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KINETICS OF PEPPERONI DRYING

ABSTRACT

The kinetics of sausage drying were described in terms of a rate equation and of the diffusivity for moisture in a sausage. The rate equation developed is: $dx/dt = -k[(x - c)/x]^2$, where k is the rate constant, t is time, x is percent yield at any time, and c is the ultimate percent yield at $t = \infty$. Data, as percent yield, are substituted into the integrated form of the equation and values for k and c are obtained. The mean square (MS) of deviations of the data from the theoretical curve was used to test agreement. Heating or fermenting the sausages reduced MS and improved agreement. After extended drying, actual percent yields of sausages approximated yields predicted by the rate equation. Estimation of final percent yield, on the basis of sausage weight early in the drying period, should facilitate efficient production and marketing of sausage. By use of equations developed by Sherwood and Newman, the apparent overall diffusivities for moisture in pepperoni were 5.7×10^{-7}, 5.6×10^{-7}, and 4.7×10^{-7} cm^2/sec, respectively, for pepperoni of starting fat content of 13.3, 17.4 and 25.1%.

INTRODUCTION

WE HAVE INVESTIGATED the influence of sausage formulation on percent yield of pepperoni at the standard drying period, 42 days of drying (Palumbo et al., 1976b). Most variations in formulation caused small, but statistically significant, differences in the percent yield of dried pepperoni.

We now evaluate the kinetics of the sausage drying process with pepperoni as a model product system. We have developed an equation that describes the drying process in terms of a rate constant and allows us to predict the percent yield of sausage after complete drying or at any time during the drying period. Further, since drying of a sausage involves the diffusion of moisture from the interior of the sausage to the surrounding environment, we have calculated the diffusivity or diffusion coefficient for moisture in a sausage.

MATERIALS & METHODS

Preparation of pepperoni

Standard pork-beef (1:1) pepperoni were processed, fermented, heated, and dried as described (Palumbo et al., 1976a, b).

Diffusivity

The diffusivity or diffusion coefficient was calculated by equations developed by Sherwood (1929) and Newman (1931a, b) as described by Treybal (1955) for unsteady-state diffusion of moisture through porous solids.

Data

The sausages were weighed weekly. Percent yield (x) was calculated as follows: weight at any time (W_x) divided by weight at the start (W_o) of drying × 100, i.e., $x = (W_x/W_o) \times 100$.

Statistical analyses

The data for percent yield and rate constant were analyzed by Duncan's new multiple range test (Steel and Torrie, 1960) by use of an IBM 1130 computer.

RESULTS & DISCUSSION

Rate equation

From percent yield data gathered weekly, the rate equation, $dx/dt = -k[(x - c)/x]^2$, was developed to describe the drying process; x is the percent yield at any time (t), c is the ultimate percent yield at $t = \infty$, k is the rate constant. The integrated form of the equation is:

$$kt = 100 - x + 2c \ln \left(\frac{100 - c}{x - c}\right) + \frac{c^2}{x - c} - \frac{c^2}{100 - c}$$

and requires the use of a computer to solve for c and k, particularly since the SS (sum of squares) must be minimized in the x direction. Thus, the equation is nonlinear in terms of the unknowns. Also, since the equation cannot be solved for x, once c and k have been estimated, the calculation of the SS is a nonlinear problem. We solved the equation with a computer program for an iterative pattern search technique (Hooke and Jeeves, 1961), and it is available on request.

After solution, the computer plotted both the actual data points and the theoretical curve (Fig. 1). The computer solu-

