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# JOURNAL OF FOOD PROCESS ENGINEERING

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and
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FOOD & NUTRITION PRESS, INC.

VOLUME 13, NUMBER 4 QUARTERLY

**JOURNAL OF FOOD PROCESS ENGINEERING**



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All subscriptions and inquiries regarding subscriptions should be sent to Food & Nutrition Press, Inc., 6527 Main Street, P.O. Box 374, Trumbull, CT 06611 USA.

One volume of four issues will be published annually. The price for Volume 13 is $98.00 which includes postage to U.S., Canada, and Mexico. Subscriptions to other countries are $115.00 per year via surface mail, and $124.00 per year via airmail.

Subscriptions for individuals for their own personal use are $78.00 for Volume 13 which includes postage to U.S., Canada, and Mexico. Personal subscriptions to other countries are $95.00 per year via surface mail, and $104.00 per year via airmail. Subscriptions for individuals should be sent direct to the publisher and marked for personal use.

The *Journal of Food Process Engineering* (ISSN: 0145-8876) is published quarterly (March, June, September and December) by Food & Nutrition Press, Inc.—Office of Publication is 6527 Main Street, P.O. Box 374, Trumbull, Connecticut 06611 USA. (Current issue is February, 1991.)

Second class postage paid at Bridgeport, CT 06602.

POSTMASTER: Send address changes to Food & Nutrition Press, Inc., 6527 Main Street, P.O. Box 374, Trumbull, CT 06611.

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FOOD & NUTRITION PRESS, INC.
TRUMBULL, CONNECTICUT 06611 USA

ISSN 0145-8876

Printed in the United States of America

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# ON BALL'S FORMULA METHOD FOR THERMAL PROCESS CALCULATIONS

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Accepted for Publication October 2, 1990

## ABSTRACT

*Discrepancies between Ball's equation and his tabulated values associated with his original formula method for thermal process calculations had initially led to arguments for the validity of the equation and finally to revised sets of tables. Here, the validity of Ball's tables (within the accuracy of graphical integration) is established. A typographical error associated with Ball's published equation was identified and the correct form is presented.*

## INTRODUCTION

The fundamental equation used by Ball to derive the tables and graphs associated with his original formula method of thermal process calculations is given by Eq. (1) as it first appeared in 1923 (Ball 1923, Eq. (17)).

$$A = \frac{f_{he}}{t_{T_g}} \{e^{g/ze} [Ei(-\frac{80}{z_e}) - Ei(-\frac{g}{z_e})] + [0.33172\, e^{-0.343m/z_e} + 0.5833 \frac{z_e}{m} e^{0.300m/z_e} E] + e^{-m/z_e} [Ei(\frac{0.657m}{z_e}) - Ei(\frac{m+g-80}{z_e})]\} \quad (1)$$

In Eq. (1) $f_{he}$ and $z_e$ are the $f_h$ and z values, respectively, based on Napierian logarithms, that is

$$f_{he} = f_h/\ln 10 \quad \text{or} \quad f_{he} = f_h/2.303 \quad (2)$$

and

$$z_e = z/2.303 \quad (3)$$

Journal of Food Process Engineering *13(1991) 255–268. All Rights Reserved.*

A is defined as the total lethality (heating and cooling) of a thermal process, and $t_{T_g}$ is the time required to destroy a given percentage of microorganisms at the highest temperature attained by the center of the can during a thermal process. Hence,

$$t_{T_g} = F^z_{T_{ref}} \, 10^{\frac{T_{ref}-T_g}{z}} = F^z_{T_{ref}} \, 10^{\frac{T_{ref}-T_{RT}}{z}} \, 10^{\frac{T_{RT}-T_g}{z}}$$

or

$$t_{T_g} = F \, F_i \, 10^{g/z} \qquad \text{or} \qquad t_{T_g} = U \, e^{2.303g/z} \tag{4}$$

The definition for the exponential integral, Ei, Ball used was (Ball 1923)

$$Ei(x) = \int_{\infty}^{-x} \frac{e^{-u}}{u} \, du \tag{5}$$

and in deriving Eq. (1) he made use of the following relationships

$$Ei(x) = \int_{-\infty}^{x} \frac{e^{u}}{u} \, du$$

and

$$Ei(-x) = \int_{\infty}^{x} \frac{e^{-u}}{u} \, du \tag{6}$$

which are equivalent for $x > 0$ (Gautschi and Cahill 1964).

In more recent publications dealing with Ball's formula method (Merson *et al.* 1978; Steele and Board 1979) the exponential integral $E_1(x)$ is used in place of $Ei(-x)$ through the following relationship

$$-Ei(-x) = E_1(x) \tag{7}$$

for $E_1(x)$ defined as (Gautschi and Cahill 1964)

$$E_1(x) = \int_x^{\infty} \frac{e^{-u}}{u}\, du \qquad (x>0) \qquad (8)$$

The quantity E appearing in Eq. (1) is an integral, graphically evaluated by Ball (1923), given by (Ball 1923; Ball and Olson 1957)

$$E = \int_{0.3m/z_e}^{0.643m/z_e} e^{-u} \sqrt{u^2 - (0.3m/z_e)^2}\, du \qquad (9)$$

From the above equations it is clear that for given z and m+g values, in order to achieve the desired degree of commercial sterility (i.e., A=1), $f_h/U$ becomes solely a function of g. Ball published $f_h/U$ versus g (and C versus g -C will be defined later) relationships for various m+g and z values in graphical or tabular form in 1923, 1928, and in Ball and Olson (1957). In all these publications, the tables and graphs under consideration were identical, and all entries referred to $f_h$ and z values were based on logarithms with base 10 rather than the Napierian logarithms initially used in deriving Eq. (1). However, the fundamental equation (i.e., Eq. (1)) was not the same in the 1923 and the 1957 publications.

Each one of the three terms in brackets in the right hand side of Eq. (1) is associated with the lethality during the logarithmic heating, the initial portion of cooling, and the final-logarithmic-part of cooling, respectively. In Ball and Olson (1957), the lethality of the heating appeared with an opposite sign compared to the corresponding term in Eq. (1). Due to the similarity of the nature of the logarithmic heating and cooling and of the way they are represented as the first and third right hand side terms in Eq. (1), this negative sign in the first term originated some discussion. Initially, Flambert *et al.* (1977) argued that the lethality during the logarithmic cooling was not appropriately represented by Ball and Olson (1957). However, Steele and Board in 1979 reestablished Ball and Olson's (1957) representation of the logarithmic cooling lethality, and they first noticed the negative sign in the lethality term during the logarithmic heating. As they pointed out, this was a result of an incorrect definition used for the exponential integral, Ei(−x), (Eq. (12.12) in Ball and Olson 1957). The use of the negative sign in the term for the heating lethality could result in unrealistic $f_h/U$ versus g values. However, the equation, as it was presented in 1957 (Eq. 12.59), was never used to create the $f_h/U$ versus g tables presented in the 1957 book. As it was mentioned earlier, the tables presented by Ball and Olson in 1957 were identical to the ones initially presented by Ball in 1923.

In a later publication, another argument arose by Steele *et al.* (1979). Comparing values generated through the use of Ball's equation (Eq. (1)) with tabulated values, they found that the tables published by Ball and Olson (1957) could result in up to 26% overestimation of process lethality as it compared to lethality evaluated by the equation. Due to the wide use of Ball's tables, revised tables to agree with Ball's equation were presented (Steele *et al.* 1979).

There is no argument that Ball's tables are not consistent with his equation. Smith and Tung (1982), comparing various formula methods for calculating thermal process lethality, reported considerable differences between Ball's equation and Ball's tables with the latter being in closer agreement with a "numerical general method" used as the reference method. The objective of this paper is to show that the tabulated values are the correct ones (within the limits of graphical integration) and not the equation as it is presented here by Eq. (1). Or, in other words, the values that Ball presented as a set of tables and graphs (1923, 1928, and Ball and Olson 1957), were obtained from an equation "slightly" different than the equation under investigation.

## BALL'S FORMULA METHOD

The intention of this paper is not to evaluate Ball's formula method per se. Discussion on its validity or its limitations can be found elsewhere (Merson *et al.* 1978). However, some background information is included so that the basic principles involved and the implications of using Ball's equation instead of the tables can be understood.

In order for Ball to establish his original formula method for thermal process calculations, he first attempted to describe the temperature history of the center of the can undergoing thermal processing by simple equations in terms of product characteristics and processing parameters. Ignoring the initial lag portion of the heating curve, as contributing negligible lethality to the process, Ball treated the heating curve, as well as the latest portion of the cooling curve, with simple logarithmic temperature drop relationships. For example, for the cooling curve, he assumed that can center temperature, T, is given by

$$T - T_{CW} = j_c \, (T_g - T_{CW}) \, e^{-2.303t/f_c} \qquad (10)$$

where $f_c$ is based on logarithmic plots with base 10, and t measures time from the axis where $j_c$ is defined.

For the initial portion of cooling, Ball assumed that the temperature of the can center can be expressed through a hyperbola given by the following equation.

$$\frac{y^2}{a^2} - \frac{t^2}{b^2} = 1 \qquad (11)$$

Time equal zero was assumed at the end of heating, while the $y=0$ axis was taken at "a" degrees higher than the can center temperature at the end of heating; that is, where the asymptotes of the hyperbola (given by the equation: $y=\pm at/b$) intersect the vertical line at the end of heating (Fig. 1). Hence,

$$y = T_g + a - T \tag{12}$$

Based on experimental observations, Ball proceeded to fix the shape of the cooling curve using the cooling medium temperature and information only from the heating portion of the process. For this, he first fixed the "straight" line portion of the cooling curve (i.e., the part given by Eq. (10)) by assuming $f_c = f_h$ and $j_c = 1.41$. Then he located the $y=0$ axis by setting $a = 0.3(T_g - T_{CW})$. Finally, he fixed the temperature at point A (Fig. 1), that is, the temperature after which the semi-log curve (Eq. (10)) applies, by locating point A at $0.343(T_g - T_{CW})$ degrees F below $T_g$. The value of b appearing in Eq. (11) was determined by requiring that both hyperbola and semi-log curve at point A coincide. Equating the times from Eq. (10) and (11) corresponding to the temperature at A, i.e., $T_A = T_g - 0.343(T_g - T_{CW})$, we obtain

from Eq. (10):
$$t_A = \frac{f_h}{2.303} \ln\left(j_c \frac{T_g - T_{CW}}{T_A - T_{CW}}\right)$$

or substituting for $T_A$,

$$t_A = \frac{f_h}{2.303} \ln\left(j_c \frac{1}{1-0.343}\right) \tag{13}$$

and from Eq. (11):
$$t_A = b\sqrt{\left(\frac{y_A}{a}\right)^2 - 1}$$

and substituting for $y_A$ (Eq. (12)) and a,

$$t_A = b\sqrt{\left(\frac{0.3+0.343}{0.3}\right)^2 - 1} \tag{14}$$

Equating $t_A$ from Eq. (13) and (14), and setting $j_c = 1.41$, we obtain

$$b = 0.175 f_h \tag{15}$$

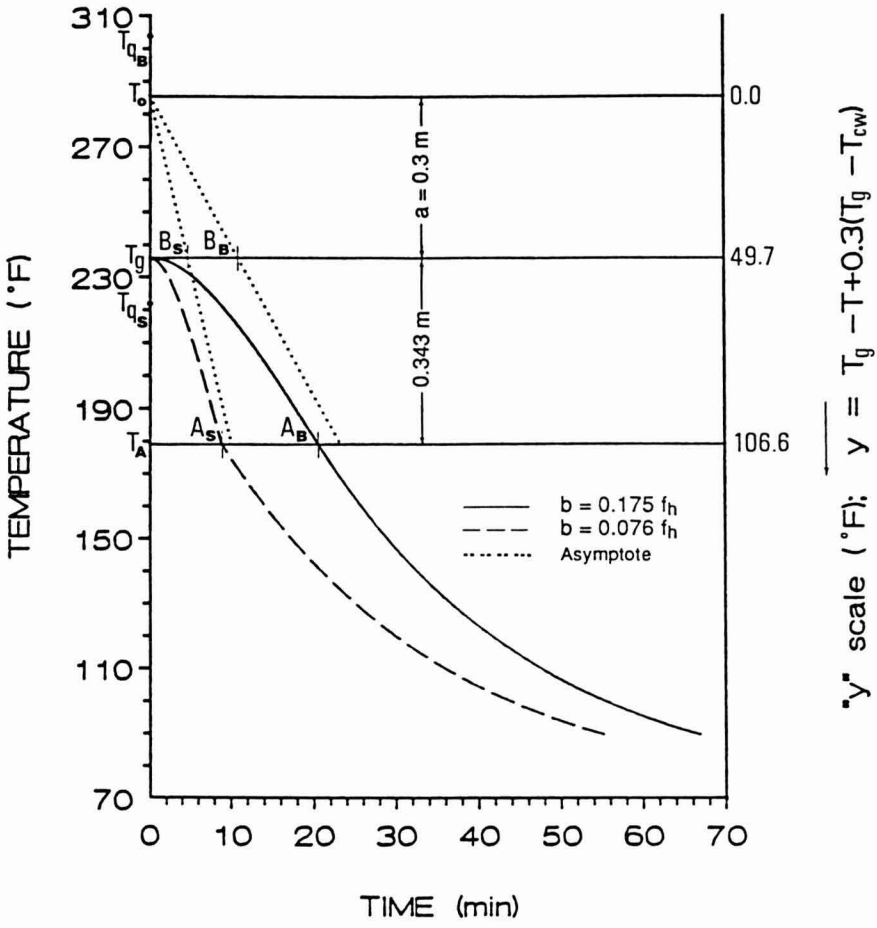


FIG. 1. HYPERBOLIC AND "SEMI-LOG" COOLING CURVES FOR $b=0.175f_h$ AND $b=0.076f_h$. Data refer to a hypothetical process with $T_{RT}=250\,°F$, $T_{IT}=T_{CW}=70\,°F$, $j_h=1.41$, $f_h=f_c=62$ min, and $g=14.29\,°F$.

**Summary of definitions and variables:** For the following definitions subscripts B and S refer to $b=0.175f_h$ ($j_c=1.41$) and $b=0.076f_h$ ($j_c=0.9155$) cases, respectively. $b_B=t_{B_B}=0.175f_h=10.85$ min, $b_S=t_{B_S}=0.076f_h=4.71$ min; $T_A=T_g-0.343(T_g-T_{CW})=178.87\,°F$; $t_A=1.8958b$, $t_{A_B}=0.3318f_h=20.56$ min, $t_{A_S}=0.1441f_h=8.93$ min; $T_q=j_c(T_g-T_{CW})+T_{CW}$, $T_{q_B}=303.65\,°F$, $T_{q_S}=221.71\,°F$; $T_o=(T \text{ for } y=0)=285.42\,°F$; $a=T_o-T_g=0.3(T_g-T_{CW})=49.71\,°F$; $T_g-T_A=0.343(T_g-T_{CW})=56.84$; Equation for the asymptote: $y=(a/b)t$.

Note in the above relationship the value of $f_h$ is taken from logarithmic plots with base 10. If the $f_{he}$ value is used, then, Eq. (15) results in $b=0.403f_{he}$. In presenting his equation, Ball (1923) implied that $b=0.175f_{he}$, an error that appeared again in Ball and Olson (1957) where b is defined as: $b=0.175f_{he}=0.0759f_h$.

In arriving at Eq. (1), Ball proceeded to calculate process lethality by integrating the temperature effects on lethality over each process period through the following equation

$$\text{Lethality} = \frac{1}{F}\int_{t_a}^{t_b} \frac{dt}{10^{(T_{ref}-T(t))/z}} \tag{16}$$

Obviously, the error in the b value propagated and affected the expression for the lethality during the initial portion of the cooling curve. Through the use of Eq. (11) and Eq. (16), Ball (1923) obtained the following relationship for the lethality of the initial portion of the cooling process

$$(\text{Lethality})_{\text{cooling lag}} = \frac{z_e b e^{a/z_e}}{a t_{T_g}} \int_{a/z_e}^{(a+0.343m)/z_e} \frac{u\,du}{e^u \sqrt{u^2-(a/z_e)^2}} \tag{17}$$

A similar expression is given by Ball and Olson (1957, Eq. (12.30)). It is obvious that the value of b proportionally affects the value of the lethality during the cooling lag. For $b=0.403f_{he}$ expression (17) results to (according to the steps outlined by Ball 1923)

$$(\text{Lethality})_{\text{cooling lag}} = \frac{2.303\, f_{he}}{t_{T_g}} \left[0.33172\, e^{-0.343m/z_e} + 0.5833\, \frac{z_e}{m} e^{0.300m/z_e} E\right] \tag{18}$$

Replacing the second term in the right hand side of Eq. (1) with the expression given by Eq. (18), substituting $f_h$ and z for $f_{he}$ and $z_e$ through Eq. (2) and (3), expressing $t_{T_g}$ in terms of U by Eq. (4), using $E_1(x)$ instead of $Ei(-x)$, i.e., Eq. (7), and setting $A=1$, then from Eq. (1) we obtain

$$\frac{f_h}{U} = \frac{e^{2.303g/z}}{C} \tag{19}$$

where C is defined by

$$C = \frac{1}{2.303}\left\{e^{2.303g/z}\left[E_1\left(\frac{2.303g}{z}\right) - E_1\left(\frac{2.303\cdot 80}{z}\right)\right] + \left[0.7638\, e^{-0.789m/z} + 0.5833\, \frac{z}{m} e^{0.692m/z} E\right] + e^{-2.303m/z}\left[Ei\left(\frac{2.303}{z} 0.657m\right) - Ei\left(\frac{2.303}{z}(m+g-80)\right)\right]\right\} \tag{20}$$

Coefficients appearing in Eq. (20) were rounded off and presented here in the same manner as they appeared in Ball and Olson (1957, Eq. 12.59). The difference between Eq. (20) and the corresponding one, as it was presented by Ball and used by Steele *et al.* (1979), is in the values of the coefficients in the second term in the right hand side of Eq. (20). In Eq. (20), the values of 0.7638 and 0.5833 were used, while Ball presented the equation (Ball and Olson 1957, Eq. (12.59)) with the values of 0.332 and 0.253, respectively. For the remainder of this paper C values obtained through Eq. (20) will be designated by $C_{20}$ while C values obtained by using the 0.332 and 0.253 coefficients will be designated with $C_s$. Note that since E, as it is defined by Eq. (9), is always positive, $C_{20}$ is greater than $C_s$. In addition, note that $C_s$'s are the C values reported by Steele *et al.* (1979), which were consistently lower than the C values reported by Ball (Ball and Olson 1957, Table 12.1).

## RESULTS AND DISCUSSION

Eliminating the integral E between Eq. (20) and the corresponding equation for $C_s$, the following relationship is obtained

$$C_{20} = 2.303\,C_s - 0.5657\,(C_h + C_{cl}) \tag{21}$$

where

$$C_h = e^{2.303g/z}\left[E_1\left(\frac{2.303g}{z}\right) - E_1\left(\frac{2.303\cdot 80}{z}\right)\right] \tag{22}$$

and

$$C_{cl} = e^{-2.303m/z}\left[Ei\left(\frac{2.303}{z}0.657m\right) - Ei\left(\frac{2.303}{z}(m+g-80)\right)\right] \tag{23}$$

Using Eq. (21), $C_{20}$ values were calculated for selected m+g, g, and z values. $C_s$ values were obtained from Steele *et al.* (1979). To evaluate $C_h$ and $C_{cl}$ through Eq. (22) and (23), the same approximations for the exponential integrals used by Steele *et al.* (1979) were used (Gautschi and Cahill 1964):
For $0 < x \leq 1$, with an error of less than $2\cdot 10^{-7}$,

$$E_1(x) = -\ln x - 0.57721566 + 0.99999193x - 0.24991055x^2 + 0.5519968x^3 - 0.00976004x^4 + 0.00107857x^5 \tag{24}$$

for $1 < x < \infty$ with an error of less than $5\cdot 10^{-5}$,

$$E_1(x) = e^{-x}\,\frac{(x^2 + 2.334733x + 0.250621)}{(x^3 + 3.330657x + 1.681534)} \tag{25}$$

and

$$Ei(x) = \gamma + \ln x + \sum_{n=1}^{\infty} \frac{x^n}{n\,n!} \tag{26}$$

for $\gamma = 0.57721566$, and the series being evaluated until the $n^{th}$ term was less than 0.00001.

