, using colorimetric methods with phenol or guaiacol as standards. However, this technique has two deficiencies: The color values for the various phenols are not always equivalent, so true quantitation was never achieved (13, 18); and individual phenols were not identified. In recent years, a number of studies have been carried out in which phenolic components of wood smoke have been identified. However, the methods of generating the smoke, collecting the phenolic fraction, and identifying the

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components have not been uniform or reproducible. Correlation of the results from these investigations to obtain meaningful information about the desirable phenol composition of smoke and the best parameters for its production is difficult.

Goos (2) has tabulated references to more than 200 compounds that were found in the condensate of the destructive distillation of wood. However, the combustion of wood for the smoking of food items involves both the destructive distillation process and oxidative interactions of the products of this process. As a result, the composition of wood smoke could be different from that of the condensate. While most of the components of smoke reported to date were found also in the destructive distillation condensates, independent identifications must be made of components of all smoke preparations to confirm their absence or presence under varying conditions of smoke production and treatment.

Eight literature reports have been collected in which individual phenols were identified (7, 3, 6, 8-10, 15, 19). Of approximately 30 phenols identified, many have not been reported by more than one investigator. The method used for identification was important with respect to the number of phenols identified. Only limited numbers were detected and identified by paper chro-

matography. The sensitivity of paper chromatographic methods is such that many components present in wood smoke may not be detected. Furthermore, identifications based solely on the comparison of GLC or paper chromatography retention times, in which a number of different compounds can give the same retention values, leave some doubt as to the actual presence of some of these compounds in smoke. In recent reports, however, in which GLC techniques were used, Jahnsen (8) identified 12 phenols and Sikorski (14) reported 17 peaks that had phenolic characteristics as determined by functional group analysis. Unfortunately, Sikorski did not identify a single one of his components.

Since studies of meat processing are likely to require a knowledge of smoke composition and reactions, this laboratory is involved in the development of reproducible methods of smoke generation which can be used to study the effects of variables, such as combustion temperature, air flow, sawdust source, and others on the chemical composition of smoke. The data reported in this paper present information on the composition of a fraction of wood smoke consisting primarily of phenols; the results of investigations of other components of this smoke preparation are being studied.