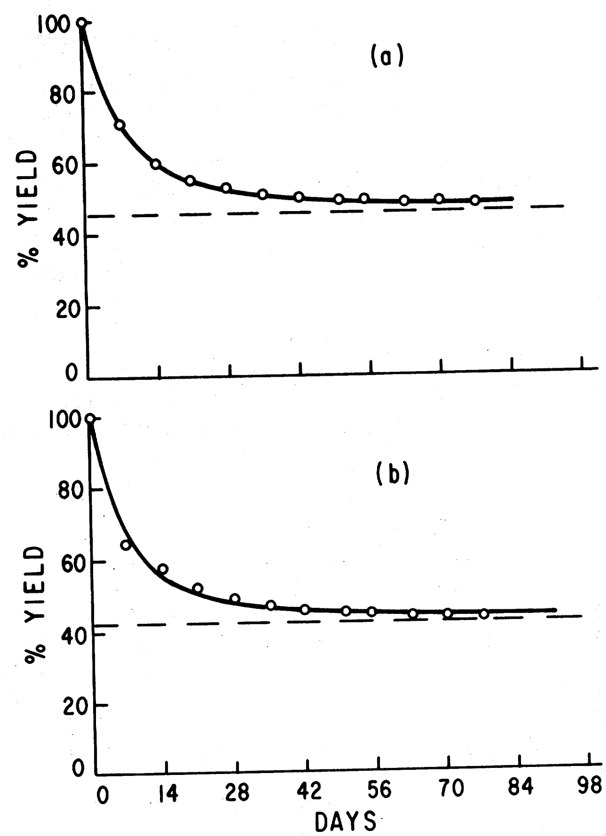

Fig. 1—Comparison of actual data points (open circles) with the theoretical computer-drawn plots (smooth curve) for two sausage variations: (a) good fit, MS = 0.12; starter culture fermented and heated to 140°F; (b) poor fit, MS = 2.20; not fermented and not heated.

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tion also provided a statistical value, the SS, which described the goodness of fit of actual data to the theoretical curve. For comparison among all our experimental data and with the data of Townsend and Davis (1972), the goodness of fit was expressed as the MS (mean square). MS is related to the SS by: $MS = SS/(n - 2)$, where n is the number of data points and 2 is the number of parameters in the rate equation. Use of MS eliminates the influence of the number of data points, which is part of SS, and thus permits comparison among experiments with different numbers of data points. The value of MS was relatively low for good visual fit of actual data to the theoretical values, but was relatively high for poorer visual fit of data to the theoretical (Fig. 1). Data for an experiment on the influence of fermentation method, pH, and final internal temperature on percent yield were evaluated by use of the rate equation. The calculated parameters are presented and the statistical analyses appear in Table 1.

The rate constants (Table 1) tended to be higher for sausages fermented with starter culture than for nonfermented sausages and for those fermented with natural flora. Duncan's test supported the interpretation of those general trends. Within each fermentation method, the rate constants were high for sausages dried without heating, and decreased as the final internal temperature increased. In contrast, the predicted ultimate percent yield increased within each fermentation method as the final internal temperature increased. To explain that phenomenon, we suggest that heating melts the fat, which then coats the meat particles and thus impedes the diffusion of moisture to the sausage surface. We observed a similar effect when the fat content of sausages was increased, and percent yield increased. In general, the trend of the predicted ultimate percent yield agrees with the trend of the percent yield at 42 days reported previously (Palumbo et al., 1976b).

The predicted ultimate percent yield was calculated from data obtained from 77 days of drying. Percent yield data for these sausages also were measured up to 106 days (Table 1). For many of the variables, the actual percent yield at 106 days was extremely close to the predicted ultimate percent yield, especially for variables with higher rate constants. The rate constant reflects the rate at which moisture is lost, so that sausages with higher rate constants lose moisture faster, and thus the actual percent yield should approach the predicted percent yield faster.

Values of goodness of fit, as defined by MS, for those data are also given in Table 1 and two examples appear in Figure 1. In general, the MS is lowest for SC-fermented pepperoni; further, within each group of fermentation methods, the MS decreased (fit was better) as the final internal temperature increased (perhaps also because the rate constant was lowered). As a group, the nonfermented sausages had high MS's, and the nonheated had the highest MS, 3.90. From a drying pattern standpoint, nonfermented sausages gave up moisture less regularly than fermented, but their predicted ultimate percent yields and rate constants were similar to those of the fermented sausages. When the data of Townsend and Davis (1972) were analyzed by the rate equation, MS was 1.84. Their sausages (Genoa salami) were neither fermented nor heated before drying, so their results agree with our findings. Denaturation of the protein whether by heat or acid (fermentation) is a requisite for a 2nd order drying pattern for sausages.

The general trends of the above calculated parameters have been observed during commercial drying of sausages. C.W. Everson (personal communication) observed that SC-fermented sausages dry faster than NF-fermented or nonfermented sausages, and the rate constants of Table 1 support his observation. The differences between fermentation methods were not as apparent when predicted ultimate percent yields were compared. For sausage varieties which are dried long periods (90–120 days), ultimate moisture content (percent yield) would be the important parameter, but for sausage varieties which are dried for relatively short periods (15–30 days), the rate constant would be the important parameter and the low pH (obtained by starter culture) would be desirable. Since desired chemical and microbiological reactions also occur during the drying period, the processor of dry sausage must consider those changes along with drying kinetics.