The results from these calculations are presented in Table 1 together with C values reported by Ball (Ball and Olson 1957, Table 12.1). Differences between $C_s$ (Steele *et al.* 1979) and calculated $C_{20}$ values were as high as 30%. As it is expected, differences are higher when the lethality accumulated during the initial part of the cooling is a high percentage of the total lethality; that is, at lower m+g and/or higher g values. Comparing the $C_{20}$ with the C values reported by Ball (Table 1), differences up to about only 5% were observed, with the $C_{20}$ values being consistently higher. This means that process lethalities evaluated using Ball's C values are lower than those obtained through the $C_{20}$ values. That is, a safety factor is incorporated if processes are designed using Ball's tables. Considering the accuracy of graphical integration (Ball 1960) and the tendency Ball had (in our opinion) to include an extra safety factor in every step of his derivations or calculations, we believe that Ball actually used the correct coefficients, as in Eq. (20), to calculate C (and thereafter $f_h/U$ versus g values) although, probably due to a typographical error, the incorrect coefficient, associated with the b value and propagated throughout the development of Eq. (1), appeared in his publications. Clearly, tabulated values presented by Ball were not obtained through the use of Eq. (1).

Another indication about the validity of Ball's tables (within the limitations of Ball's formula method) versus the equation, as it is presented here by Eq. (1), follows: Time-temperature data were created for a hypothetical process under the following conditions: $T_{RT}=250\,°F$, $T_{IT}=T_{CW}=70\,°CF$, $j_h=1.41$, and $f_h=f_c=62$ min. For the heating portion of the process, temperature data were obtained through an equation similar to Eq. (10), that is, assuming "straight" line heating, up to either $g=1.987$ or $g=14.29\,°F$. After the time corresponding to the specific g value was reached, temperatures were calculated assuming the equation of the hyperbola that Ball used (Eq. (11)), which when solved for T results to

$$T = 0.3\,(T_g - T_{CW})\,[\,1 - \sqrt{1 + (t/b)^2}\,] + T_g \tag{27}$$

Equation (27) was used until T reached the $T_A$ value ($T_A=T_g-0.343\,(T_g-T_{CW})$, as defined by Ball), and thereafter, the "straight" line cooling was assumed (Eq. (10) with $j_c(T_g-T_{CW})$ being replaced by $T_A-T_{CW}$, and time, t,

TABLE 1.
SELECTED C VALUES OBTAINED WITH VARIOUS METHODS

| m+g (°F) | g (°F) | z (°F) | $C_s$ | $C_{20}$ | C by Ball |
|---|---|---|---|---|---|
| 180 | 1.0 | 18 | 0.8295 | 0.8794 | 0.8589 |
| 160 | 20.0 | 18 | 0.1733 | 0.2304 | 0.2231 |
| 150 | 10.0 | 18 | 0.2622 | 0.3192 | 0.3167 |
| 130 | 25.0 | 18 | 0.1600 | 0.2271 | 0.2176 |
| 130 | 25.0 | 26 | 0.2122 | 0.2967 | 0.2814 |

measured from $t_A$). For lethality calculations, end of cooling was assumed when T reached the value of $T_{RT}-80$. The data so created were in complete agreement with all Ball's assumptions used in deriving his formula method as long as b was properly defined.

For each of the above g values, two data sets were created; one corresponding to $b=0.175f_h$, and the other to $b=0.076f_h$, that is, $b=0.175f_{he}$. Time-temperature data for both b values and for $g=14.29\,°F$ are shown in Fig. 1. To calculate the resulting F values of the processes, a general method scheme, with $z=18\,°F$ was employed. F values were also calculated using the $f_h/U$ versus g tables presented by Ball (Ball and Olson 1957, Table 12.2) and by Steele *et al.* (1979). The results are summarized in Table 2. For purposes of comparison, F values were also calculated using the tables presented by Stumbo (1973) for two $j_c$ values; $j_c=1.40$ and $j_c=0.9155$. The latter value was chosen because the use of Ball's equation (Eq. (1)) or the tables by Steele *et al.* (1979) it actually implies $j_c=0.9155$ instead of 1.41.

TABLE 2.
F VALUES (MIN) OF THE PROCESS CALCULATED WITH VARIOUS METHODS

| | g=1.987°F | g=14.29°F |
|---|---|---|
| General Method with $b=0.175f_h$ | 32.03 | 2.59 |
| Ball's Tables | 31.00 | 2.48 |
| Stumbo's Tables for $j_c=1.40$ | 33.31 | 3.01 |
| General Method with $b=0.076f_h$ | 29.62 | 2.07 |
| "Steele's" Tables | 29.98 | 2.16 |
| Stumbo's Tables for $j_c=0.9155$ | 29.69 | 2.00 |

TABLE 3.
F VALUES (MIN) FOR EACH PORTION OF THE PROCESS CALCULATED BY THE GENERAL METHOD

| | g=1.987°F | | g=14.29°F | |
|---|---|---|---|---|
| | $b=0.175f_h$ | $b=0.076f_h$ | $b=0.175f_h$ | $b=0.076f_h$ |
| Logarithmic Heating | 27.772 | 27.772 | 1.675 | 1.675 |
| Initial Cooling | 4.255 | 1.848 | 0.917 | 0.398 |
| Logarithmic Cooling | 0.001 | 0.001 | 0.000 | 0.000 |

Eliminating $t_A$ between Eq. (13) and (14) the following relationship between $j_c$ and b is obtained (Larkin 1989)

$$j_c = 0.657 e^{4.365 b/f_h} \tag{28}$$

which, for $b=0.076f_h$, yields $j_c=0.9155$.

From Table 2 it can be seen that results obtained using Ball's tables are in agreement (within the accuracy of the C values) with General Method data for $b=0.175f_h$ (i.e., $j=1.41$). On the other hand, results obtained using Steele's *et al.* (1979) tables correspond to General Method data with $b=0.076f_h$ ($j_c=0.9155$). As it can be inferred from the discussion made earlier concerning the C values, differences in the F values between Ball's and Steele's *et al.* tables are more noticeable for the larger g value.

The effect of b on the F values, can be seen better from Table 3 where F values calculated by the General Method for each portion of the process are presented. As it is expected (Eq. (17)), for a given g value, the ratio of the F values achieved during the initial cooling for the different b values is simply the ratio of the corresponding b values.

Ending our discussion, we want to mention that the better agreement, appearing in Table 2, between the General Method and Stumbo's tables for $j_c=0.9155$ compared to that for $j_c=1.40$ is believed to be coincidental. The hyperbolic shape assumed by Ball for the initial part of cooling during the thermal sterilization process was based on experimental observations, and it is very closely associated with the three variables as they have been defined by Ball (i.e., $a=0.3(T_g - T_{CW})$, $T_A=T_g-0.343(T_g - T_{CW})$, and $j_c=1.41$). Ball's derivation can not be extended for different $j_c$ values assuming the same relationships for a and $T_A$. For example, for these conditions, as Eq. (28) shows, $j_c$ can never be less than 0.657.

## SUMMARY AND CONCLUSION

The validity of Ball's tables versus his equation was presented. Based on Ball's analysis, the equation thought to have been used by Ball to establish his tables, associated with his original formula method for thermal process calculations, was derived. The agreement between Ball's tables and the assumptions led to these tables was demonstrated. Revised tables presented in the literature, to agree with Ball's equation as he had published it, were shown to be related to different process conditions without any theoretical or experimental basis.

## NOMENCLATURE

| | |
|---|---|
| A | total process lethality given by Eq. (1), expressed also as the ratio of the process F value over the required F value, dimensionless |
| a | parameter in hyperbola, Eq. (11), $a=0.3(T_g-T_{CW})$, °F |
| b | parameter in hyperbola, Eq. (11), min |
| C, $C_{20}$ | dimensionless variables defined by Eq. (20) |
| $C_s$ | the C value obtained by replacing the 0.7628 and 0.5833 coefficients in Eq. (20) by 0.332 and 0.253, respectively |
| $C_h$, $C_{cl}$ | dimensionless variables defined by Eq. (22) and (23), respectively |
| E | numerically evaluated integral defined by Eq. (9) |
| Ei | exponential integral defined by Eq. (5) |
| $E_1$ | exponential integral defined by Eq. (8) |
| $F^z_{T_{ref}}$ | time at a reference temperature, $T_{ref}$, required to destroy a given percentage of microorganisms whose thermal resistance is characterized by z; noted here also simply as F |
| F | time required to destroy a given percentage of microorganisms at a given temperature, min |
| $F_i$ | dimensionless factor, which when multiplied by $F^z_{T_{ref}}$ gives the F value at retort temperature, $F_i=10^{(T_{ref}-T_{RT})/z}$ |
| f | time required for the difference between medium temperature and food temperature to traverse a logarithmic cycle, min |
| $f_c$ | f value in a cooling process obtained from a base 10 logarithmic plot |
| $f_h$ | f value in a heating process obtained from a base 10 logarithmic plot |
| $f_{he}$ | f value in a heating process obtained from a Napierian logarithmic plot, related to $f_h$ by Eq. (2) |
| g | difference between retort temperature and maximum temperature attained at the center of a can during its processing, $g=T_{RT}-T_g$, °F |

| | |
|---|---|
| $j$ | dimensionless coefficient defined as $j = (T_m - T_{oe})/(T_m - T_o)$, where $T_m$ is the constant medium temperature of a process, $T_o$ the initial temperature of the center of the can, and $T_{oe}$ the extrapolated initial can center temperature, obtained by assuming an exponential function for the entire processing curve, and defined on $t=0$ or another specified time axis. Note, for j to be completely defined, the time at which $T_{oe}$ is defined must be specified |
| $j_c$ | j value for a cooling curve defined at the beginning of cooling |
| $j_h$ | j value for a heating curve defined at the beginning of heating |
| $m$ | difference between cooling water temperature and maximum temperature attained at the center of a can during its processing, $m = T_g - T_{CW}$, °F |
| $T$ | temperature at the center of the can, °F |
| $T_A$ | temperature at the can center at point A, i.e., where the hyperbola and the semi-log portion of the cooling curve coincide |
| $T_{CW}$ | temperature of cooling water |
| $T_g$ | maximum temperature attained at the can center during processing |
| $T_{IT}$ | initial can center temperature |
| $T_{ref}$ | reference temperature |
| $T_{RT}$ | retort temperature |
| $t$ | time, min |
| $t_A$ | time at point A, i.e., where the hyperbola and the semi-log portion of the cooling curve coincide |
| $t_a$ | time at the beginning of a process |
| $t_b$ | time at the end of a process |
| $t_{T_g}$ | F value at $T_g$; Ball (1923) used the symbol t |
| $U^g$ | F value at $T_{RT}$, $U = F^z_{T_{ref}} F_i$ |
| $y$ | dependent variable in hyperbola, Eq. (11), defined by Eq. (12), °F |
| $y_A$ | y value at point A, i.e., where the hyperbola and the semi-log portion of the cooling curve coincide |
| $z$ | temperature interval required for the time required to destroy a certain percentage of microorganisms to traverse a logarithmic cycle, °F |
| $z_e$ | z value obtained from a Napierian logarithmic plot |

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# THERMAL PROCESS TIME CALCULATIONS FOR THIN PROFILE PACKAGES: COMPARISON OF FORMULA METHODS

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Accepted for Publication October 23, 1990

## ABSTRACT

*The process time calculation accuracy of five formula methods were evaluated in relation to predictions from a computer model based on finite difference numerical solution to three-dimensional heat conduction equations with finite surface convection as applicable to processing of packaged foods in thin profile forms. Heat penetration data were obtained from the computer model for a range of package sizes, food properties and processing conditions. The centerpoint time-temperature data were used to calculate process times required for a target lethality of 5.0 min using the General method approach (employing numerical integration). The study indicated that process time prediction errors for the different methods calculated as percent deviations from those computed using the General method were relatively small (below 4% on average) within the range of experimental conditions.*

## INTRODUCTION

A primary objective of processing of foods in thin profile forms is to promote better retention of product quality. In order to maximize the quality retention, the process times should be kept at a minimal level required to assure commercial sterility in the product. The required process times are based on heat penetration data obtained for test packages under commercial processing conditions. Calculations can be based either on General method (Ball 1923, Bigelow *et al.* 1920 and Patashnik 1953) which involves integration of the lethal effects at the product center or one of the formula methods (Ball 1923, Stumbo 1973, Hayakawa 1970, Griffin *et al.* 1971, Steele and Board 1979, Pham 1987, 1990)

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Journal of Food Process Engineering *13(1991) 269–282. All Rights Reserved.*

which integrate the lethal effects using mathematical procedures. The formula methods developed for process calculations have been classified into two groups (Hayakawa 1978) with Group I methods based on the accumulated lethality at the slowest heating point and Group II methods based on the mass average lethality. Although, these procedures were primarily developed for cylindrical containers, they are also used for process time calculations involving packaged food in thin profile forms (Spinak and Wiley 1982).

Inaccuracies in the formula methods could lead to overestimation of the lethal value and reduce the margin of safety of processes or result in overprocessing with diminished product quality as have been reported by several investigators (Smith and Tung 1982, Steele *et al.* 1979, Hayakawa 1978). The container size and unaccomplished temperature difference at the end of heating (g-value) have been identified as factors significantly influencing the accuracy of formula methods used for conduction heating foods in cylindrical containers (Smith and Tung 1982). No detailed evaluation of formula methods has been reported for process calculations involving foods in thin profile forms.

The objective of this study was to evaluate selected formula methods for process time calculations involving packaged foods in thin profile forms using a finite difference computer simulation for gathering temperature-time data under conditions commonly applicable to processing of these products.

## METHODOLOGY

Five thermal process formula methods were evaluated in relation to a computer model based on a finite difference numerical solution to the governing differential equations involving conduction heat transfer with surface convection:

### Ball's Method

Ball's formula has been the industry standard since it was first introduced (Ball 1923). Some inaccuracies in the development of this method have been reported (Merson *et al.* 1978), but still it is the most widely used method in the food industry. One of the primary limitation of this method is the use of a constant cooling lag factor of $1.41(j_{cc})$. The procedure makes use of experimentally evaluated heat penetration parameters ($f_h$ and $j_{ch}$) and the operating conditions (RT and IT), and a set of tables or figures to compute the process time required to achieve a given process lethality (Fo) or vice versa. The American Can Company has developed more detailed tables, interpolating and extrapolating the tables that were originally published by Ball. These tables (Lopez 1987) were used for the evaluation of this method.

### Stumbo's Method

Stumbo and Longley (1966) published several tables for process evaluation taking into account the variability of $j_{cc}$ values. The Stumbo formula method is essentially similar to the Ball formula method except that it is somewhat more versatile in accounting for the thermal effects of cooling when the cooling lag factor ($j_{cc}$) differs from 1.41 as assumed by Ball. These original tables were obtained through planimeter measurements of hand-drawn temperature histories plotted on lethal rate papers, and subsequent interpolation of the graphs. Revised tables were later developed (Stumbo 1973) through the use of computer integration of thermal histories generated from finite difference simulations of heat transfer equations. These tables have been reported to produce results which are often in better agreement with those obtained from general method calculation than do similar calculations using Ball's method (Smith and Tung 1982). In the present evaluation, the tables in Stumbo (1973) were used. A common z value of 10 °C was used in all process calculations.

### Steele *et al* . Method

Steele *et al.* (1979) provided revised tables for $f_h/U$:g similar to those published by Ball and Olson (1957). They reported that the original tables published in Ball and Olson (1957) before the advent of sophisticated computers could be up to 26% off in relation to those obtained by them using the CYBER 76 computer. Steele *et al.* (1979) also reported that this meant that most of the F values calculated from Ball and Olson (1957) tables are overestimates and reduce the margin of safety associated with thermal processing. Steele *et al.* (1979) also developed polynomial equations (similar to those published by Vinters *et al.* 1975) to approximate table values for use with hand calculators and on-line computers. This method was evaluated using the published tables [S(T)] as well as the polynomial equations [S(E)].

### Pham's Method

Pham (1987) developed two sets of simple algebraic equations and simiplified tables for thermal process calculations, one for $U/f_h > 1$ and the other for $U/f_h < 1$. In his paper (Pham 1987), Pham claims that his method provides values at least as accurate as Stumbo's and is more versatile because his one table substitutes for the 57 tables published by Stumbo. This method could also be used for mass-average lethality similar to Stumbo's method (1973). In the present work, the published equations (Pham 1987) were used. Recently Pham amended his equations to cover situations in which the heating and cooling rates differ, i.e., $f_h$ not equal to $f_c$ (Pham 1990). The accuracy of the modified formulae were reported to be as good as the ones earlier reported for $f_h = f_c$ situations.

## Reference Method

The reference method to which the formula methods were compared was a numerical General method. This method used time-temperature data generated by a finite difference computer model for three-dimensional heat conduction with surface convection. The lethal rates at various temperatures were obtained as $10^{(T-121.1)/z}$ employing a z value of 10 °C and a reference TDT of 1 min at 121.1 °C. The accumulated centerpoint lethality ($F_0$) was obtained by the numerical integration of lethal rate versus time (Patashnik 1953). Because of the small time intervals (< 0.8s), the lethality calculated from this method was assumed to give accurate values.

## Computer Model

The computer program written in BASIC was a modified program of Chang and Toledo (1989) to cover a brick-shaped geometry rather than cubes. The program was also modified to accommodate different retort come-up profiles. Time increments of 0.4 to 0.8 s were used with a three dimensional space grid of 10 × 10 × 10. In order to avoid divergent oscillations of the nodal temperature, a necessary condition for stability of the finite difference solution to three-dimensional heat conduction equations must be satisfied (Carslaw and Jaeger 1959). This condition for stability requires that the equation for the nodal Biot number in the three dimensions must be chosen by proper selection of the nodal distance and the time increment. Each simulation was prechecked for the stability using the criterion reported by Chang and Toledo (1989); the time increment chosen was well below the maximum required for stability (nodal distance varied for each simulation since a space grid of 10 × 10 × 10 was used irrespective of brick-dimensions).

Base conditions used for evaluating the formula methods are given in Table 1 representing typical processing conditions for a thin profile package. The thermal conductivity and thermal diffusivity are typical of a conduction heating food (Mohsenin 1980). It has been reported that the surface heat transfer coefficient associated with the heating media for processing of retortable pouches cannot be neglected as with conventional processing of cans (Ramaswamy *et al.* 1983, Peterson and Adams, 1983). A base value of heat transfer coefficient of 2000 W/m² °C was chosen and subsequently varied between 100 and 5000 W/m² °C. The same heat transfer coefficient was used for heating and cooling, and cooling water temperature was kept constant at 20 °C with a step-change in retort temperature from the operating temperature to cooling water temperature at the beginning of the cool.