The processor of dry sausage could use the rate equation to estimate the final weight (amount of product) from any given production run and thus adjust marketing and production accordingly. The processor would weigh the sausages at intervals in the early stages of drying and use these percent yields in a complete computer program to solve the equation. To illustrate its usefulness, percent yield data for the first 3 wk of drying for the experiment of Table 1 were inserted into the rate equation and then percent yield at 42 days was estimated (Table 2). Predicted yields were further from the actual values for the nonfermented pepperoni (variables 5–8) than for those

Table 1—Influence of fermentation method (FM), pH, and final internal temperature on the drying rate constant, predicted ultimate percent yield, and mean square (as calculated from the rate equation) and actual percent yield at 106 days of drying for pork-beef pepperoni

Sausage no.	Description of sausage			Rate constant[b,c]	Predicted ultimate % yield[c]	Actual % yield at 106 days	Mean square
	pH	Int. temp °F	FM[a]				
1	4.6	not heated	SC	24.08b	43.70g	43.80	0.95
2	4.7	120	SC	35.56a	45.20ef	45.35	0.21
3	4.7	130	SC	22.54bc	45.88cd	46.15	0.28
4	4.7	140	SC	20.81cd	46.16c	46.55	0.24
5	5.8	not heated	not fer.	19.51de	42.76h	42.30	3.90
6	6.1	120	not fer.	17.57e	44.78f	44.85	1.70
7	6.1	130	not fer.	18.74de	45.62de	45.75	1.31
8	6.1	140	not fer.	18.86de	46.00cd	46.30	1.27
9	4.8	not heated	NF	21.15cd	45.79cd	45.75	1.85
10	4.9	120	NF	20.73cd	46.81b	47.20	0.83
11	4.9	130	NF	18.56de	47.42a	48.05	0.40
12	4.9	140	NF	16.90e	47.75a	48.65	0.19

[a] FM (fermentation method): SC = starter culture; not fer. = not fermented; NF = natural flora fermented.
[b] Calculated from duplicate percent yields obtained from 11 weekly readings (77 days of drying); units of rate constant (k), g H_2O/g sausage/day.
[c] Means within the same column having one of the same letters are not significantly different at the 95% confidence level.

fermented by either SC (variables 1–4) or NF (variables 9–12). In another experiment (data not presented), sausages were weighed every 3–4 days for the first 3 weeks of drying, but predicted percent yields were not improved. Though the predicted yields tend to be higher than observed, the data could estimate the amount of product any given production run will give.

When data from studies of other variables (Palumbo et al., 1976b) were analyzed by the rate equation, MS's were comparable to those of Table 1, indicating that the rate equation adequately described the drying of those sausages. Tests of the equation with different data, ours and those of Townsend and Davis (1972) suggested that it could be used to describe the drying of any type of sausage.

By further analysis of our data by the rate equation (Palumbo et al., 1976b), we found that the parameter 0% NaCl had a very high MS, 2.99. Furthermore, sausages that were not fermented, not heated (Table 1) or had 0% NaCl gave very high MS; also they had extremely poor, nontypical texture. Thus, poor irregular drying of sausages produced poor texture in the dry product, and was characterized by a high MS when the data are analyzed by the rate equation. For sausages with a regular drying pattern that develop firm cohesive final texture, either fermentation or heating and at least 1% salt are required.

Diffusivity

The drying of a sausage which we have described by means of the rate equation and yields, also can be described as the movement of moisture from the interior of the sausage to the surrounding environment. By use of the equations of Sherwood (1929) and Newman (1931a, b) for unsteady state diffusion in porous solids (as described by Treybal, 1955), the diffusivity or diffusion coefficient (D) for moisture in pepperoni was calculated.

Percent yield, percent moisture at 85 days, and the ultimate percent yield, as predicted by the rate equation for pork-beef pepperoni prepared with 3 fat levels (Table 3), were used to calculate D_a, defined as the apparent overall diffusivity constant. For those calculations, the sausages were assumed to be cylinders with sealed ends; that assumption was validated by data published by Palumbo et al. (1976b). The diameter of the cylinder was assumed to be the mean of initial and final diameters $(5.5 + 4.1)/2 = 4.8$ cm, or the radius (a) = 2.4 cm.

To calculate D_a, a plot of log E vs θ (time in days) is first required for each pork-beef pepperoni (Fig. 2). In this case,

$$E = \frac{g\ H_2O \text{ at } \theta - g\ H_2O \text{ at } \infty}{g\ H_2O \text{ at zero time} - g\ H_2O \text{ at } \infty}$$

grams H_2O at θ – g H_2O at ∞ is a measure of the amount of water still unremoved; g H_2O at zero time – g H_2O at ∞ is a measure of the amount of water available for removal. Thus, E represents the fraction of available water unremoved. Best fit straight lines were determined by linear regression for the data between 14 and 70 days. The respective correlation coefficients (r) for the 13.3, 17.4 and 25.1% fat pepperoni were –0.996, –0.996, and –0.994, respectively.

At E = 0.1 (i.e., log = –1.0), the respective values of θ for 13.3, 17.4 and 25.1% fat pepperoni were 38.3, 39.0 and 46.6 days (Fig. 2). From Treybal (1955), at E = 0.1, $(D_a\theta)/a^2 = 0.33$, for a cylinder with sealed ends. Thus,

$$D_a = \frac{0.33 \times (2.4\ \text{cm})^2}{38.3\ \text{days} \times 24\ \text{hr/day} \times 3600\ \text{sec/hr}}$$

or $D_a = 5.7 \times 10^{-7}$, 5.6×10^{-7}, and 4.7×10^{-7} cm²/sec, respectively, for moisture in pepperoni containing 13.3%,

Table 2—Predicted and actual 42-day percent yields for sausage variables described in Table 1

Sausage variable no.[a]	Mean % yield at 42 days: Predicted[b]	Actual	Difference
1	48.18	46.05	2.13
2	48.49	47.70	0.79
3	49.87	48.90	1.03
4	50.66	49.60	1.06
5	47.60	46.00	1.60
6	50.26	48.65	1.61
7	51.90	49.45	2.45
8	51.12	49.70	1.42
9	49.54	49.00	0.54
10	52.21	50.20	2.01
11	53.38	51.65	1.73
12	53.38	52.35	1.03

[a] See Table 1 for description of sausage.
[b] Percent yield data for 7, 14 and 21 days of drying were used in the rate equation to predict percent yield at 42 days.

Table 3—Compositional analyses and calculated values for pork-beef pepperoni prepared with 3 starting fat levels

Starting % fat	% moisture at 85 days of drying	Rate constant	Ultimate % yields
13.3	17.5	16.7	44.1
17.4	16.0	15.2	46.7
25.1	15.8	14.0	50.4

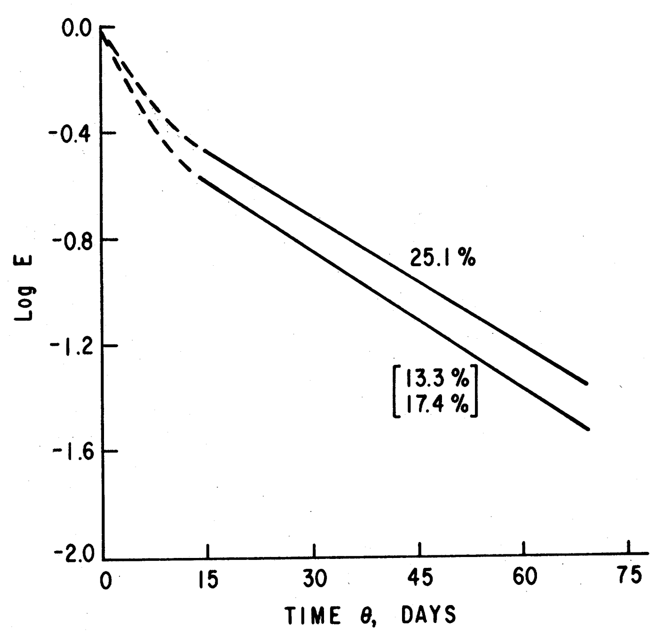

Fig. 2—Influence of percent fat on E [the fraction of water unremoved, (g H_2O at θ – g H_2O at ∞)/(g H_2O at zero time – g H_2O at ∞)] during drying of pork-beef pepperoni.

17.4%, and 25.1% fat. All values for D_a were determined at 12°C, the temperature of the drying room.

Diffusivity for a meat product apparently has not been reported previously, but has been determined for other foods such as potatoes (Saravacos and Charm, 1962). Jason (1958) reported $D_a = 8.3 \times 10^{-7}$ cm²/sec for dogfish, which has relatively high fat content. He also observed that the diffusion coefficient decreased with increasing fat content of the various fish, thus agreeing with our observation of the influence of fat content in sausages.