Effect of other factors were also studied by varying them one at a time from the base condition: retort temperature, 110 to 132.2 °C; initial temperatures,

TABLE 1.
LEVELS OF SELECTED PARAMETERS EMPLOYED FOR EVALUATING FORMULA METHODS

| Parameter | Base conditions | Levels tested |
|---|---|---|
| Retort temperature ($^{o}$C) | 121.1 | 110.0 - 132.2 |
| Initial food temperature ($^{o}$C) | 65.6 | 37.8 - 82.2 |
| Cooling water temperature ($^{o}$C) | 20 | |
| Dimensions of the package: | | |
| Thickness (cm) | 2.0 | 1.0 - 5.0 |
| Width (cm) | 10.0 | 2.0 - 10.0 |
| Length (cm) | 15.0 | 3.0 - 15.0 |
| Thermal conductivity (W/m $^{o}$C) | 0.5 | 0.3 - 0.7 |
| Thermal diffusivity ( ) ($m^2$/s) | $1.5 \times 10^{-7}$ | 1.0 - 2.0 ($\times 10^{-7}$) |
| Heat transfer coefficient (W/$m^2$ $^{o}$C)* | 2000 | 100 - 5000 |
| Come-up time (min) | 0.0 | 5 - 15 |
| Total process lethality (min) | 5.0 | |
| Microorganism z-value (z), $^{o}$C | 10 | |

* both during heating and cooling

37.8 to 82.2 °C; thermal diffusivity, $1.0 \times 10^{-7}$ to $2 \times 10^{-7}$ $m^2$/s; thermal conductivity, 0.3 to 0.7 W/m °C. Various thickness (1.0 to 5.0 cm), widths (2.0 to 10.0 cm) and lengths (3.0 to 15.0 cm) were investigated in order to find out what represents an infinite slab and to evaluate the effect of size.

For each time-temperature curve generated, all thermal process parameters $f_h$, $f_c$, $j_{ch}$ and $j_{cc}$ were computed by manually locating the linear portions of the heating and cooling curves. The time-temperature data were also used to obtain the required process time ($t_{ref}$) to achieve a process lethality of 5.0 min. For this procedure, the computer simulation was initially set to obtain the process time for a heating period lethality of 5.0 min which was subsequently reduced by trial and error to get an overall process lethality (heating plus cooling) of 5.0 min. By using the evaluated heat penetration parameters and processing conditions, process times were calculated by the selected formula methods ($t_{test}$). Percentage deviations of $t_{test}$ from $t_{ref}$ were calculated as prediction errors associated with the test method using equation (1):

$$\text{Error} = (t_{test} - t_{ref}) / t_{ref} \times 100\% \quad (1)$$

Positive errors associated with a formula method indicate that it will result in overprocessing while negative errors indicate the contrary.

Additional time-temperature data were gathered under base conditions to test the influence of come-up time (5, 10 and 15 min) with a linear come-up profile on the accuracy of the different formula methods. For these calculations, a come-up period (CUT) efficiency of 42% (Ball 1923) was employed to calculate $t_{ref}$. In other words, $t_{ref}$ = (Total heating time-0.58*CUT). Further, data were also gathered at selected temperatures (115.6, 121.1 and 126.7 °C) to study the effect of g value (0.05, 0.1, 1, 5, 10 and 15 °C) on process calculations.

## RESULTS AND DISCUSSION

### Comparison of Formula Methods Under Base Conditions

The process time obtained by the reference method ($t_{ref}$) to achieve a process lethality of 5.0 min was 19.0 min under the base conditions. The evaluated process times by the formula methods were: 19.32 min (Ball), 19.54 min (Steele *et al.* - equation and table), 18.99 (Stumbo) and 19.04 min (Pham) giving only marginal errors which were all positive except with Stumbo which gave a -0.05% error. The formula methods were evaluated subsequently under a range of processing conditions that resulted in a range of process times varying from 11.8 min to 78 min all resulting in an equivalent process lethality of 5 min (Table 2 and 3).

### Influence of Package Size

Package sizes were initially selected to arrive at a dimension that represented an infinite plate. This could be obtained by evaluating the influence of each dimension on the resulting heating rate index ($f_h$) or process time. With thickness and width at 2 cm each, increasing the length dimension beyond 6 cm did not result in any further change in process time (Table 2) or $f_h$ (5.46 min). With length kept at 15 cm, increasing the width showed additional deterioration of heating rate and resulted in increased process times up to a width of 7 cm. Although not indicated by the process time, the width factor showed additional small influence up to 9 cm as indicated by the $f_h$ values. A package size of dimension 10 (width) × 15 (length) with a thickness of 2.0 cm was therefore considered adequate to provide an infinite plate configuration. The various combinations of length, width and thickness provided a wide data base for verification of formula methods with process times varying from a low value of 11.8 min to a high 62.6 min. These conditions showed some differences in the accuracy of different methods (Table 2). The errors were generally minimal under the infinite plate configurations and higher when the heat transfer contributions

TABLE 2.
INFLUENCE OF PACKAGE DIMENSIONS ON PROCESS TIMES AND CALCULATION ERRORS

| Dimensions (cm) | | | Process Time (min) | Error (%) | | | | |
|---|---|---|---|---|---|---|---|---|
| T | W | L | | B(T)[1] | S(E)[2] | S(T)[3] | St(T)[4] | P(E)[5] |
| 2.0 | 2.0 | 3.0 | 11.80 | 4.92 | 5.85 | 5.51 | 0.59 | 1.44 |
| 2.0 | 2.0 | 6.0 | 12.67 | 4.66 | 5.84 | 5.60 | 2.60 | 3.24 |
| 2.0 | 2.0 | 9.0 | 12.67 | 3.79 | 5.05 | 4.74 | 1.26 | 1.97 |
| 2.0 | 2.0 | 12.0 | 12.67 | 3.79 | 5.05 | 4.74 | 1.18 | 1.97 |
| 2.0 | 2.0 | 15.0 | 12.67 | 3.79 | 5.05 | 4.74 | 1.18 | 1.97 |
| 2.0 | 3.0 | 15.0 | 15.60 | 3.46 | 4.55 | 4.62 | 0.45 | 1.09 |
| 2.0 | 4.0 | 15.0 | 17.20 | 2.85 | 3.90 | 4.19 | 0.70 | 0.93 |
| 2.0 | 5.0 | 15.0 | 18.00 | 2.44 | 3.56 | 3.89 | 0.72 | 0.83 |
| 2.0 | 6.0 | 15.0 | 18.40 | 2.17 | 3.32 | 3.70 | 0.60 | 0.65 |
| 2.0 | 7.0 | 15.0 | 19.00 | 2.21 | 3.37 | 3.79 | 0.74 | 0.74 |
| 2.0 | 8.0 | 15.0 | 19.00 | 1.84 | 3.05 | 3.53 | 0.16 | 0.21 |
| 2.0 | 9.0 | 15.0 | 19.00 | 1.68 | 2.95 | 3.37 | 0.32 | 0.26 |
| 2.0 | 10.0 | 15.0 | 19.00 | 1.63 | 2.84 | 3.26 | 0.26 | 0.21 |
| 1.0 | 10.0 | 15.0 | 9.00 | 1.00 | 1.67 | 1.78 | -0.33 | 0.00 |
| 1.5 | 10.0 | 15.0 | 13.40 | 1.49 | 2.69 | 2.54 | -0.22 | 0.22 |
| 2.0 | 10.0 | 15.0 | 19.00 | 1.68 | 2.89 | 3.32 | -0.21 | -0.16 |
| 2.5 | 10.0 | 15.0 | 25.30 | 1.74 | 3.64 | 4.03 | 0.00 | 0.20 |
| 3.0 | 10.0 | 15.0 | 32.30 | 1.36 | 4.43 | 4.58 | -0.15 | 0.34 |
| 3.5 | 10.0 | 15.0 | 40.00 | 2.23 | 5.27 | 5.15 | -0.47 | 0.23 |
| 4.0 | 10.0 | 15.0 | 47.30 | 2.68 | 6.00 | 5.64 | -0.97 | -0.11 |
| 4.5 | 10.0 | 15.0 | 55.00 | 3.44 | 6.91 | 6.45 | -0.78 | 0.15 |
| 5.0 | 10.0 | 15.0 | 62.60 | 3.85 | 7.40 | 6.82 | -1.02 | -0.02 |

T=thickness; W width; L=length; 1=Ball's table method;
2=Steele *et al.* equation method; 3=Steele *et al.* table method;
4=Stumbo's table method; 5=Pham's equation method.

from all the width and length dimensions were significant. Steele *et al.* methods showed the largest errors (1.67 to 7.40%, equation; 1.78 to 6.82%, table) with the Ball method coming next (1.00 to 4.92%) followed by Stumbo (-1.02% to 2.6%) and Pham (-0.16 to 3.24%).

## Influence of Other Factors

Table 3 shows the influence of various parameters on the reference process time, evaluated process parameters and corresponding errors associated with formula methods within the range tested. Retort temperature was obviously the most dominant factor influencing the process times with the lower retort temperature (110°C) requiring a high 78 min process while it was a short 10.8 min process at 132.2°C. The other factor of some significance was the heat

TABLE 3.
ERRORS IN CALCULATED PROCESS TIMES USING FORMULA METHODS: INFLUENCE OF VARIOUS PARAMETERS

| Factor | Process Time (min) | $f_h$ (min) | $j_{ch}$ | $j_{cc}$ | Error (%) B(T)[1] | S(E)[2] | S(T)[3] | St(T)[4] | P(E)[5] |
|---|---|---|---|---|---|---|---|---|---|
| **Thermal diffusivity ($m^2/s \times 10^{-7}$)** | | | | | | | | | |
| 1.0 | 25.00 | 16.31 | 1.24 | 1.30 | 0.72 | 2.48 | 2.92 | -1.08 | -0.84 |
| 1.2 | 22.00 | 13.59 | 1.28 | 1.27 | 1.55 | 3.05 | 3.50 | 0.36 | 0.14 |
| 1.4 | 20.00 | 11.64 | 1.28 | 1.27 | 1.55 | 2.85 | 3.30 | 0.30 | 0.15 |
| 1.5 | 19.00 | 10.89 | 1.28 | 1.27 | 1.63 | 2.84 | 3.26 | 0.26 | 0.21 |
| 1.6 | 18.40 | 10.18 | 1.28 | 1.27 | 1.63 | 2.77 | 3.15 | 0.11 | 0.16 |
| 1.8 | 17.00 | 9.07 | 1.27 | 1.27 | 1.59 | 2.76 | 3.06 | 0.18 | 0.24 |
| 2.0 | 16.00 | 8.16 | 1.27 | 1.26 | 1.69 | 2.75 | 2.94 | 0.12 | 0.25 |
| **Initial temperature ($^oC$)** | | | | | | | | | |
| 37.8 | 21.00 | 10.86 | 1.28 | 1.27 | 1.48 | 2.57 | 2.95 | 0.19 | -0.05 |
| 48.9 | 20.33 | 10.89 | 1.27 | 1.25 | 1.67 | 2.86 | 3.25 | 0.54 | 0.34 |
| 60.0 | 19.50 | 10.89 | 1.27 | 1.24 | 1.64 | 2.82 | 3.23 | 0.46 | 0.36 |
| 65.6 | 19.00 | 10.89 | 1.28 | 1.27 | 1.63 | 2.84 | 3.26 | 0.26 | 0.21 |
| 71.1 | 18.50 | 10.89 | 1.27 | 1.24 | 1.62 | 2.92 | 3.41 | 0.49 | 0.38 |
| 82.2 | 17.67 | 10.89 | 1.27 | 1.23 | 1.58 | 2.82 | 3.28 | 0.23 | 0.23 |
| **Retort temperature ($^oC$)** | | | | | | | | | |
| 110.0 | 78.00 | 10.89 | 1.27 | 1.26 | 0.64 | 1.09 | 1.28 | 0.27 | 0.15 |
| 115.6 | 33.00 | 10.89 | 1.27 | 1.26 | 1.33 | 2.09 | 2.06 | 0.09 | 0.21 |
| 121.1 | 19.00 | 10.89 | 1.28 | 1.27 | 1.63 | 2.84 | 3.26 | 0.26 | 0.21 |
| 126.7 | 13.60 | 10.89 | 1.28 | 1.28 | 1.10 | 4.56 | 4.12 | -0.81 | -0.07 |
| 132.2 | 10.80 | 10.89 | 1.27 | 1.28 | 2.31 | 5.74 | 5.65 | 0.37 | 0.74 |
| **Heat trans. coef. ($W/m^2\ ^oC$)** | | | | | | | | | |
| 100 | 30.50 | 21.49 | 1.23 | 1.17 | 0.10 | 2.46 | 2.72 | -0.66 | -0.33 |
| 500 | 21.00 | 12.47 | 1.27 | 1.26 | 1.43 | 2.76 | 3.24 | 0.24 | 0.10 |
| 1000 | 19.50 | 11.40 | 1.27 | 1.24 | 1.59 | 2.92 | 3.38 | 0.56 | 0.36 |
| 1500 | 19.00 | 11.04 | 1.28 | 1.26 | 1.63 | 2.84 | 3.26 | 0.26 | 0.16 |
| 2000 | 19.00 | 10.89 | 1.28 | 1.27 | 1.63 | 2.84 | 3.26 | 0.26 | 0.21 |
| 5000 | 18.60 | 10.57 | 1.28 | 1.27 | 1.67 | 2.85 | 3.28 | 0.22 | 0.22 |
| **Thermal conductivity ($W/m\ ^oC$)** | | | | | | | | | |
| 0.3 | 18.70 | 10.67 | 1.27 | 1.24 | 1.55 | 2.78 | 3.26 | 0.37 | 0.32 |
| 0.5 | 19.00 | 10.89 | 1.28 | 1.27 | 1.63 | 2.84 | 3.26 | 0.26 | 0.21 |
| 0.7 | 19.40 | 11.09 | 1.27 | 1.27 | 1.55 | 2.84 | 3.25 | 0.21 | 0.15 |

1 = Ball's table method; 2 = Steele et al. equation method; 3 = Steele et al. table method; 4 = Stumbo's table method; 5 = Pham's equation method.

transfer coefficient that resulted in process time variations 18.6 to 30.5 min. Initial temperature and thermal diffusivity had similar influence on process times within the range studied. Thermal conductivity was the least influential of all the parameters presumably because most processing situations covered involved considerably large Biot numbers (10–500). However, none of these factors had any major effect on error magnitudes by the different process calculation methods beyond what was observed under the base condition (Table 3). Some variability was noted especially at the high processing temperatures (126.7 – 132.2 °C) with errors reaching 4.1 to 5.8% for the Steele and Board methods. For Stumbo and Pham and errors were 1% or lower.

## Influence of g Value

The g value was reported to be a major factor influencing the accuracy of lethality estimations from different models with errors ranging from 15.9 to 60.3% when processing at 120 °C with a g value of 15 °C (Smith and Tung 1982). Controlled g value results at three retort temperatures (Table 4) indicated that the formula methods were relatively more sensitive g value than to any factor other than size (Table 2, 3 and 4), but the error magnitudes were considerably smaller than reported by Smith and Tung (1982). The process times at various temperatures to achieve a given g value were similar, with the process times at 115.6 °C and 126.7 °C within a ± 0.5 min of the process time at the mid temperature of 121.1 °C. However, the smaller g values (0.1 °C or lower), which also required somewhat similar process times of 30 to 34 min, resulted in large lethality differences (58–70 min at 126.7 °C to about 5 min at 115.6 °C). The g values of 10 and 15 °C resulted in negligible lethalities (0.08 and 1.04 min) in the short heating times required (7–10 min). Although the errors were generally high at the high g value of 15 °C, within the region of interest (g values 0.1 to 5 °C) the errors associated with the model were not different from those reported earlier. The overall influence of g values (at a retort temperature of 121.1 °) on the accuracy of process calculation methods are shown in Fig. 1 which shows the association of larger errors with higher g values. The results at the other retort temperatures were similar. Errors associated with Steele *et al.* models appear to increase systematically with g value while no specific trends were observed wtih others. Pham's method had errors exceeding 1% only with a g value of 15 °C. This is perhaps due to the insufficient development of a logarithmic temperature profile at the geometric center within the short process time required to achieve the g value of 15 °C (7–8 min). In any case, the process times calculated by Stumbo and Pham methods were much closer to the reference process time.

TABLE 4.
INFLUENCE OF g-VALUE AND RETORT TEMPERATURE ON ERROR PERCENTS

| g-value (°C) | Process Time (min) | Lethality (min) | Error(%) B(T)[1] | S(E)[2] | S(T)[3] | St(T)[4] | P(E)[5] |
|---|---|---|---|---|---|---|---|
| **RT=121.1 °C** | | | | | | | |
| 0.05 | 34.0 | 19.10 | 0.82 | 1.53 | 1.56 | -0.74 | -0.41 |
| 0.1 | 30.8 | 15.95 | 1.79 | 2.11 | 1.82 | -0.13 | -0.03 |
| 1.0 | 20.2 | 6.04 | 0.94 | 2.18 | 2.52 | -0.74 | -0.64 |
| 5.0 | 12.6 | 1.18 | 1.27 | 5.00 | 4.52 | -1.51 | -0.63 |
| 10.0 | 9.4 | 0.29 | 1.81 | 5.11 | 5.43 | -1.06 | 0.00 |
| 15.0 | 7.6 | 0.09 | 1.58 | 5.26 | 5.66 | -2.24 | 3.03 |
| **RT=115.6°C** | | | | | | | |
| 0.05 | 33.6 | 5.40 | 1.34 | 1.99 | 2.11 | -0.06 | 0.24 |
| 0.1 | 30.4 | 4.51 | 2.17 | 2.50 | 2.24 | 0.30 | 0.36 |
| 1.0 | 19.6 | 1.71 | 1.99 | 3.32 | 3.62 | 0.05 | 0.10 |
| 5.0 | 12.2 | 0.34 | 0.82 | 4.75 | 4.18 | -1.64 | -0.82 |
| 10.0 | 9.0 | 0.08 | 1.44 | 4.78 | 5.11 | -1.33 | -0.22 |
| 15.0 | 7.0 | 0.02 | 3.71 | 7.57 | 8.14 | -1.14 | 4.57 |
| **RT=126.7 °C** | | | | | | | |
| 0.05 | 34.4 | 69.24 | 1.45 | 2.06 | 2.12 | 0.03 | 0.41 |
| 0.1 | 31.2 | 57.82 | 2.24 | 2.60 | 2.34 | 0.35 | 0.54 |
| 1.0 | 20.6 | 21.86 | 1.50 | 2.57 | 2.91 | -0.24 | -0.05 |
| 5.0 | 13.2 | 4.26 | 0.08 | 3.71 | 3.18 | -2.05 | -1.21 |
| 10.0 | 9.8 | 1.04 | 2.35 | 5.41 | 5.71 | -0.82 | 0.20 |
| 15.0 | 8.0 | 0.30 | 5.62 | 5.62 | 6.00 | -1.75 | 3.00 |

1 = Ball's table method; 2 = Steele et al. equation method; 3 = Steele et al. table method; 4 = Stumbo's table method; 5 = Pham's equation method.

## Influence of Come-up Time

The influence of come-up time on process calculations with a 42% come-up period effectiveness factors is shown in Table 5. As with the other factors, the prediction errors associated with the formula methods under the various come-up situations were small (< 3.5%). This suggests that the use of a 42% effectiveness factor as suggested by Ball (1923) is acceptable with the linear come-up profile employed in the study. This supports similar observation by Ramaswamy and Tung (1986) who also reported that the effectiveness could be over 80% with a logarithmic come-up profile.