Frueland (1970) has studied moisture movement in Danish salami by assuming that salami drying followed a capillary flow mechanism. He showed that the conductivity factor (K_w) varied greatly with the moisture content of the sausage. This fact renders the mathematical treatment used by Frueland very difficult especially without a computer and not very practical from the standpoint of a sausage maker.

Drying of foods usually occurs in two phases: the constant rate or initial phase, and the falling rate or final phase (Charm, 1963). Drying proceeds at a constant rate as long as evaporation from the surface controls the rate, then shifts to the falling rate phase when migration of moisture to the surface governs the drying. The data in Figure 2 indicate that internal diffusion of moisture controls the rate of drying and that the plots are essentially straight on this semilogarithmic graph at values of the ordinate below −0.6 as predicted by diffusion theory. For diffusion-controlled drying, the drying time between fixed moisture contents should be proportional to the square of the thickness of product. We could not test this hypothesis however, because drying data were available for only one thickness of sausage. At the dyring conditions used, the initial phase of drying was short and apparently was followed by a short period of unsaturated surface evaporation. With sausages, the diffusion-controlled falling-rate period may be considered to begin almost immediately after drying starts. The percent yields at 70 days for 13.3, 17.4, and 25.1% fat pepperoni were 45.8, 48.2, and 52.3, respectively and are within 96% of the respective predicted ultimate percent yields (Table 3) for those sausages.

The drying of sausages is a diffusion-limited process, and air velocity should have no effect. Townsend et al. (1975), however, have reported small (ca 1%), but statistically significant increases in the percent shrink of sausages dried in high air velocities compared with sausages dried in low air velocities. Their finding can be explained by the fact that they studied only the early portion of the drying period, i.e., the initial phase. In that phase, evaporation from the surface controls the rate, so that high velocities can increase the percent shrink simply by faster removal of moisture from the surface. Since the small increase in percent shrink was accomplished by more than tripling the air velocity (from 35 to 120 ft/min), the practical significance of their finding is limited. Air velocity must be high enough to prevent mold growth, but not high enough to cause case hardening. Further, the relative humidity of the air in the drying room influences the drying process primarily through its effect on the equilibrium moisture content of the sausages. We have observed that the equilibrium relative humidity (water activity × 100) of pepperoni held in the drying room for extended periods, ca 90 days, approximated the relative humidity of the drying room.

Jason (1958) showed that diffusion is temperature-dependent and even calculated the energy of activation (E_a) for the process in fish. We determined D_a at 12°C, but could determine D_a at other temperatures and then calculate E_a for diffusion of moisture in sausage. When E_a is known, D_a at any temperature can be determined. Thus, by incorporating the proper dimensional term into the diffusion equation, the moisture level (percent yield) or degree of dryness of any size sausage at any stage of drying at any temperature could be calculated.

When the equilibrium moisture content (or % yield) of a sausage is known, as well as its apparent overall diffusivity D_a, the diffusion equation may be readily used to predict weight loss with time. This, of course, is only feasible with sausages of a set formulation. However, the use of the diffusion equation along with Raoult's Law offers a practical tool for estimating % yield and predicting drying behavior for sausages with different formulations. Using Raoult's Law, or the equations devised by Ross (1975), it is possible to estimate the water activity, and, therefore, the % yield for any sausage knowing its final composition after a drying time long enough to establish equilibrium with the surrounding air. The apparent overall diffusivity of a sausage formulation may be predicted by assuming that the contribution of each nonfat constituent of the sausage to the total apparent overall diffusivity is proportional to the concentration of each constituent, i.e., $D_a = \sum_i^n w_i D_i$, where w_i = weight of ingredient i divided by the total weight of nonfat solids.

The fat is hydrophobic and offers resistance to diffusion. A refined method of treating diffusion in the presence of a constraining solid structure involves defining a "pore shape factor" K which provides a measure of the true length of the diffusion path (Treybal, 1955). The mathematical procedures used above are then considered to yield values of