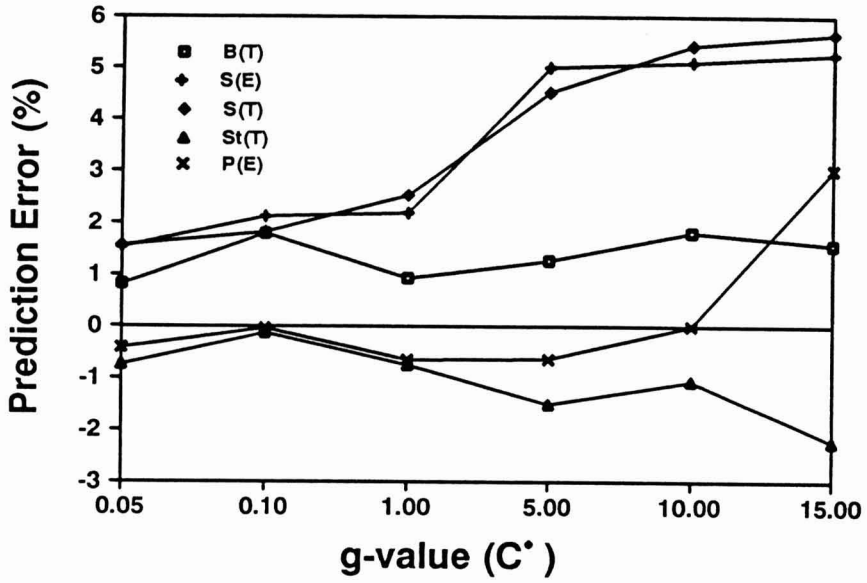


FIG. 1. ACCURACY OF FORMULA METHODS AS RELATED TO SELECTED g VALUES AT A RETORT TEMPERATURE OF 121.1 °C [B(T), BALL'S TABLE METHOD; S(E), STEELE *ET AL.* EQUATION METHOD; S(T), STEELE *ET AL.* TABLE METHOD: St(T); STUMBO'S METHOD; P(E), PHAM'S METHOD]

TABLE 5.
INFLUENCE OF COME-UP TIME ON PROCESS CALCULATIONS AT 42% EFFECTIVENESS

| Come-up Time (min) | Process Time (min) | Error (%) | | | | |
|---|---|---|---|---|---|---|
| | | B(T)[1] | S(E)[2] | S(T)[3] | St(T)[4] | P(E)[5] |
| 0 | 19.0 | 1.63 | 2.84 | 3.26 | 0.26 | 0.21 |
| 5 | 19.1 | 1.62 | 2.83 | 3.25 | 0.21 | 0.21 |
| 10 | 19.2 | 1.82 | 2.97 | 3.44 | 0.36 | 0.36 |
| 15 | 19.6 | 1.79 | 2.96 | 3.37 | 0.56 | 0.46 |

1 = Ball's table method; 2 = Steele et al. equation method; 3 = Steele et al. table method; 4 = Stumbo's table method; 5 = Pham's equation method.

## CONCLUSIONS

The overall errors and their standard deviations associated with process calculation by formula as applicable to processing of foods in thin profile containers were: 3.8% and 1.4% (Steele and Board - equation); 4.0% and 1.2% (Steele and Board - table); 2.3% and 1% (Ball); 0.7% and 0.9% (Pham) and 0.5% and 0.6% (Stumbo). Although Stumbo and Pham methods showed smaller errors compared to the others, the overall difference was relatively small to favor any one method over the other within range of experimental conditions. This may be due to the fact that the processing of thin profile conditions provide thermal processing parameters (especially $j_{cc}$) that better approximate the assumptions on which the formula methods are based.

## ACKNOWLEDGMENT

The authors wish to acknowledge the financial support for the project from Natural Sciences and Engineering Research Council's Operating and Strategic Grants Programs.

## NOMENCLATURE

| | |
|---|---|
| B(T) | Ball's table method |
| CUT | Come-up time |
| D | Decimal reduction time of a microorganism |
| $f_c$ | Cooling rate index |
| $f_h$ | Heating rate index |
| $F_o$ | Process lethality |
| g | Difference in temperature between product center and retort at the end of the heating period |
| IT | Initial temperature |
| $j_{cc}$ | Cooling lag factor |
| $j_{ch}$ | Heating lag factor |
| L | Length |
| P(E) | Pham's equation method |
| RT | Retort temperature |
| S(E) | Steele *et al.* equation method |
| S(T) | Steele *et al.* table method |
| St(T) | Stumbo's table method |
| T | Temperature |
| t | time |

| | |
|---|---|
| T | Thickness |
| $t_{ref}$ | Reference process time from computer simulation |
| $t_{test}$ | Process time from formula methods |
| U | Sterilizing value at the retort temperature |
| W | Width |
| z | Temperature difference resulting in a ten-fold change in D value of a microorganism |

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# BARLEY β-AMYLASE HYDROLYSIS OF STARCH DURING TWIN SCREW EXTRUSION

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Accepted for Publication November 26, 1990

## ABSTRACT

*Effect of moisture content, screw rpm and β-amylase dose on the rate of maltose production in a Baker-Perkins twin screw food extruder has been studied. Fifteen experiments were conducted using a fixed screw configuration at a fixed mass flow rate of 15 kg/h, under isothermal conditions (t=57°C) and a pH of 5.5. Each of the three variables was found to have a significant effect (P< .05) on maltose production. Using response surface regression analysis on the experimental data, a quadratic objective function was derived. The function was optimized, subjecting it to three explicit boundary conditions, using the adaptive complex method. The optimization process yielded a maximum maltose production of 34% at 54% moisture content, 30 rpm screw speed and 157 units of enzyme/g of starch. The extent of saccharification was about 30%.*

## INTRODUCTION

Increasingly, maltose has been used to replace sucrose and glucose in foods such as confectionery, frozen dessert, bakery and brewery products. In 1978, about 20,000 tons of normal-maltose (50–60%) and high maltose (> 80%) syrups were produced (Norman 1981). Maltose is not as sweet as sucrose (it contains only 30–40% sucrose), therefore its consumption leaves no aftertaste in the mouth. It is relatively thermostable, and has low hygroscopicity, low tendency to crytalize, low solution viscosity and high fermentability (Linko *et al.* 1984). Maltose also has better antiseptic properties than sucrose and can be used in intravenous feeding at high concentration without raising the blood glucose level, making it suitable for diabetics (Takasaki and Yamanobe 1981).

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Journal of Food Process Engineering *13(1991) 283–295. All Rights Reserved.*

Maltose can be produced through a saccharification process using β-amylase, and starch preliquefied by acid and/or enzyme (usually α-amylase) hydrolysis. It can also be produced by enzyme-catalyzed extrusion (reactive extrusion) of carbohydrate substrates in a single extrusion pass (Ofoli *et al.* 1990). Extrusion cooking promotes thermomechanical degradation of starch, and can lead to formation of low dextrose equivalent (DE) liquefied starch suitable for subsequent saccharification (Linko *et al.* 1984). The maltose yield depends on the initial DE of the liquefied starch. The lower the DE, the higher the maltose produced, with a small formation of maltotriose and high oligosaccharides (Takasaki and Yamanobe 1981).

During saccharification, B-amylase splits off maltose in a stepwise manner from the non-reducing end of the starch chain, but cannot bypass the branches of the amylopectin chain. Therefore, the maximum theoretical yield of maltose is 60%. With addition of a debranching enzyme (isoamylase or pullulanase), α-1,6-glucosidic bonds are cleaved to linear chain molecules for β-amylase, therefore a higher maltose yield results. In this regard, isoamylase is better than pullulanase because it produces smaller molecules for β-amylase reaction (Norman 1981). Because β-amylase is heat sensitive, its rate of reaction is slow. The saccharification is generally carried out at a pH of 5-5.5 and at 50 °C. So far, most of the saccharification processes have been carried out in a batch system over long reaction times to obtain high maltose yield.

Although use of a twin screw food extruder as a bioreactor for starch liquefaction has been extensively studied, few investigations of its application to saccharification has been reported. In particular, there are few models or analytical studies available to characterize and help understand the reactive extrusion process. In addition, the complex interactions of the various extrusion process variables are largely unknown in many simple as well as reactive processes.

The objectives of this study were to (1) determine the effect of moisture content, screw rpm and enzyme dose on maltose production in a twin screw extruder, and (2) assess what combination of conditions maximize or optimize maltose yield in the twin screw extruder.

## MATERIALS AND METHODS

A Baker-Perkins MPF-50D co-rotating twin screw food extruder (APV Baker, Grand Rapids, MI) was used for all runs. The screw profiles in the twin screw extruder (TSE) consisted of feed screws, single lead screws, and forwarding and reversing paddles arranged in three zones (Table 1). Two circular dies 3.2 mm in diameter and 25.4 mm in length were used.

Pre-gelatinized corn starch (American Maize Company, IN) was used as substrate. The starch and acetate buffer (30 mM) at a pH of 5.5 were mixed in

TABLE 1.
SCREW PROFILE USED IN THIS STUDY

| Zone | Type of screw element | Length (cm) | Location in extruder |
|---|---|---|---|
| 1 | Spacer | 0.6 | Prior to feed port |
| 1 | Feed screws | 25.4 | Pre-mixing zone |
| 2 | 30° forwarding paddles | 8.9 | Beginning of reaction zone |
| 2 | Feed screws | 12.7 | - |
| 2 | Single lead screws | 5.1 | - |
| 2 | 45° reversing paddles | 29.2 | - |
| 2 | 30° reversing paddles | 26.7 | End of reaction zone |
| 2-3 | Orifice plug | - | Divides zones 2 and 3 |
| 3 | Single lead screws | 5.1 | Beginning of deactivation zone |
| 3 | 45° reversing screws | 6.4 | - |
| 3 | Single pitch feed screws | 5.1 | Prior to exit dies |

the extruder (zone 1). Barley $\beta$-amylase (Spezyme BBA 1500, Finnsugar Biochemicals, Inc., IL) was diluted in the same buffer and was fed into the extruder. Enzyme activity of 10,000 units/mL was determined by the procedure described by Saha *et al.* (1987), where one unit is defined as the quantity of enzyme which liberates 1 micromole of glucose per min per mL of enzyme under the specified condition. A diagram of the extruder setup is shown in Fig. 1. All experiments were conducted at a fixed mass flow rate of 15 kg/h. The barrel temperature was set at 57 °C in the reaction zone and 93–107 °C in the deactivation zone.

A fractional factorial design with three factors (moisture content, screw rpm and enzyme dose) and three levels were used (Table 2). Treatments 13–15 were replicates, to enable the mean of the base point to be obtained.

After the extruder reached steady state, about 10 g of sample was collected and immediately immersed in liquid nitrogen to terminate the reaction. Three samples were collected for each condition and stored at −17 °C for subsequent analyses. The mean residence time was also measured in some runs via a tracer method, using 0.5 g of dye ball made up of a mixture of starch and 4% erythrosine solution. The mean residence time was calculated by the first moment formulation (Levenspiel 1972). The moisture content of the sample was determined by the oven method at 100–105 °C.

The frozen samples were thawed in a plastic bag at room temperature for 10 min. A mixture containing one g of the sample, 0.5 mL of 0.1 M NaOH and 10 mL of distilled water was homogenized in a Polytron homogenizer (Brinkman Instruments, NY) at 13,000 rpm for 10s. The solution was heated at 100 °C for 10 min, then cooled and brought up to 25 g with water. The solution was centrifuged at 13,000 rpm for 15 min using a Sorvall centrifuge (Dupont, IL). The supernatant was removed and subjected to sugar analyses.

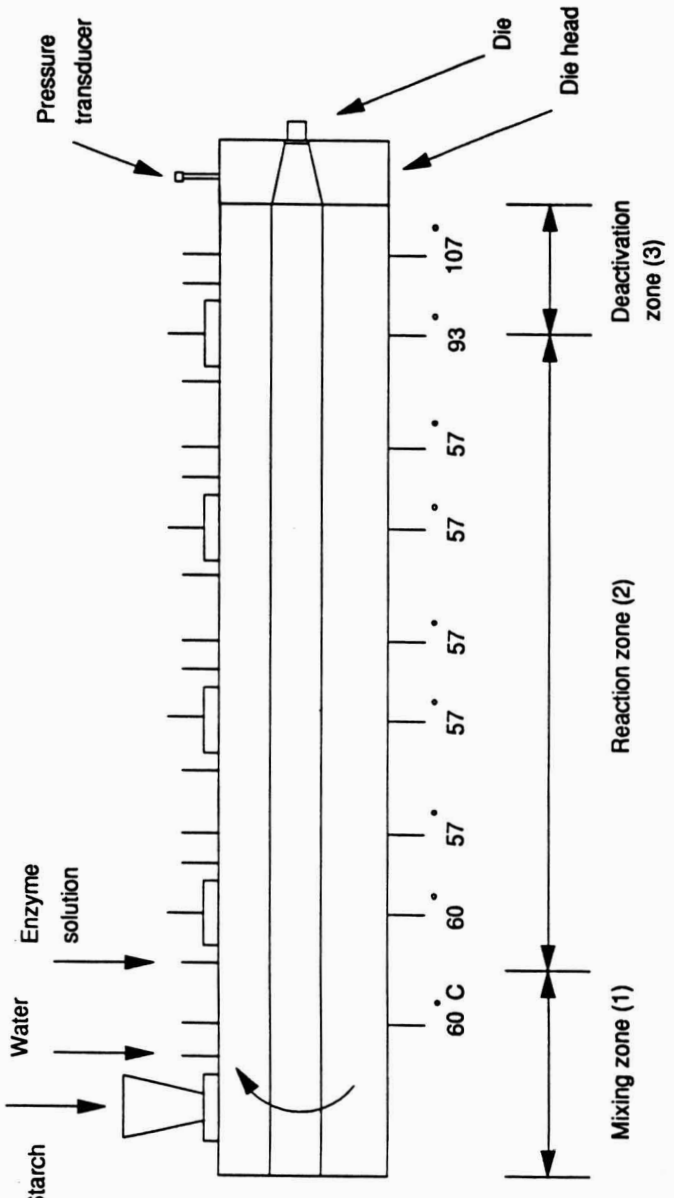


FIG. 1. EXTRUDER SETUP FOR ALL EXPERIMENTS, SHOWING THE ENTRY POINT OF THE ENZYME SOLUTION AND PRESET TEMPERATURES IN EACH ZONE

TABLE 2.
FRACTIONAL FACTORIAL DESIGN

| Run no. | % MC | RPM | Enzyme dose (units/g starch, DS) |
|---|---|---|---|
| 1 | 35 | 100[1] | 100 |
| 2 | | 150 | 100 |
| 3 | | 100 | 60 |
| 4 | | 100 | 150 |
| 5 | 50 | 60 | 60 |
| 6 | | 150 | 60 |
| 7 | | 60 | 150 |
| 8 | | 150 | 150 |
| 9 | 65 | 60 | 100 |
| 10 | | 150 | 100 |
| 11 | | 100 | 60 |
| 12 | | 100 | 150 |
| 13 | 50 | 100 | 100 |
| 14 | | 100 | 100 |
| 15 | | 100 | 100 |

[1]Lowest rpm at which extruder will operate at this moisture content.

The amount of reducing sugars was determined by the dinitrosalicyclic acid (DNS) and DE methods. The DNS procedure was developed by Bernfeld (1955) and modified by Saha *et al.* (1987). The DE method was developed by Dygert *et al.* (1965). The quantity of maltose was determined by the HPLC procedure described by Saha *et al.* (1988). The HPLC system consisted of a saccharide analysis column (Aminex HPX-42A, Bio-Rad Laboratories, Richmond, CA) which was connected to a carbohydrate deashing system. The column was maintained at 85 °C, and maltose was eluted with distilled water.

The amount of unreacted starch was analyzed by the UV method as described by Anon. (1987), whereby soluble starch is removed from the insoluble unreacted starch using 40% (v/v) ethanol in water. The unreacted starch was then solubilized in dimethylsulfoxide (DMSO) and then hydrolyzed by amyloglucosidase (AGS) to form glucose. Using hexokinase (HK), glucose-6-phosphate dehydrogenase (G6P-DH) and nicotinamide-adenine dinucleotide phosphate (NADP), the glucose formed was subsequently transformed by the reactions catalyzed by these enzymes to form reduced nicotinamide-adenine dinucleotide phosphate (NADPH), which was then measured spectrophotometrically at 340 nm. The absorbance of the NADPH was used to calculate the amount of unreacted starch in the sample.

## RESULTS AND DISCUSSION

### Maltose Production by β-Amylase in the Extruder

Maltose production, reducing sugar content and DE were all determined and are summarized in Table 3. The actual moisture contents of the extrudates are listed, and show slight variations from the target moisture contents in Table 2. These variations can cause some fluctuations in screw rpm and enzyme dosage used. However, no significant fluctuations in screw rpm were observed from the control panel of the TSE, and the actual enzyme dosage was impossible to assay unless all thermal and shear effects are known. In this study, the target screw rpm and enzyme dosage were assumed to be relatively constant in all analyses.

A plot of percent reducing sugar (RS) and DE versus maltose production showed that both percent RS and DE are highly correlated to maltose production (Fig. 2). Therefore, either of the two indicators can be used to measure the extent of product formation. The extent of saccharification, measured in terms of fractional conversion (X) is also presented in Table 3.

The fractional conversion was calculated by:

$$X = 1 - \frac{C_s}{C_{so}} \tag{1}$$

Analysis of variance (Table 4) shows that moisture content, screw rpm and enzyme dosage all significantly affect the yield of maltose ($P < .05$).

### Model Development and Optimization

The objective function describing maltose production as a function of moisture content, screw rpm and enzyme level was statistically determined using the SAS program for personal computers (Anon. 1985). SAS response surface regression analysis for the three-factor quadratic model was applied to the experimental data, each of which was normalized by its base point to increase the sensitivity of analysis. The results are summarized in Table 5, and show that there is agreement between the data and the model. Linear and quadratic terms were highly significant ($P < .05$), but cross-product terms were insignificant. The model parameters were estimated and are presented in Table 6. For the full model which contained all linear, quadratic and cross-product terms, the overall $R^2$ was 0.963. The full model is expressed by:

$$Y = 24.0 + 9.78X_1 - 2.43X_2 + 8.68X_3 - 14.7X_1^2 - 0.017X_2^2 - 3.81X_3^2$$

$$-1.1X_1X_2 + 1.31X_1X_3 + 1.0X_2X_3 \tag{2}$$

where

$$X_1 = \frac{X_1' - 48.7}{15} \quad (3)$$

$$X_2 = \frac{X_2' - 100}{50} \quad (4)$$

and

$$X_3 = \frac{X_3' - 100}{50} \quad (5)$$

TABLE 3.
MALTOSE PRODUCTION, REDUCING SUGAR LEVELS, DE AND FRACTIONAL CONVERSION (X) FOR ALL EXTRUSION RUNS

| Run no. | % MC | % Maltose (s.d.)[1] | % Reducing sugar (s.d.)[1] | DE (s.d.)[1] | X |
|---|---|---|---|---|---|
| 1 | 34.9 | 3.37(0.23) | 5.15(0.15) | 4.12(0.24) | 0.17 |
| 2 | 34.8 | 1.90(0.00) | 3.70(0.27) | 2.95(0.31) | 0.16 |
| 3 | 39.3 | 3.03(0.83) | 3.75(0.28) | 2.27(0.28) | 0.15 |
| 4 | 34.3 | 2.90(0.17) | 4.95(0.27) | 3.87(0.24) | 0.20 |
| 5 | 49.0 | 17.30(0.20) | 17.09(0.62) | 13.80(1.48) | 0.17 |
| 6 | 49.8 | 10.50(0.00) | 11.35(0.62) | 8.08(0.24) | 0.16 |
| 7 | 48.0 | 31.10(0.72) | 24.82(0.42) | 20.62(0.42) | 0.31 |
| 8 | 46.5 | 26.00(0.72) | 22.26(0.53) | 17.57(0.78) | 0.30 |
| 9 | 60.7 | 23.40(0.97) | 20.84(0.29) | 17.38(0.46) | 0.30 |
| 10 | 61.0 | 18.80(4.70) | 18.60(0.88) | 14.52(0.86) | 0.26 |
| 11 | 62.2 | 12.33(1.03) | 12.65(0.65) | 11.92(0.91) | 0.29 |
| 12 | 60.6 | 28.13(0.97) | 22.73(0.38) | 18.38(0.91) | 0.30 |
| 13 | 49.1 | 25.03(4.00) | 20.17(0.43) | 15.49(0.38) | 0.28 |
| 14 | 48.7 | 21.40(0.00) | 21.06(0.18) | 16.86(0.38) | 0.24 |
| 15 | 48.4 | 25.40(1.04) | 22.96(0.80) | 18.14(0.47) | 0.33 |

[1]standard deviation.

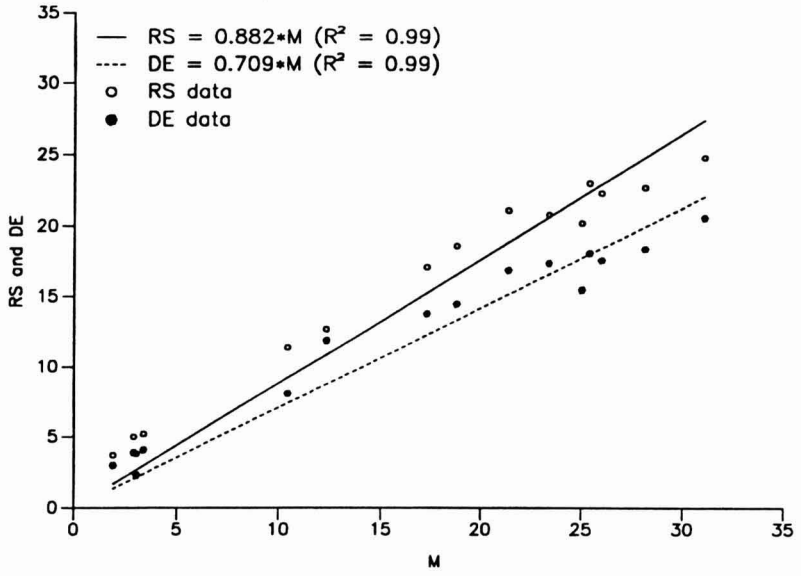


FIG. 2. PERCENT REDUCING SUGAR (RS) AND DEXTROSE EQUIVALENT (DE) VERSUS PERCENT MALTOSE (M)

TABLE 4.
ANALYSIS OF VARIANCE OF FACTOR EFFECTS

| Factor | Degrees of Freedom | Sum of Squares | Mean Square | F-Ratio | Prob > F |
|---|---|---|---|---|---|
| X1 | 4 | 2949.3 | 737.3 | 156.3 | 0.0000 |
| X2 | 4 | 119.1 | 29.8 | 6.3 | 0.0006 |
| X3 | 4 | 1290.2 | 322.6 | 68.4 | 0.0000 |

TABLE 5.
RESPONSE SURFACE ANALYSIS FOR % MALTOSE

| | |
|---|---|
| Response Mean | 16.7 |
| Root MSE | 2.2 |
| R-Square | 0.963 |
| Coeff. of Variation | 13.0 |

| Regression | Degrees of Freedom | Type I Sum of Squares | R square | F-Ratio | Prob > F |
|---|---|---|---|---|---|
| Linear | 3 | 2901.7 | 0.644 | 205.1 | 0.0000 |
| Quadratic | 3 | 1412.0 | 0.313 | 99.8 | 0.0000 |
| Crossproduct | 3 | 27.1 | 0.006 | 1.9 | 0.1453 |
| Total Regress | 9 | 4340.8 | 0.963 | 102.3 | 0.0000 |

TABLE 6.
ESTIMATION OF MODEL PARAMETERS

| Parameter | Degrees of Freedom | Parameter Estimate | Standard Error | T for HO: Parameter = 0 | P >\|T\| |
|---|---|---|---|---|---|
| Intercept | 1 | 24.01 | 0.72 | 33.53 | 0.0000 |
| X1 | 1 | 9.78 | 0.59 | 16.49 | 0.0000 |
| X2 | 1 | -2.43 | 0.66 | -3.69 | 0.0008 |
| X3 | 1 | 8.68 | 0.54 | 16.09 | 0.0000 |
| X1*X1 | 1 | -14.70 | 0.95 | -15.44 | 0.0000 |
| X2*X1 | 1 | -1.07 | 1.06 | -1.00 | 0.3223 |
| X2*X2 | 1 | -0.02 | 0.91 | -0.02 | 0.9856 |
| X3*X1 | 1 | 1.31 | 0.87 | 1.50 | 0.1428 |
| X3*X2 | 1 | 1.04 | 0.78 | 1.35 | 0.1906 |
| X3*X3 | 1 | -3.81 | 0.84 | -4.52 | 0.0001 |

When the cross-product terms were excluded, the goodness of fit dropped slightly ($R^2 = 0.957$). Since the contribution of the cross-product terms was less than 1%, they were eliminated. The final model is:

$$Y = 24.0 + 9.78X_1 - 2.43X_2 + 8.68X_3 - 14.7X_1^2 - 0.017X_2^2 - 3.81X_3^2 \quad (6)$$

The global optimum conditions for maltose production in the extruder were determined using the adaptive complex method (Manetsch 1990). The method is based on the complex method (Box 1965) which selects an appropriate starting point and uses several feasible points to form a geometric space to search for the location of the global optimum. In the adaptive complex method, the search process is more systematic, and reduces the expected number of function evaluations.

The maximization of the objective function was accomplished, subject to the following practical boundary conditions:

$$20 \leq X_1' \leq 80 \quad (7)$$

$$30 \leq X_2' \leq 450 \quad (8)$$

$$0 \leq X_3' \leq 300 \quad (9)$$

These boundary conditions were selected to include the experimental conditions used in this study. Results from the adaptive complex method showed that the maximum maltose production was 34% at 54% mc, 30 rpm and an enzyme dose of 157 activity units/g of starch. The effect of shear on $\beta$-amylase activity was pronounced; therefore, the maximum yield of maltose was obtained at minimum shear or at the lowest possible screw rpm. Increased moisture content and enzyme dosage led to increased maltose production.

At a fixed screw rpm of 30, the effect of moisture content and enzyme level on maltose production is shown in Fig. 3. Clearly, maltose production increased with increased enzyme level, as would be expected. At a fixed moisture content of 50%, the effect of screw rpm and enzyme level on maltose yield was evaluated and plotted in Fig. 4. Maltose production increased with increased enzyme level but decreased with increased screw rpm. This may be the result of shear deactivation of the enzyme as the screw rotation increased; however, it may also be due to a reduction in residence time.

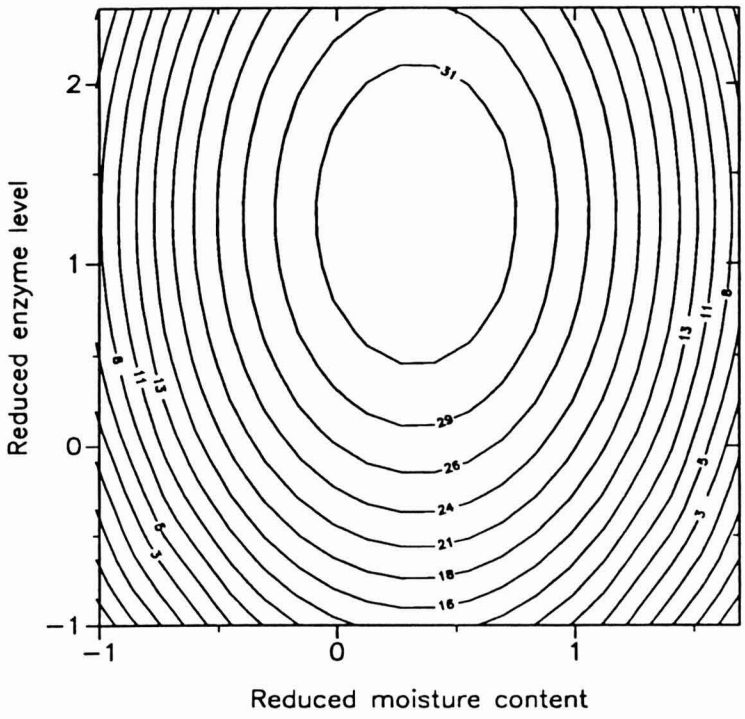


FIG. 3. CONTOUR LINES OF PERCENT MALTOSE VERSUS MOISTURE CONTENT AND ENZYME LEVEL AT 30 RPM

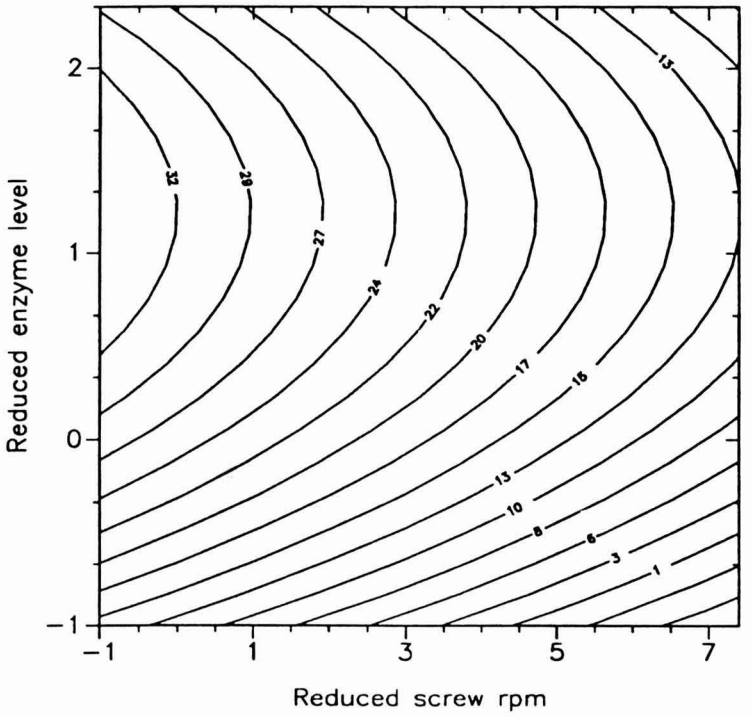


FIG. 4. CONTOUR LINES OF PERCENT MALTOSE VERSUS SCREW RPM AND ENZYME LEVEL AT 50 PERCENT MOISTURE CONTENT

The authors recognize that the model resulting from this study is highly empirical, and would need to be modified to make it applicable for use with other extruders. While a truly theoretical model should be developed on the basis of process mechanisms, this is a difficult task to achieve for even simple extrusion processes, let alone one that involves significant chemical reaction. The number of variables involved and the fact that these variables are related in ways that have not been fully understood yet complicate analysis. However, this should not limit the usefulness of this study. First, the study may provide relevant information that could be used in other reactive extrusion processes. Secondly, the trends reported in this study are representative of what might occur in other extruders. Finally, the systematic methodology used in this study is universally applicable to other extruders and extrusion processes.

To develop a truly generalized model for enzymatic maltose production in an extruder, information on material rheology, heat transfer, reaction kinetics and the nature of the flow regime would be required.

## CONCLUSIONS

The rate of maltose production was found to depend significantly on moisture content, screw rpm and $\beta$-amylase dosage. The three variables were incorporated into a model developed through response surface regression analysis of the experimental data. The model was then used to predict the optimum maltose production by an adaptive complex method, giving a maximum maltose production of 34% at 54% moisture content, 30 rpm and 157 units of enzyme/g of starch. This corresponded to about 30% starch conversion.

The process for producing maltose by reactive extrusion is dynamic. The quantity of maltose produced depends on enzyme reaction kinetics. Maltose production also depends on the residence time and, in general, the longer the residence time, the greater the quantity of maltose obtained. However, the residence time is governed by other process parameters such as mass flow rate, material rheological behavior, screw rpm, and moisture content, among others. For example, a longer residence time may produce a greater strain history than $\beta$-amylase can tolerate. The mean residence time in this study varied within a small range of 7–9 min. Since the screw profile was complex, the flow model was difficult to characterize, and would require further studies. The use of a dispersion model to characterize the enzymatic reaction by $\alpha$-amylase was recently investigated (Komolprasert 1989) and may provide a tool for this type of analysis.

## NOMENCLATURE

| | |
|---|---|
| $C_S$ | Starch concentration, % w/w |
| $C_{SO}$ | Initial starch concentration, % w/w |
| X | Fractional conversion, dimensionless |
| $X_1$ | Moisture content, % w/w |
| $X_2$ | Screw rpm, $min^{-1}$ |
| $X_3$ | Enzyme level, activity units/g starch |
| $X_1$ | Reduced percent moisture content, dimensionless |
| $X_2$ | Reduced screw speed, dimensionless |
| $X_3$ | Reduced enzyme level, dimensionless |
| Y | Maltose yield, % w/w |

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# REPARTITION OF WATER IN PLANT TISSUES SUBJECTED TO OSMOTIC PROCESSES

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## ABSTRACT

*A study was conducted to investigate the osmotic behavior of potato tissue in equilibrium with an osmotic medium consisting of various solutions ranging from 5% to 60% sucrose at 40°C. At equilibrium, the total volume changes of the potato tissue with respect to the initial volume were constant for a wide range of sucrose concentration of osmotic solutions (10% to 40%). In order to explain this behavior, the analysis was transposed from the global description of the entire structure to the analysis of the behavior of the cells. It was found that the cells of the potato tissue were plasmolyzed in osmotic media from 10% to 40% sucrose, that is, the protoplast of the plant cell separates from the cell wall due to the efflux of water from the cell. The extracellular volume, which comprises the cell wall and the intercellular space, increased whereas the cellular volume, which is equivalent to the protoplast, decreased to maintain a constant total volume. When the tissue was in equilibrium with highly concentrated sucrose solutions (40% to 60%), a loss of the integrity of the cells seemed to happen.*

*Using a typical composition of the potato tissue and the expressions relating the equilibrium water content of each phase to the chemical potential of water, the calculated volume changes were predicted in good agreement with the experimental volume changes of the lumped cellular volume of the tissue. Thus, from the thermodynamic approach, one can predict quantitatively the repartition of water in the tissue.*

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Journal of Food Process Engineering *13(1991) 297–320.* 

## INTRODUCTION

The process of drying a plant material by immersion in an osmotic solution has recently received an increasing attention. It is regarded as a potential alternative to conventional drying operations.

Some of the advantages of direct osmosis in comparison with other drying processes include minimized heat damage, less discoloration of the fruit by enzymatic browning (Ponting *et al.* 1966, Pontin 1973, Contreras and Smyrl 1981), increased retention of volatiles (Flink and Karel 1970; Dixon *et al.* 1974) improved textural quality (Shipman *et al.* 1972) and low operating costs (Bolin *et al.* 1983).

The osmotic process is a complex case of mass transfer through a tissue. According to Karel (1975), the process of dehydration of the plant material in an aqueous solution is characterized by at least two major and simultaneous counter-current flows. Water comes out from the biological structure to the osmoticum and solutes migrate from the osmotic solution into the plant tissue.

The quantity and the rate of water removal depend on several variables. The choice of solutes and their concentration in the osmotic solution, the temperature and time of the treatment have been identified as important parameters which affect the osmotic process (Ponting *et al.* 1966, Farkas and Lazar 1969, Hawkes and Flink 1978, Contreras and Smyrl 1981, Conway *et al.* 1983).

Numerous authors have also pointed out the important role that the plant tissue may play as a living material in the mass transport phenomena upon osmosis. According to Ponting (1973), there is a wide variation in the osmotic behavior of fruits which reflects itself in the state of the final osmotically dried products. As an example, due to the excessive loss of juice, tomatoes and citrus fruits are poorly suited for an osmotic drying process. However, the process is very successful for apples, potatoes and bananas. Flink (1975) found that under the same conditions of osmosis, the increase in solids concentration in the plant material varied with respect to the kind of fruits. Giangiacomo *et al.* (1987) observed that the difference between the data obtained for cherries and peaches under the same conditions of osmosis seemed to involve the intrinsic properties of these two fruits such as the structure and the compactness of the tissue. Stahl and Loncin (1979) investigated the diffusion of cyclohexane in potato tissue. They indicated that cell walls and membranes could influence the rate of transport of cyclohexane in potato tissue.

It is generally well known that the most important organ controlling the osmosis phenomenon is the plasmalemma membrane (Nobel 1983). Osmosis occurs as long as solute movement is restricted compared to water movement. In the osmotic water removal from fruits exposed to a sucrose solution, the dehydration is possible because the cell membranes are semi-permeable, that is, water passes through the membranes more readily than sugar. Since the osmotic

concentration is possible because of the selective permeability of the cell membranes, any treatment which involves the destruction or disruption of the cells prior or during the osmotic process results in poor osmosis allowing a greater uptake of sugar for a lower loss of water. For example, blanching, prior to the osmotic water removal treatment, is found to be detrimental to the osmotic process (Ponting 1973). Lenart and Flink (1984b) have pointed out that beyond transport related to diffusion, other mechanisms, such as the shrinkage of the tissue, could be of importance with respect to the overall mass transport during osmosis.

The phenomenon of osmosis has also been studied by plant physiologists but most of the models on the fundamentals of water relations in plant tissue have been developed in order to describe the behavior of biological structures under normal growing conditions (Noggle and Fritz 1976) taking into consideration the cellular structure of the plant tissue (Philip 1958a,b,c; Molz and Hornberger 1973, Molz and Ikenberry 1974, Molz 1976, Molz *et al.* 1979, Toupin 1986).

Dainty (1963) reviewed the equilibrium water relations of plant cells with an attempt to give a thorough thermodynamic approach to the phenomenon. The same author (1976) pointed out that the chemical potential is the most suitable parameter describing the state of water in any system.

This current investigation was undertaken to identify, quantify and model the internal structural changes of potato tissue subjected to an osmotic treatment in a sucrose solution, at equilibrium. A model has been developed for the prediction of the equilibrium data based on the previous thermodynamic consideration of equal chemical potential in all intervening phases. The present contribution extends the thermodynamic approach in order to quantify and predict the equilbrium state of a plant material subjected to an osmotic treatment.

## MATERIALS AND METHODS

Potatoes, Russet Burbank cultivar, grown in Northern Alberta were provided by a local food processor. They were classified according to their weight. The study was conducted using mature potato tubers weighing between 200–300 g. Potatoes were stored at 4 °C at least a month prior to the osmotic treatment but less than 6 months.

### Conditioning of the Potato Tissue

Potato tubers (10) were selected from the cold room and were used to obtain 280 disks of parenchyma tissue. A few cores were cut from the tubers using a cork borer (size 14). These cylinders were sliced using a sharp blade. The resulting disks (22 mm diameter, 5 mm thickness) were soaked in distilled water for at least 20 h at 4 °C prior to the osmotic treatment.

## Sample Treatment

Solutions with 5%–60% sucrose concentrations were prepared in triplicates by blending an amount of sucrose with distilled water on a weight to weight basis.

An osmotic experiment was conducted in the following manner: 18 slices (approximately 35g) were selected, blotted, weighed together accurately ($m^{\circ}_{p}$), and finally added to a vessel containing 500 mL of an osmotic solution measured with a volumetric flask. Gentle agitation was provided by a magnetic stirrer and the temperature of the vessel was maintained at 40 °C in a water bath for 24 h. To prevent evaporation during the treatment, a plastic wrap was put on top of the vessel.

At the end of the osmosis period, the slices were rinsed quickly in five different beakers containing distilled water and gently blotted with paper in order to remove the surface solution.

The material was weighed again ($m_p$) to calculate the mass loss (ML) or weight loss. The mass loss (ML) is defined as the net loss in weight by the potato material on an initial potato weight basis:

$$ML = \frac{m_p - m_p^{\circ}}{m_p^{\circ}} \times 100 \qquad [1]$$

The slices were then divided in three sets of six slices. Each set was used for three replicates (2 slices each) of either, total solids measurements or, density determination or, sugar content and insoluble solids measurements.

## Measurement of Total Solids

Amount of total solids was determined gravimetrically by air drying at 105 °C for a 24 h period. Two slices were transferred to a preweighed aluminum dish, weighed and dried in the oven. After cooling in a dessicator under vacuum, the dish was reweighed and the total solids (TS) were calculated.

## Density Determination

The volume displacement technique was used in order to determine the density. Weight and volume were measured by weighing two disks in a preweighed empty pycnometer, filling up with water and reweighing. From the weight of the empty pycnometer and the volume of the pycnometer, predetermined by weighing the pycnometer filled with water at 23 °C, the weight, the volume, and subsequently the density ($\varrho$) of the disks were calculated. The volume of the empty pycnometers was determined as an average of 5 measurements.