$$\frac{\sum_i^n w_i D_i,}{K^2}$$

where $\sum_i^n w_i D_i$ is the apparent overall diffusivity in the absence of fat and K is a factor that measures the resistance offered by fat solids. Fat added to sausage increases its size (per unit weight of nonfat solids). If the sausage is cylindrical, its radius increases as the factor $(V_1/V_2)^{1/2}$, where V_1 = total volume of all constituents of the sausage, and V_2 = volume of nonfat constituents. The increase in the length of path traversed by the water due to the addition of fat is also approximately equal to $(V_1/V_2)^{1/2}$. Heating disperses the fat, further increasing the path traversed by the water by a factor h. The total contribution of fat to the "pore shape factor" K defined above is thus $(V_1/V_2)^{1/2}h$. In other words, the values for D_a reported previously are actually equal to $(\sum_i^n w_i D_i)/(V_1/V_2)h^2$ where $\sum_i^n w_i D_i$ is the apparent overall diffusivity of moisture in the absence of fat constituents, $(\sum_i^n w_i D_i)/(V_1/V_2)$ is the apparent overall diffusivity of moisture in a sausage containing unheated fat, and $(\sum_i^n w_i D_i)/(V_1/V_2)h^2$ is the apparent overall diffusivity of moisture in a sausage which has been heated before drying. It would be expected that h values would be different for sausages heated to different temperatures prior to drying due to increased dispersion of fat with increased temperature.

Ignoring differences in density between fat and the other sausage ingredients, $V_1/V_2 \approx 100/N$, where N = percentage of nonfat ingredients in the sausage. We calculated $\sum_i^n w_i D_i$ values by multiplying experimental $D_a = (\sum_i^n w_i D_i)/V_1/V_2)$ values for sausages containing three different fat levels in which the fat had not been heated prior to drying by the appropriate 100/N values. These corrected apparent overall diffusivities, $\sum_i^n w_i D_i$, in the absence of fat were 6.6×10^{-7}, 6.8×10^{-7}, and 6.3×10^{-7} cm²/sec for fat levels 13.3, 17.4, and 25.1% respectively. The constancy of these values shows that in the absence of fat, the apparent overall diffusivity is ca 6.6×10^{-7} cm²/sec, and that the effect of different amounts of fat on diffusivity can be readily calculated.

Fat addition has the same effect on the rate constants as on the diffusion constants: the rate constants decrease as the fat content increases (Table 3). For example, the k value for 25.1% fat content pepperoni can be predicted from the k = 16.7 value (Table 3) obtained at 13.3% fat content by multiplying it by the factor (100 – 25.1)/(100 – 13.3). Thus 16.7 (100 – 25.1)/(100 – 13.3) = 14.4, which compares favorably with the value of 14.0 (Table 3) obtained from the rate equation.

The percent yield after a long dehydration time (equilibrium moisture content) can also be predicted for sausages with different fat levels as long as the sausages have not been heated. That prediction is readily demonstrated by calculating yield for a sausage containing 25.1% fat from the yield (44.1%) for a sausage with 13.3% fat content (Table 3). On a basis of 100g of sausage, the initial content of nonfat ingredients in the 13.3% fat sausage was 100 – 13.3 = 86.7g, the amount of moisture removed during prolonged drying was 100 – 44.1 = 55.9g, and the amount of nonfat solids plus bound moisture present in the sausage at equilibrium was 44.1 – 13.3 = 30.8g. A sample (100g) of a sausage containing 25.1% fat would initially have 100 – 25.1 = 74.9g of nonfat solids plus moisture. If in the first case, 86.7g of mixture yielded 30.8g of nonfat solids plus bound moisture at equilibrium, 82.6g in the second case should yield 30.8 (74.9/86.7) = 26.6. If to these 26.6g of nonfat solids plus bound moisture one adds the fat, which is equal to 25.1g, the final total equilibrium weight of the sausage is: 26.6 + 25.1 = 51.7g. That weight (yield) compares reasonably well with the experimental value of 50.4% (Table 3). If the sausages have been heated, however, such calculations are not valid since after heating, moisture content at equilibrium was reduced further. Heating melts the fat, which in turn spreads throughout the sausage, and, being hydrophobic, apparently changes the moisture adsorption characteristics of the internal surfaces. Different degrees of heating effect different changes within the sausage; consequently, the equilibrium moisture content or the diffusivity cannot be predicted on sausages that have been subjected to different heat treatments.

Before heating, fat and meat particles are separate entities, but during heating, fat enters some capillaries inside meat particles. The high MS (and SS) values calculated from experimental data for nonheated sausages probably are associated with nonuniform dispersion of the ingredients, especially fat among batches and among sausages. Heating increases homogeneity of the mixture as melted fat is redistributed throughout the sausage, and, consequently, MS and SS values are lowered.

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Ms received 9/26/76; revised 11/30/76; accepted 12/8/76.

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