## Measurement of Sucrose Content

### LC System

The HPLC system consisted of a Bio-Rad model 1330 pump (Bio Rad, Richmond, CA) and a Waters Associates model R401 differential refractometer (RI) detector. The system was equipped with a 20mL Rheodyne loop injector. A Bio-Rad Aminex HPX-87H ion exclusion column (300 × 7.8 mm i.d.) was used for the separation of sugars; the column being protected by a 40 × 4.6 mm guard column. Quantitation was performed electronically with a Hewlett-Packardd 3388A integrator.

### LC Separation Conditions

The mobile phase used was 0.01 N $H_2SO_4$. The solution was prepared using an analytical grade sulfuric acid obtained from Fisher Scientific and LC-grade water which was prepared by reverse osmosis (Milli-RO) and further purified by using a (Milli-Q) system (Millipore, Bedford, MA). After degassing, the mobil phase was used for LC analysis with a flowrate of 0.8mL/min at ambient temperature. Standard solutions of mixtures of analytical grade sugars (fructose, glucose and sucrose) ranging from 1–10 mg/mL were run into the system.

### Extraction and Measurement of Soluble Sugars

Potato slices (approximately 4g or 2 disks) were crushed with a mortar and pestle. The grindings were placed into a preweighed 50 mL centrifuge tube and weighed prior to homogenization.

Extraction of sucrose, glucose and fructose was achieved by boiling the sample with double the volume of 80% ethanol for 15 min. Supernatant was poured off and vacuum filtered through a preweighed Whatman #4 filter paper with a Buchner funnel.

A fresh volume of 80% ethanol was added to the grindings for a second 15-min extraction. The extraction and the residue were poured off and vacuum filtered. The residue was well washed with 80% ethanol. The filtrate was made up to volume with distilled water either in a 200 mL volumetric flask (for osmotic treatment with a 60% sucrose solution) or in a 100mL volumetric flask (for osmotic treatment with 10–40% sucrose solution). A certain volume of this extraction solution was filtered through a 0.45 $\mu$m millipore membrane and an aliquot was injected into the HPLC system for the quantitation of sugars. The sugar content (SC) of the sample was calculated by comparing the concentration of the standards to the concentration of the unknown sample taking into account the dilution factor and the mass of the sample ($m_p$).

### Measurement of Insoluble Solids

The residue of the extraction was transferred with the preweighed filter paper from the buchner funnel into a preweighed aluminum dish to be air dried in the oven at 105 °C for 24 h. After cooling in a dessicator under vacuum the dish was reweighed and the insoluble solids content was calculated (IS) on the basis of the actual weight of potato material.

## RESULTS

### Experimental Equilibrium Parameters of Potato Tissue as a Function of the Sucrose Concentration of the Osmotic Solution

The experimental measurements of the mass loss (ML), the density ($\varrho$), the total solids (TS), the sugar content (SC), the insoluble solids (IS) are listed in Table 1. A statistical analysis was performed for the mass loss (ML), total solids (TS), density ($\varrho$), sucrose content (SC) and insoluble solids (IS) using an Analysis of Variance (ANOVA) coupled with a Duncan Multiple Range Test (DMRT) at 1% level and 5% level of significance (Marcotte 1988).

No significant difference arose in the mass loss values with 30% and 40% osmotic solutions at the 5% confidence level, all others being significant. Among the treatments, only the treatment in a 60% sucrose solution was found to be significantly different from the others at the 1% level.

The data for total solids and density of samples untreated and treated in a 5% osmotic solution seemed to indicate that there was equilibrium between the fresh potato material and the osmosed samples. There was no significant difference between the density of the fresh material, the density of the potato soaked in a 5% sucrose solution as well as the density of the slices after a treatment in a 10% osmotic solution at 1% level. For $\alpha = 0.05$, there was no significant difference between the density of the fresh material and the potato slices soaked in a 10% osmotic solution. The total solids of the material in a 5% osmotic treatment and the fresh material were not statistically significantly different for both confidence levels. Similar experimental results for total solids have been observed by Lenart and Flink (1984a).

The insoluble solids (IS) were found to be statistically constant for any treatment. This is consistent with the literature (Lenart and Flink 1984a). Although no measurements of this kind were published it was always a basic assumption. The mass of insoluble solids remaining constant allowed for the following statement to be made: there was no leakage of solids from the cellular volume, indicating a minimum damage to the cells. This supported the fact that the osmotic water removal process is usually considered as a mild treatment compared to any other conventional drying operation.

TABLE 1.
EXPERIMENTAL MEASUREMENTS OF MASS LOSS (ML), DENSITY ($\varrho$), TOTAL SOLIDS (TS), SUGAR CONTENT (SC) AND INSOLUBLE SOLIDS (IS) OF POTATO SLICES IN EQUILIBRIUM WITH AN OSMOTIC SOLUTION AT 40°C

| Sucrose Solution (%) | Mass Loss (ML) (%)** | Density ($\rho$) ($kg/m^3$)* | Total Solids (TS) (%)* | Sugar Content (SC) (%)* | Insoluble Solids (IS) (%)* |
|---|---|---|---|---|---|
| Blank[x] | -- | 1058±6 | 15±1 | ≅0.23 | 13±2 |
| Fresh | NM | 1090±10 | 21±1 | ≅0.3++ | NM |
| 5 | -30±2 | 1080±20 | 19±3 | NM | NM |
| 10 | -32.6±0.8 | 1098±6 | 26±2 | 4.6±0.4 | 19±4 |
| 20 | -30±2 | 1132±9 | 32±1 | 10±1 | 19±1 |
| 30 | -28.4±0.6 | 1171±4 | 39.6±0.9 | 17±4 | 19±1 |
| 40 | -28±1 | 1210±10 | 45.8±0.7 | 23.5±0.2 | 20±1 |
| 60 | -46±1 | 1310±10 | 65.3±0.6 | 33.8±0.8 | 24±1 |

x Blank is the full turgor tissue after soaking in distilled water for at least 20 hours prior to any treatment.
++ From the literature.
* These are % of current weight
** These are % of initial weight

Based on the experimental measurements, the following equilibrium parameters have been calculated: water loss (WL) and sugar gain (SG).

As described by Lenart and Flink (1984a), the water loss can be defined as the net loss of water from potato on an initial potato weight basis. It was calculated from the mass loss (ML), the total solids of the potato material at full turgor (TS°) and the total solids of the potato material after the osmotic treatment (TS):

$$WL = \left[\left[\left[\frac{ML}{100} + 1\right]\left[1 - \frac{TS}{100}\right]\right] - \left[1 - \frac{TS^{\circ}}{100}\right]\right] \times 100 \qquad [2]$$

The sugar gain (SG) is defined as the net uptake of sucrose by the potato material based on the initial weight of potato. It is a function of the mass loss (ML) and the sugar content of the material after the treatment (SC) since the amount of sucrose in the full turgor material was negligible:

$$SG = SC \times \left[\frac{ML}{100} + 1\right] \qquad [3]$$

These parameters are plotted against the sucrose concentration of the solution in Fig. 1 as well as the mass loss values (ML).

The mass loss profile appeared to go through a minimum value of 28% at around 30%–40% sucrose solution. The mass loss of the potato tissue with a 10% sucrose solution (32.6%) was greater than with a 40% sugar solution (28%).

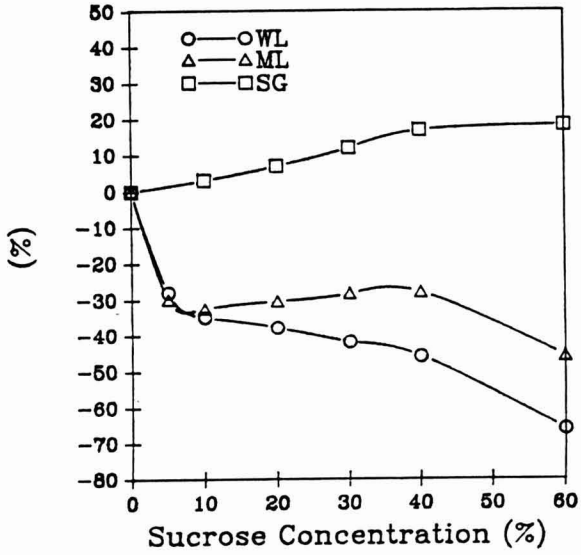


FIG. 1. EFFECT OF VARIOUS SUCROSE CONCENTRATION OF OSMOTIC SOLUTIONS ON EXPERIMENTAL EQUILBRIUM PARAMETERS OF THE POTATO SLICES AT 40°C

The water loss during an osmotic treatment varied from 28%–66% for a range of 10%–60% sucrose solution.

Figure 1 shows that the sugar uptake is significant. However, the sugar gain seemed also to slow down for a treatment in a solution between 40% to 60% sucrose solution.

The final amount of water loss and sugar gain were similar to those observed by Lenart and Flink (1984a).

## Experimental Extracellular and Cellular Volumes as a Function of the Sucrose Concentration of the Osmotic Solution

Considering that the plant tissue structure is made up of cells, the analysis of the equilibrium state of the potato tissue with an osmotic solution was transposed from the global description of the entire structure down to the analysis of the behavior of the cells.

A typical potato plant cell consists of a vacuole, a cell wall and a layer of cytoplasm between the vacuole and the cell wall. The plasmalemma which separates the cell wall from the cytoplasm controls the passage of substances from one compartment to another. The selective permeability of the membrane determines the osmotic behavior.

Data have been transformed in order to show the separate behavior of the total volume (V), the extracellular volume or free space ($V_i$) which comprises the cell wall and the intercellular space, and the cellular volume ($V_c$) which includes the vacuole bounded by the tonoplast, the cytoplasm and the membrane. At full turgor, cells of potato tubers are closely packed but there is a certain amount of intercellular space (Woolley 1962). The porosity of potatoes at full turgor is low, between 1 to 3% (Crapiste and Rotstein 1982).

The concept of free space has been discussed by Salisbury and Ross (1969). They indicated that the apparent free space was the portion of the volume of a tissue which was in equilibrium with an outside medium. They also suggested a calculation method for the estimation of the apparent free space. Since sucrose is nonpermeating to the membrane, it was only present in the intercellular space and the spaces of the cell wall. At equilibrium, the liquid phase in the free space or extracellular volume was assumed to be at the same concentration as the concentration of the osmotic solution.

The experimental total volumes were calculated from the mass loss (ML) and the density of the potato tissue at full turgor ($\varrho^\circ$) and after the osmotic treatment ($\varrho$). The proportion of total volume based on the initial volume at full turgor was determined by:

$$\frac{V}{V^\circ} = \left(1 + \frac{ML}{100}\right) \times \frac{\rho^\circ}{\rho} \qquad [4]$$

From the sucrose content measurements (SC) the proportion of volume of the extracellular space was calculated based on the initial volume at full turgor:

$$\frac{V_i}{V^\circ} = \frac{\frac{SC}{100}\left[1 + \frac{ML}{100}\right]}{\rho_s} \times \rho^\circ \qquad [5]$$

$\varrho_S$ is the volumetric mass of sucrose for a particular solution available in the literature (Weast *et al.* 1984).

By difference the cellular volume, as a proportion of the initial total volume, was calculated:

$$\frac{V_c}{V^\circ} = \frac{V - V_i}{V^\circ} \qquad [6]$$

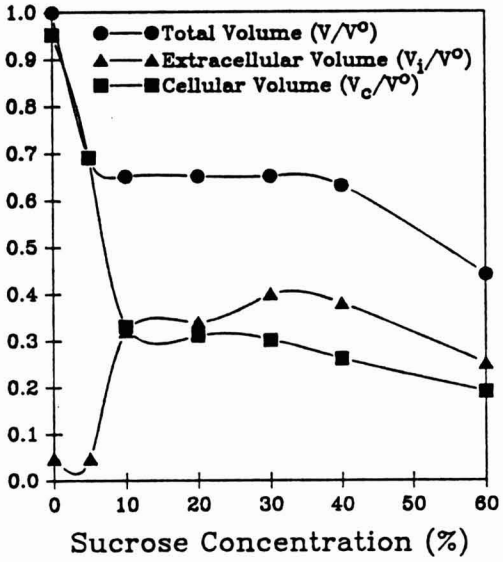


FIG. 2. INFLUENCE OF SUCROSE CONCENTRATION OF THE OSMOTIC SOLUTION ON EXPERIMENTAL EQUILBRIUM VOLUME CHANGES OF THE POTATO SLICES AT 40°C

In Fig. 2, the total volume ratio plotted against the concentration of the osmotic solution gives at first, a straight line which bends sharply to the horizontal for a wide range of concentration and then decreases again for highly concentrated sucrose solutions.

Figure 2 shows that the extracellular volume ratio ($V_i/V°$) was constant from 0 to $\approx$ 10% sucrose solution where the extracellular space increased sharply and then slightly for higher concentrations.

The cellular volume ratio ($V_c/V°$) of the tissue decreased. Since the loss of water can only come from the cellular volume (i.e., sucrose is nonpermeating to the plasmalemma membrane) this type of profile was expected.

Figure 2 confirms that the behavior of the tissue can be explained from the behavior of the cells. From a cell point of view, the point at which the total volume levels off is considered as the point at which incipient plasmolysis or isotonicity takes place (Noggle and Fritz 1976). In this particular case, it seems to occur at around 5% to 10% sucrose. By plasmolysis, it is meant the separation of the protoplast of a plant cell from the cell wall, due to the efflux of water from the cell.

Qualitatively, this phenomenon has been observed by plant physiologist at the cellular level. Noggle and Fritz (1976) reported that a close observation under a microscope of a single cell upon immersion in an osmotic medium revealed that the cell looses its turgidity in a short time; the volume of the protoplast, which

comprises the vacuole, the tonoplast and the cytoplasm, decreases; and the protoplast separates away from the wall. The space between the protoplast and the cell wall becomes filled with the plasmolyzing solution. The extent to which the cell plasmolyzed is a function of the concentration of the osmotic solution.

## DISCUSSION

### Comparison Between the Experimental Equilibrium Parameters and the Corresponding Calculated Parameters

The proposed method for water accounting in the biological structure is based on the fact that the chemical potential is the most suitable parameter describing the state of water in any system particularly the equilibrium state of a multiphase system such as a plant cell with the environment (Dainty 1976). Restricting the treatment to nonelectrolytes with the reference state of pure water at the temperature under consideration and atmospheric pressure, the water chemical potential of a vegetable system is defined as:

$$\mu_W - \mu_W^\circ = RT \ln \hat{a}_W + V_W \varphi_m + V_W(P - P^\circ) \qquad [7]$$

The energy state of water is split in three major components. The first term, $RT \ln \hat{a}_W$, or osmotic potential, reflects the contribution of dissolved solutes to the chemical potential of water. The second term, $V_W \varphi_m$, or matrix potential, arises because of strong interactions between water and solids and large area of interface present in the system and the final term, $V_W( P - p^\circ)$ expresses the dependence of the chemical potential on hydrostatic pressure.

Considering the vegetable system in equilibrium with an osmotic solution:

$$\mu_W - \mu_W^\circ = RT \ln \hat{a}_{WO} \qquad [8]$$

The freezing point depression ($\Delta$) of the solutions allowed for the calculation of the water activity for each of the osmotic solution by the following equation (Wall 1974).

$$-\log \hat{a}_{WO} = 4.209 \times 10^{-3}(\Delta) + 0.215 \times 10^{-5}(\Delta)^2 \qquad [9]$$

which has to be corrected for the temperature (Marcotte 1988):

$$\ln \frac{\hat{a}_{WO}^T}{\hat{a}_{WO}^{To}} = 0.010146\, m_s^2 \ln \frac{T}{To} \qquad [10]$$

where $m_S$ is the molality of sucrose solution, To is the freezing temperature, T is the temperature at which the water activity ($\hat{a}_{wo}^{T}$) is required and $\hat{a}_{wo}^{To}$ is the water activity at the freezing temperature (To).

The results are listed in Table 2 for each sucrose concentration of osmotic solutions. The values of water activity indicate that osmotic drying processes operate in the high moisture content region of an isotherm.

TABLE 2.
WATER ACTIVITY FOR THE OSMOTIC SOLUTION AT 40 °C

| Solution (%) | $\hat{a}_{wo}$ |
|---|---|
| 5 | 0.9972 |
| 10 | 0.9942 |
| 20 | 0.9867 |
| 30 | 0.9770 |
| 40 | 0.9634 |
| 60 | 0.8956* |

*The value of water activity for a 60% sucrose solution has been obtained from a correlation based on experimental data of activity coefficient for the sucrose-water system, at 25 °C (Hougen *et al.* 1954). The value was corrected for the temperature using eq. [10].

Assuming that the potato tissue is in equilibrium with an osmotic solution of known $\hat{a}_{wo}$, the total moisture content (X) of the potato tissue on a dry weight basis is estimated from the Hasley (1948) correlation.

$$\hat{a}_{wo} = \exp\left[\frac{-B}{RT}\,\frac{1}{X^{C}}\right] \qquad [11]$$

where: $B = 5.586 \times 10^4$; $C = 1.648$; $T = 313K$. The Hasley (1948) correlation was found to provide a good fit for the high moisture content region (Crapiste and Rotstein 1982).

A typical composition of the potato tissue was required in order to assign the proper weight for the contribution of different phases in the potato tissue. Table 3 reports the average composition of the potato tissue which was found to agree with the composition of the potato tubers (Russet Burbank variety from the northern part of Alberta) selected for the experimental work (Chung 1979).

The potato cell possesses a single large vacuole which may occupy over 90% of the total cell volume. This vacuole is a relatively dilute, homogenous aqueous phase. Minerals and soluble sugars contribute to the vacuole solution. The

TABLE 3.
SELECTION OF THE REPRESENTATIVE COMPOSITION OF THE DRY MATTER OF THE RUSSET BURBANK POTATO TUBERS

| Constituent | Composition (dry weight basis) |
|---|---|
| Starch | 0.68 |
| Proteins | 0.11 |
| Glucose | 0.02 |
| Fructose | 0.02 |
| Sucrose | 0.015 |
| $K_2SO_4$ | 0.015 |
| $K_3PO_4$ | 0.04 |
| Cellulose | 0.03 |
| Others | 0.07 |

solutes constitute the major contribution to the osmotic potential. From the composition of the potato tuber in Table 3, it can be seen that the main solute constituents are glucose, fructose, sucrose, $K_3PO_4$ and $K_2SO_4$. However, the concentration of the major solutes such as sucrose, glucose and fructose may vary tremendously due to storage conditions (Talburt *et al.* 1975).

The cytoplasm is a more complex phase containing many colloids and membrane-bounded organelles. In the cytoplasm phase, the effects of the numerous interfaces and colloidal materials are important. Thus the prevailing term is the matrix potential. Reserve materials such as starch and proteins are also present. Both are important consistuents of the cytoplasm. They represent almost 80% of the dry matter. Data for starch in equilibrium with moist air are available in the literature and they were correlated by Crapiste and Rotstein (1982).

$$\mu_w - \mu_w^\circ = RT \ln (1 - \exp(-53.4759 X_{st}^{2.3015})) \qquad [12]$$

Similar correlation was also developed for proteins in equilibrium with moist air by Crapiste and Rotstein (1982) based on the experimental sorption equilibrium data for proteins:

$$\mu_w - \mu_w^\circ = RT (-0.0208 X_{pr}^{-1.6129}) \qquad [13]$$

Assuming an equilibrium between the osmotic solution and the potato material (i.e., $\mu_w - \mu_w^\circ$ is equal to RT ln $\hat{a}_{wo}$), the water content of the starch ($X_{st}$) and the water content of proteins ($X_{pr}$) were calculated using Eq. [12] and Eq. [13].

Since the total water content was previously calculated using Eq. [11], the moisture content of the vacuole ($X_v$) was determined by difference from:

$$X_v = X - X_{st} w_{st} - X_{pr} w_{pr} \qquad [14]$$

Due to the findings of Crapiste and Rotstein (1982), the contribution of the cell wall phase was considered negligible. Since Rotstein and Cornish (1978) found that for the prediction of sorptional equilibrium relationship for apples and pressure potential of the cell could be neglected, this term was therfore not taken into consideration.

The value of the total moisture content ($X^\circ$) at full turgor was calculated from the total solids measurements at full turgor:

$$X^\circ = \frac{(1 - TS^\circ)}{TS^\circ} \qquad [15]$$

From the total water content at full turgor ($X^\circ$), the corresponding value of water activity ($\hat{a}_{wo}$) was calculated from Eq. [11] as being 0.9985. This value was used to calculate the water content of the starch ($X^\circ_{st}$) from Eq. [12] and proteins ($X^\circ_{pr}$) from Eq. [13], at full turgor. The water content of the vacuole ($X_v$) was obtained by difference using Eq. [14].

In order to compare with the experimental data, the water loss of the cell ($WL_c$) as well as the contribution of the different phases such as the vacuole and the cytoplasm with respect to the water loss were calculated. The mass of dry matter ($m_{dm}$) was assumed constant in the cell volume. This is a reasonable assumption since it is well known that sucrose is non-permeating to the cell membrane. The water loss of the potato cell ($WL_c$) was defined:

$$WL_c = \left[ \frac{X}{X^\circ} - 1 \right] w_w \times 100 \qquad [16]$$

$w_w$ represents the proportion of water of the full turgor material on a wet basis.

The water loss by the cytoplasm ($WL_{cy}$) was determined by:

$$WL_{cy} = \left[ \frac{X^\circ_{st} w_{st}}{X^\circ} \left[ \frac{X_{st}}{X^\circ_{st}} - 1 \right] + \frac{X^\circ_{pr} w_{pr}}{X^\circ} \left[ \frac{X_{pr}}{X^\circ_{pr}} - 1 \right] \right] w_w \times 100 \qquad [17]$$

and the water loss of the vacoule by:

$$WL_v = \left[ \frac{X_v^o}{X^o} \left[ \frac{X_v}{X_v^o} - 1 \right] \right] w_w \times 100 \qquad [18]$$

In Fig. 3, the calculated water loss of the potato cell ($WL_c$), the water loss by the cytoplasm ($WL_{cy}$) and the vacuole ($WL_v$) are plotted against the sucrose concentration of the osmotic solution and compared with the experimental results of mass loss (ML), water loss (WL), sugar gain (SG).

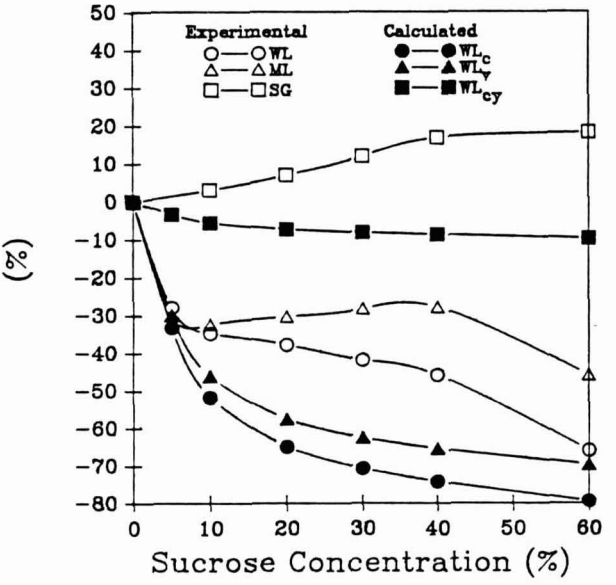


FIG. 3. COMPARISON BETWEEN THE EXPERIMENTAL AND CALCULATED PARAMETERS AT VARIOUS SUCROSE CONCENTRATION OF THE OSMOTIC SOLUTION, IN EQUILIBRIUM, AT 40 °C

From full turgor to around 10% sucrose solution, the experimental water loss and mass loss were similar to the calculated ones. The contribution of the vacuole was dominant compared to the contribution of the cytoplasm.

From a 10% to a 60% osmotic solution, there is an important discrepancy between the calculated and experimental water and mass loss. The uptake of sugar is so important that the discrepancy between the calculated and experimental water and mass loss is significant. However, not only sucrose is gained by the biological structure, but also the water of the sucrose solution enters, as well, in-

to the potato material. Consequently, the experimental water and mass loss are less than the calculated ones.

Assuming that, at equilibrium, the extracellular space is filled up with the osmotic solution, it is possible to calculate the water gain due to the fact that the sucrose solution penetrates the extracellular space. From the sugar content measurements, the water gain by the extracellular space ($WG_i$) is calculated as:

$$WG_i = (SG \times 100 / \%OS) - SG \quad [19]$$

The total calculated water loss (WL) is therefore equal to the water loss by the cell volume plus the water gain by the extracellular volume:

$$WL = WL_c + WG_i \quad [20]$$

The calculated mass loss is therefore calculated in the following manner:

$$ML = WL_c + WG_i + SG \quad [21]$$

In Fig. 4, the experimental mass and water losses as well as the calculated mass and water losses are represented. When considering the total calculated water loss which represents the sum of the water loss by the cell volume and the water gain by the extracellular space due to the penetration of the sucrose solution in the extracellular volume, there was no discrepancy between the experimental and the calculated water loss. Furthermore, the experimental and calculated mass losses were found to be in good agreement.

## Comparison Between the Experimental Cell Volume and the Corresponding Calculated Cell Volume

The changes occurring in the different phases of the potato cell were investigated and compared with the experimental changes observed in the potato tissue. In order to fully understand and explain the phenomenon, the behavior of different phases of the cell were considered in terms of volumes and compared with the experimental volumes.

The calculated cell volumes were determined on a dry matter basis:

$$\frac{V}{m_{dm}} = \frac{X}{\rho^*_w} + \frac{1}{\rho^*_{dm}} \quad [22]$$

The volume of the cytoplasm was calculated in the following manner:

$$\frac{V_{cy}}{m_{dm}} = \left[ \frac{X_{st} w_{st}}{\rho^*_w} + \frac{w_{st}}{\rho^*_{st}} \right] + \left[ \frac{X_{pr} w_{pr}}{\rho^*_w} + \frac{w_{pr}}{\rho^*_{pr}} \right] \quad [23]$$

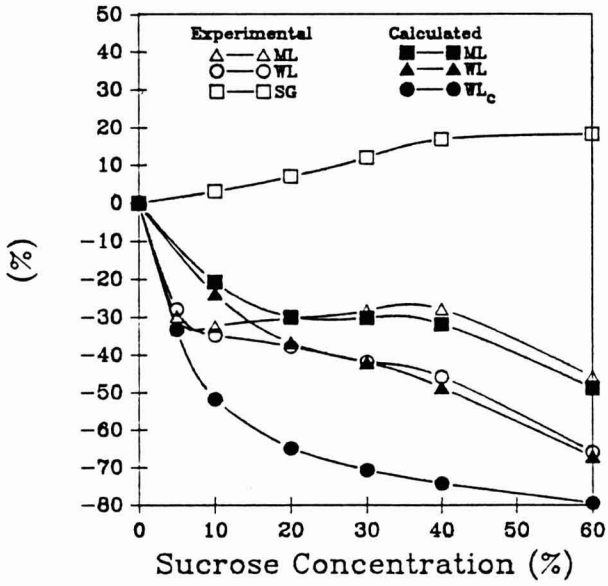


FIG. 4. COMPARISON BETWEEN EXPERIMENTAL AND CALCULATED MASS AND WATER LOSSES AT VARIOUS SUCROSE CONCENTRATION OF THE OSMOTIC SOLUTION, IN EQUILIBRIUM, AT 40°C

Finally, the volume of the vacuole was estimated by difference:

$$\frac{V_v}{m_{dm}} = \frac{V}{m_{dm}} - \frac{V_{cy}}{m_{dm}} \qquad [24]$$

The density of starch ($\varrho^*_{st}$), proteins ($\varrho^*_{pr}$) and other components of the potato tissue were found in the literature (Peleg and Bagley 1983). The average density of dry matter ($\varrho^*_{dm}$) was calculated from the composition given in Table 3 and the density of individual components.

In Fig. 5, the volumes are reported as a proportion of the initial total volume (full turgor material) (i.e., total volume $V/V°$, volume of the cytoplasm $V_{cy}/V°$ and volume of the vacuole $V_v/V°$) and plotted against the sucrose concentration of the osmotic solution. They are compared with the cellular volume obtained from the experimental data. Figure 5 shows that from full turgor to incipient plasmolysis the vacuole has undergone the major decrease in volume. In solutions of concentration higher than 20% sucrose, the volume of the vacuole was smaller than the volume of the cytoplasm. Figure 5 shows also that the experimental cellular volume of the tissue has matched the calculated cell volume.

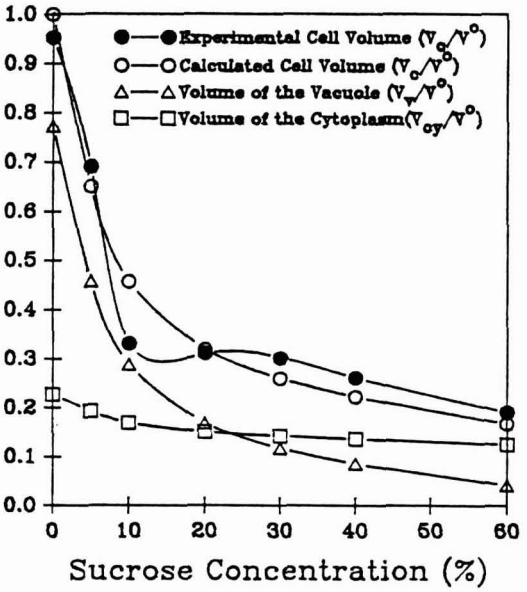


FIG. 5. EFFECT OF SUCROSE CONCENTRATION OF THE OSMOTIC SOLUTION ON CALCULATED AND EXPERIMENTAL VOLUME CHANGES IN EQUILIBRIUM AT 40°C

The behavior of the lumped cellular volumes of the potato material is well predicted by the calculated cell volumes from the sorption data. This implies two major consequences. First, sucrose is truly nonpermeating, that is, it does not enter the cellular volume of the tissue since the calculations of the cell volumes account for the water transport only. Second, the water loss by the cellular volume of the tissue can be quantified, from a cell point of view, taking into account the contribution of two important phases present in the cellular volume: the vacuole and the cytoplasm. Consequently, one may infer the repartition of water and sugar in the cellular volume of the potato material from the proposed thermodynamic approach.

In summary, a picture of the structural changes of a plant cell undergoing an osmotic treatment is represented on Fig. 6. Starting at full turgor, the extracellular space of the cell is minimum. The cellular volume pushes the walls (Fig. 6a). As water is lost from the cellular volume the total volume is affected whereas the extracellular volume stays constant.

As soon as incipient plasmolysis or isotonicity (Fig. 6b) is reached, any further loss in cellular volume is compensated by a proportional increase in extracellular space (Fig. 6c).

Up to this point, this typical shrinkage behavior applies to most conditions of an osmotic treatment of the tissue, as seen on Fig. 2. According to Stadelman

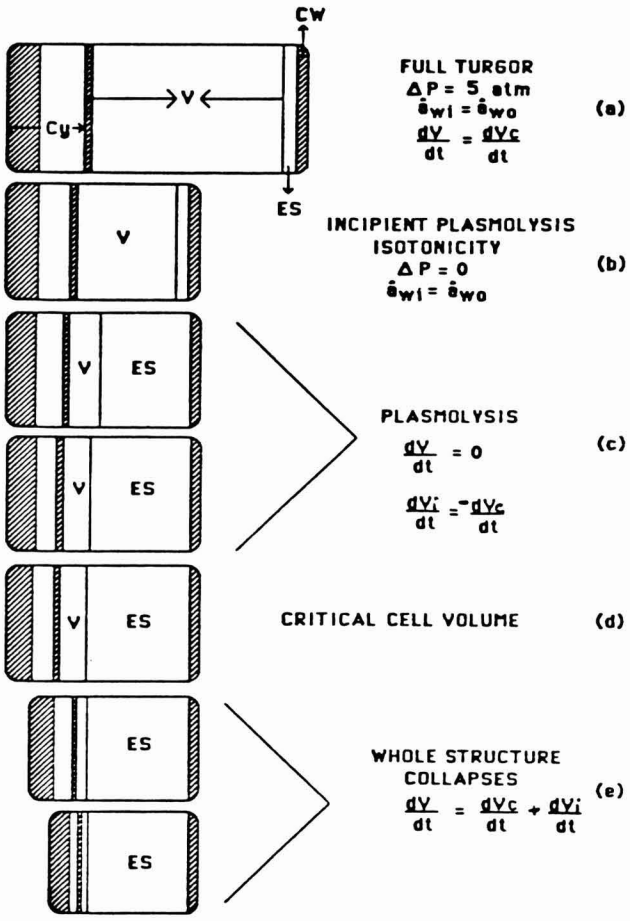


FIG. 6. PHYSIOLOGICAL PREPRESENTATION OF THE SHRINKAGE BEHAVIOR

(1966), cells with highly stretchable walls could exhibit a ratio of total volume at full turgor to total volume at incipient plasmolysis as high as 1.50. This corresponds to the experimental total volume ratio of 1.54 observed here.

Figure 2 shows also that the total volume is significantly smaller for the potato tissue in equilibrium with a 60% sucrose solution. A loss of the integrity of the cells seems to happen. The extracellular volume is also smaller as well as the cellular volume (Fig. 6d, e).

## SUMMARY

A model has been developed based on the equality of water activity between the osmotic solution and the potato material. Using the data on the cellular structure and the composition of the potato material, the equilibrium state of the potato material in sucrose solution in the range of 5%–60% was quantified.

When mass loss (ML), water loss (WL) and sugar gain (SG) were considered and compared with the calculated water loss, the water loss by the cytoplasm and the water loss by the vacuole, some discrepancies were found from around 10% to 60% sucrose solution due to the uptake of sucrose and water as a solution by the potato material. Assuming that the movement of sucrose is restricted because of the selective permeability of the cell plasmalemma membranes, the repartition of water and sucrose in the outer space (lumped extracellular volumes) and in the inner space (lumped cellular volumes) of the tissue was done. The resulting experimental total, extracellular and cellular volume changes were calculated.

A comparison between the experimental volume changes and the predicted volume changes was done. The proposed method was able to predict in good agreement the behavior of the experimental lumped cellular volumes of the potato tissue providing a better understanding regarding the mechanisms of osmosis.


## NOMENCLATURE

| | |
|---|---|
| $\hat{a}_w$ | activity of water in the vacuole solution (J/kmol) |
| B | constant, Eq. [11] |
| C | constant, Eq. [11] |
| IS | insoluble solids (%) |
| m | mass (kg) |
| $m$ | molality of sucrose solution (kmol/1000kg of water) |
| ML | mass loss (%) |
| %OS | sucrose concentration of the osmotic solution (kg/kg) |
| P | hydrostatic pressure ($N/m^2$) |
| R | universal gas constant (J/kmol K) |
| SC | sucrose content (%) |
| SG | sugar gain (%) |
| T | temperature (K or °C) |
| To | freezing point temperature (°C) |
| TS | total solids (%) |
| V | Volume ($m^3$) |
| $\overline{V}$ | partial molar volume ($m^3$/kmol) |
| WG | water gain (%) |

| | |
|---|---|
| WL | water loss (%) |
| w | weight fraction on a dry weight basis (kg/kg) |
| $w$ | weight fraction on a wet basis (kg/kg) |
| X | water content on a dry weight basis (kg/kg) |
| Y | sucrose concentration (mg/mL) |

## GREEK SYMBOLS

| | |
|---|---|
| $\alpha$ | level of confidence |
| $\Delta$ | freezing point depression (°C) |
| $\mu$ | chemical potential (J/kmol) |
| $\varrho$ | density (kg/m$^3$) |
| $\varphi_m$ | matrix potential (N/m2) |

## SUBSCRIPTS

| | |
|---|---|
| c | cellular volume |
| cy | cytoplasm |
| dm | dry matter |
| i | extracellular volume |
| o | osmotic solution |
| p | potato |
| pr | proteins |
| s | sucrose |
| st | starch |
| v | vacuole |
| w | water |

## SUPERSCRIPTS

| | |
|---|---|
| * | referred to the pure component |
| o | referred to a reference state either full turgor potato material or pure water at the temperature and atmospheric pressure. |

## ACKNOWLEDGMENTS

The authors express their thanks to Dr. Thanh Nguyen for her help in the HPLC analysis of sugars. This project was supported by a study grant from FCAR (Fonds pourla Formation des Chercheurs et l'Aide a la Recherche), Government of Quebec, Canada and a grant from NSERC.

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# DRYING OF GRAINS IN A DRAFTED TWO DIMENSIONAL SPOUTED BED

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Accepted for Publication November 3, 1990

## ABSTRACT

*A two-dimensional spouted bed with draft plates for grain drying was tested using soybean, wheat and corn. Drying rate and material temperature were measured as functions of the dryer geometry and operating parameters. The hypothesis of intermittent drying, consisting of a heating period in draft channel and a tempering period in the downcomer was verified. The drying rate was found to be related to the measured rate of solids circulation. For mathematical description of the drying process in the configuration investigated, the Page's equation for thin layer drying of grains was used. The two parameters in this model were correlated versus the bed geometry and the operating parameters of the spouted bed.*

## INTRODUCTION

A drafted two-dimensional spouted bed (TDSB) is one of the several possible modifications of the classical spouted bed (Mujumdar 1984, Passos *et al.* 1987) which allows reduction of the pressure drop, improved solids circulation while overcoming to some extent, the scale-up problems by extension of the pilot apparatus along the bed depth (length in the direction of scale-up)(Mujumdar 1984, Mujumdar 1989).

A two-dimensional spouted bed is usually formed in a rectangular chamber with a slanted base and "draft" (by analogy with draft tubes in clyindrical spouted beds) plates centered above the gas inlet. Particles entering the jet region are coveyed between the draft plates by a high velocity gas stream and form a characteristic fountain above the bed surface, and after separation from the gas stream (aided frequently by deflector plate) fall as a moving bed towards the gas jet (Fig. 1.).

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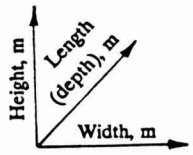
Journal of Food Process Engineering *13(1991) 321–332. All Rights Reserved.*

| | Laboratory scale | Pilot scale |
|---|---|---|
| L , m | 0.04 | 0.15 |
| w , m | 0.5* | 0.75* |
| H , m | 1.0 | 2.0 |
| $H_d$ , m | 0.9* | 1.35* |
| $H_0$ , m | 0.1* | 0.2* |
| $w_d$ , m | 0.07* | 0.1* |
| $w_i$ , m | 0.033* | 0.05* |
| s , m | 0.05* | 0.15* |
| θ , ° | 60 | 60 |



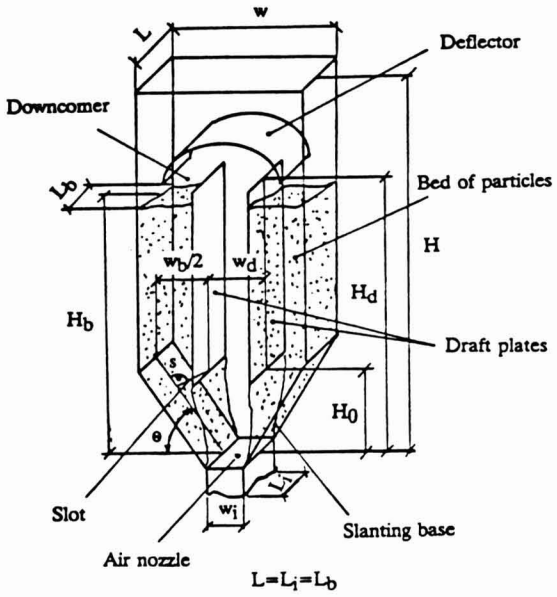




FIG. 1. SCHEMATICS OF THE TWO-DIMENSIONAL SPOUTED BEDS WITH DRAFT PLATES (*indicates the maximum dimension)

Previous studies on drafted two dimensional spouted beds covered a broad spectrum of hydrodynamic aspects with special reference to stability of their operation at optimum conditions (Swaminathan and Mujumdar 1984, Kalwar *et al.* 1986, Kalwar *et al.* 1988a, Kalwar *et al.* 1988b) as well as heat transfer in a gas-solid system (Kudra *et al.* 1989). This paper aims to evaluate the performance of the two dimensional spouted bed as a dryer for grains.

## MATERIALS AND METHODS

The experiments were carried out in laboratory as well as pilot scale two-dimensional spouted bed units with their geometries designed to ensure optimal hydrodynamics of the bed at the operating conditions appropriate for the drying process (Kalwar *et al.* 1986, Kalwar *et al.* 1988a, Kalwar *et al.* 1988b). Both dryers were made of 0.01 m thick plexiglas to visualize the solids flow pattern. A special design of the drying chambers with movable walls allowed us to vary the bed height, width and depth (length in the direction of scale-up) as well as the height and spacing of the two vertical draft plates installed centrally above the gas inlet. To eliminate potential stagnation zones, the slanting base plates were

inclined at 60 °(Passos *et al.* 1987). A parabolic deflector was mounted above the draft plates to limit the spout height and aid the solids circulation by directing the particles from the spout to the downcomer. The rectangular air inlet nozzle was covered with 0.001 m wire mesh.

Three evenly spaced holders fitted with fast-response thermocouples for sampling and solids temperature measurement, were mounted in the downcomer zone along the height of the draft plate; one near the top of the plate, one near the air inlet nozzle and one about midway between the two. The inlet and outlet gas temperatures were monitored continuously using K-type thermocouples (Omega Engineering Corp.) located at the gas slot below the wire mesh and at the deflector level. The wall temperature (required for heat loss calculation) was measured by two thermocouples embedded 0.5 mm in the downcomer wall and attached to it by a silver epoxy. The temperature at all measuring points was recorded with an OM-302 temperature logger (Omega Engineering Corp.) with a rated accuracy of $\pm 0.5$ °C. The air supplied by a high pressure blower was maintained at pre-selected temperatures by an electric heater fitted with an autotransformer and a temperature controller. A Dwyer 5106 thermal anemometer (accuracy $\pm 3\%$) was used to measure the air velocity in the air inlet nozzle. Air humidity at the dryer inlet and outlet was monitored continuously by a Vaisala HMI 32 Humidity Meter with an accuracy of $\pm 1.5\%$ of the relative humidity and verified periodically with a pre-calibrated 1200 APS Dewpoint Hygrometer (General Eastern). The grain moisture content was determined gravimetrically following the ASAE standard (Agricultural Engineering Yearbook 1982).

Freshly harvested wheat, soybean and corn with properties as specified in Table 1, were used as test materials in 56 runs. To obtain the desired initial moisture content in experiments with initial moisture content as a variable (8 runs), a given mass of wheat was sprayed with tap water and stored hermetically at ambient temperature. A conditioning period of 48 h with thorough mixing was found to be sufficient for reproducible water absorption and uniform moisture distribution within the grain.

The drying kinetics were measured in the batch mode. Each run was started with an initial period of hydrodynamic and thermal stabilization by spouting a dry grain load of equivalent mass. When all monitored parameters remained constant for 20 min the grain load was replaced with the desired mass of wet material. During experiments the samples for moisture content determination were taken at five minute intervals for periods of 50–90 min, depending on the inlet air temperature. Values of other parameters were noted at intervals of two minutes. The inlet air temperature was varied at three levels, viz. 50, 70 and 90 °C. The gas velocity in the air inlet nozzle required for stable operation of a TDSB was 10.7 - 16.7 m/s for wheat, 19.1 m/s for soybean and 19.1 -21.5 for corn. The range of operating parameters is specified in Table 2.

TABLE 1.
CHARACTERISTICS OF GRAINS USED

| Parameter | Corn (Yellow dent) | Soybean (Maple arrow) | Wheat (Laval-19) |
|---|---|---|---|
| Equivalent sphere diameter, m | 0.00969 | 0.00877 | 0.00486 |
| Sphericity[1], - | 0.707 | 0.827 | 0.593 |
| Angle of repose,$^o$ | 29.2 | 33.3 | 28.2 |
| Density (dry),kg/m$^3$ | 1397 | 1210 | 1440 |
| Density (wet[2]),kg/m$^3$ | 1300 | 1130 | 1300 |
| Specific heat, J/(kgK) | 2401 | 2072 | 2198 |
| Hygroscopic moisture content[3], % wb | 23.8 | 25.8 | 25.6 |
| Initial moisture content, % wb | 21-25.9 | 22.9 | 13-36.6 |

1. Volume of solid/volume of circumscribed sphere (Mohsenin, 1970)
2. For fresh harvested grains
3. At T = 25$^o$C (Pabis, 1982)

TABLE 2.
RANGE OF OPERATING PARAMETERS

| Parameter | Range |
|---|---|
| Inlet air temperature, °C | 50-90 |
| Gas velocity (at the inlet nozzle), m/s | 10.7-21.5 |
| Gas velocity / minimum spouting velocity, - | 1.1-1.5 |
| Static bed height, m | 0.33-1.35 |
| Bed width, m | 0.3-0.75 |
| Spout width, m | 0.05-0.1 |
| Slot width, m | 0.035-0.15 |
| Width of air nozzle, m | 0.033-0.05 |
| Air nozzle / draft channel aspect ratio, - | 0.5-0.8 |
| Drying time, min | 50-90 |
| Mass of wet material, kg | 2-33 |

## RESULTS AND DISCUSSION

Inlet and outlet air humidity measurements yielded a drying curve that was within ±9% of that one obtained from gravimetrically determined grain moisture content. Therefore, the data used for analysis in this work were based only on the gravimetric method. Figure 2 shows a representative plot of the grain moisture content, outlet air temperature and grain temperature versus drying time. One general observation from these data is that the kinetics of grain drying in a drafted two-dimensional spouted bed follow the typical kinetics for convective drying, e.g., a constant drying rate period precedes a falling drying rate period if the initial moisture content is above the critical one. In the constant rate drying period, the rate of drying does not depend on the initial moisture content. The distinct straight line which appears for short period at initial moisture contents below the critical value can be attributed to evaporation of water from the near-surface region which is continuously moistened in the tempering period in the downcomer due to levelling of the moisture profile. This line, however, is not representative of the first drying rate period in the sense of free surface water evaporation since in this period the material temperature rises continuously, and always exceeds the wet bulb temperature (20–25 °C) corresponding to the inlet air conditions. Typically the grain temperature was found to be noticeably lower (about 10 °C) than the outlet air temperature in contrast to spouted beds without draft plates where this difference was 2–3 °C only. This means that no thermal equilibrium between the air and grains is attained in the spout. It implies further that the contact time in a drafted spout bed (even of industrial scale) is too short to utilize effectively the sensible heat of the gas stream. In a "drafted" bed, however, because of regular grain circulation, the drying is more uniform than in conventional spouted beds where significant variation in the solids moisture content has been observed (Viswanathan and Lyall 1984).

As expected, there is no measurable influence of gas velocity on the drying kinetics of grains (Fig. 3) since the mass transfer rate is internally controlled (Biot number for mass transfer is in the order of $10^6$ – $10^7$). Further, even for grains with high initial moisture content this influence is found to be negligible. The possible reasons are: (1) The time spent by grains between draft plates is very short; under experimental conditions the transit time in the draft channel is in the order of 0.3–0.5 s whereas the time spent in the downcomer is in the order of 13–110 s. Thus particles which accelerate from zero velocity in the slot region do not reach their terminal velocity in the draft channel. Under these conditions the particles are always subjected to a turbulent flow of air stream with large relative velocity between the grain and air, and a further increase in gas velocity does not affect noticeably the rate of moisture evaporation (Henderson and Pabis 1962); (2) The downcomer region is affected by the gas flow only to a small extent since the major part of the gas stream (estimated as 80–95%) flows through the draft channel (Kalwar *et al.* 1988a).

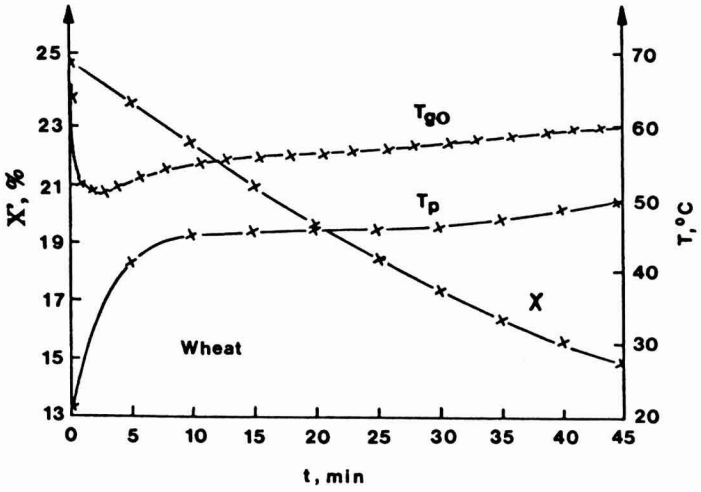



FIG. 2. DRYING AND TEMPERATURE CURVES FOR WHEAT

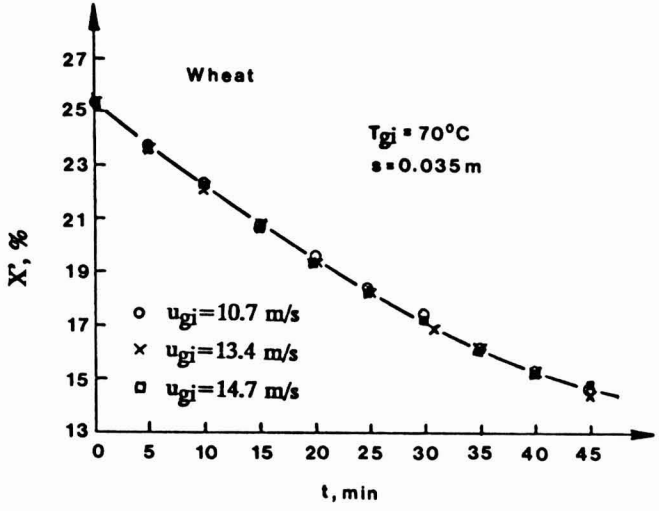



FIG. 3. EFFECT OF AIR VELOCITY ON DRYING RATE

Figure 4 displays a set of drying curves for wheat with the inlet gas temperature as a parameter. The inlet gas temperature increases the drying rate throughout the whole drying period. This effect is especially significant during the final stage of drying when the release of moisture from the receded evaporation front requires a large temperature difference between the material surface and evaporation front.

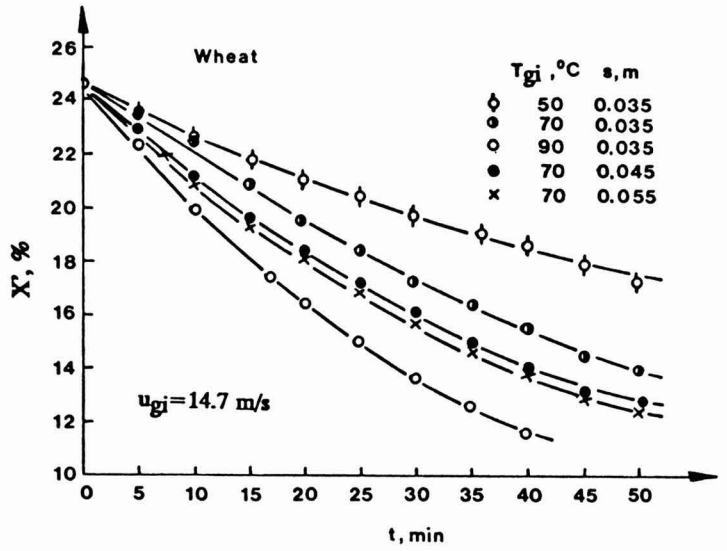



FIG. 4. EFFECT OF AIR INLET TEMPERATURE AND SLOT WIDTH ON DRYING RATE FOR WHEAT

The drying rate is noticeably affected by the slot width, i.e., the spacing between the draft plates and the inclined side wall. As can be seen from Fig. 4 and 5, a wider slot results in higher drying rates under the otherwise identical operating conditions (the limiting slot width which adversely affects the bed hydrodynamics was determined to be 0.25 m). In the configuration studied, the drying material is subjected to the hot air stream primarily during its transport between the draft plates since a major fraction of a gas stream flow through the central channel formed by the draft plates. Because the Luikov number (the ratio of mass to heat diffusivities) for grains is low (in the order of 0.001–0.1), the sensible heating of grains and moisture evaporation occur between the draft plates. In the downcomer, where descend particles are exposed to a relatively low air flow, the tempering process takes place with moisture migration towards the grain surface and some evaporation due to the sensible heat gained during transit in the draft channel. Thus, a drafted TDSB operates in an intermittent

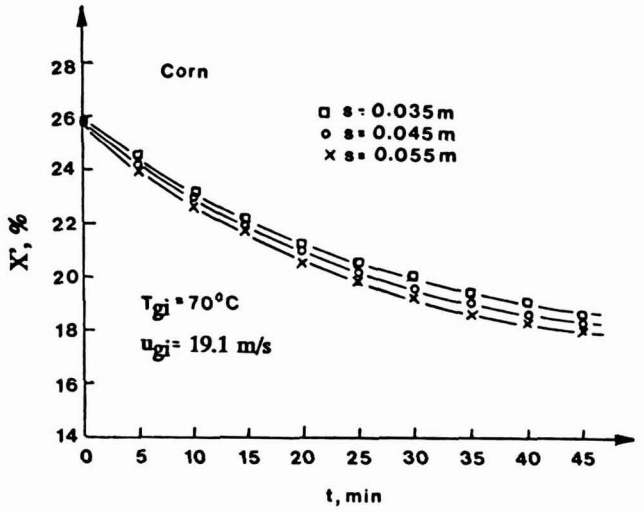



FIG. 5. EFFECT OF SLOT WIDTH ON DRYING RATE

mode with respect to heat transfer. Since the time spent between the draft plates is only a fraction of that in the downcomer, it is clear that the solids circulation rate, which depends to a great extent on the slot width, influences the heat-moisture diffusion cycel and hence the drying rate. A similar trend on the influence of the exposure time on drying rate in intermittent drying was noted by Chakraverty (1975).

In the draft channel of a TDSB the grains are fully exposed to an air stream of constant parameters (except in the initial drying period), i.e., temperature and humidity, which satisfy conditions for thin layer drying. Drying in the descending moving bed of grain in the downcomer cannot be treated as thin layer drying. However, our hypothesis on intermittent operation boils down to the assumption of drying only in the draft channel, although some effect of the time delay due to tempering period in the downcomer may be expected. Therefore, the Page's equation for thin layer drying of grains, in which the parameter "n" provides a tempering effect of time and accounts for possible errors resulting from the neglect of the internal resistance to moisture transfer, has been chosen to correlate the experimental data. Among the various models tested, e.g., equations based on Newton's law of cooling, simple or extended equations of diffusion (Kalwar 1991), the Page equation:

$$\frac{X-X_{eq}}{X_0-X_{eq}} = \exp(-kt^n) \tag{1}$$

was found to yield the best fit to experimental data (Fig. 6). This indirectly supports out hypothesis about the intermittent mode of drying with drying in a thin layer between the draft plates.

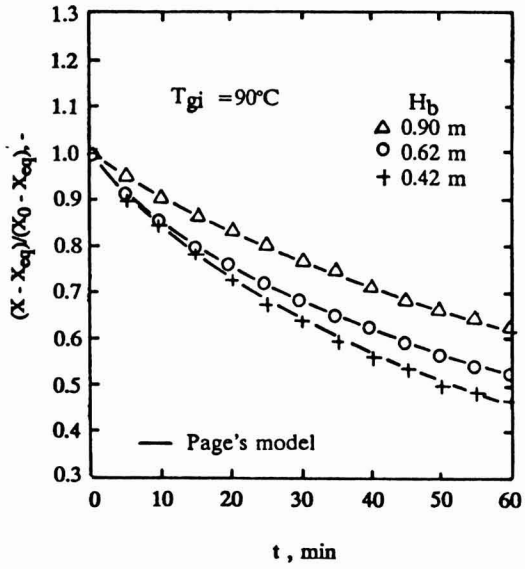



FIG. 6. MOISTURE RATIO VERSUS DRYING TIME FOR CORN (experimental and fitted data)

Drying parameters k and n in the Page equation were correlated statistically with the following variables: bed height, draft plate height, spout height, bed depth, separation height, initial grain temperature, inlet air temperature, equilibrium air humidity and circulation time. From a variety of possible equations, regression models were chosen for n and k based on their $R^2$ values, standard errors and residual plots. As an example, the following equations (Kalwar 1991) are recommended for corn drying over the range of parameters reported in this paper:

$$n = 186\,X_0\frac{w_i s}{w_d H_d} + 0.363\left(\frac{w_b}{H_b}\right) - 5.85\left(\frac{T_{p0}}{T_{gi}}\right) - 34.23\left(\frac{T_{p0}}{T_{gi}}\right)^2 - 62.12\left(\frac{T_{p0}}{T_{gi}}\right)^3 + 3.618t_c - 2.835t_c^3 + 0.056\varphi_{eq} - 0.00007\varphi_{eq}^3 + 0.717m_S^{0.5} - 2.103m_S^{0.333} \quad (2)$$

with $R^2 = 0.9993$ and SE = 0.02363.

$$k = 39.7\frac{w_i s}{w_d H_d} - 1.56\frac{w_b}{H_b} + 0.82\left(\frac{w_b}{H_b}\right)^{1.5} - 0.95\left(\frac{H_b}{H_d}\right)^2 + 0.56\left(\frac{H_b}{H_d}\right)^3 + 0.47\left(\frac{T_{p0}}{T_{gi}}\right)^3 - 0.78t_c^2 + 0.0076t_c^3 - 0.577X_0^{0.5} - 0.0076\varphi_{eq} + 0.000234\varphi_{eq}^2 - 0.00125m_S + 0.0312m_S^{0.5} \quad (3)$$

with $R^2 = 0.9996$ and SE = 0.000372.

where $\varphi_{eq}$ in Eq. (2) and (3) is the relative humidity in equilibrium with the initial moisture content of the grain at the inlet gas temperature (Hall and Rodriguez-Arias 1958).

## CONCLUSIONS

A two-dimensional spouted bed can be used for drying of grains. Page's equation with drying parameters related to the system geometry, operating parameters and grain characteristics was found to be appropriate to describe the drying kinetics. Empirical correlations are presented for the drying parameters in Page's equation; these can be used to predict the drying rates over parameter ranges studied.

## NOMENCLATURE

| | |
|---|---|
| $H_b$ | bed height, m |
| $H_d$ | spout height, m |
| $H_0$ | separation height, m |
| L | length of the dryer, bed or gas inlet, m |
| $m_S$ | mass of solid (bone dry), kg |
| s | slot width, m |
| t | time (elapsed), s |
| $t_c$ | cycle time, min |
| T | temperature, °C |
| $u_{gi}$ | gas velocity (in gas inlet), m/s |
| $w_b$ | bed width, m |
| $w_d$ | spout width, m |
| $w_i$ | width of gas inlet, m |
| X | moisture content (db), kg/kg |
| X' | moisture content (wb), % |
| $\varphi$ | gas humidity, % |
| SE | standard error |

### Subscripts

| | |
|---|---|
| g | gas |
| p | particle |
| S | solid |
| i | inlet |
| o | outlet |
| 0 | initial |
| eq | equilibrium |

## ACKNOWLEDGMENT

The authors are grateful to the Ministry of Eduction, Quebec for an FCAR grant and to NSERC for an operating grant.

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