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# MOISTURE SORPTION ISOTHERMS OF PECTINS

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## ABSTRACT

*The water adsorption isotherms of LM and HM pectins and pectin gels (made from HM pectin with the addition of sugar) were determined at 25C, using the standard gravimetric method developed by the European Cooperation Project COST 90. The water-binding properties of these materials were studied through examination of the sorption data. The experimental curves were fitted to one three-parameter equation (GAB equation), eight two-parameter equations reported in the literature, and a two-parameter equation developed in this work. The GAB equation gave the best fitting; however, the results showed that it should not be used for the estimation of the monolayer moisture content.*

## INTRODUCTION

The interaction of pectins and water is of considerable biological and industrial interest. Pectins are used in the food industry as thickeners, stabilizers and gelling agents, and they exert their biological role in an aqueous environment (intermediate moisture foods). However, experimental data on the sorption of water by pectins are scarce in the literature, and the water binding mechanism has not been yet clarified (Labuza and Busk 1979). Palmer *et al.* (1948) determined the moisture sorption isotherms of low methoxyl (LM) and high methoxyl (HM) pectins. The equilibrium moisture content was found to be essentially independent of the methoxyl content, a rather surprising result. Bettelheim *et al.* (1956) studied the structural changes in the polygalacturonide chains during water adsorption.

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Bandyopadhyay *et al.* (1980) compared the sorption properties of apple (HM) pectin with those of other food constituents. Padival *et al.* (1981) studied the stability of pectins during storage. Wallingford and Labuza (1983) determined the moisture sorption isotherms of LM and HM pectins. They found that the LM pectin adsorbs higher amounts of water than the HM pectin for the same water activity.

The objective of this study was: (1) to provide reliable experimental data for the sorption characteristics of apple and citrus pectins and pectin gels using a static standardized method, (2) to investigate the water binding mechanism for LM pectins, HM pectins and pectin gels and (3) to suggest simple mathematical models that describe their sorption behavior.

## MATERIALS AND METHODS

### Materials

The pectin samples used in this investigation were: (1) apple pectin with a degree of esterification (DE) 70–75% and high methoxyl content (12% MeO) and (2) citrus pectin of DE 10% and low methoxyl content (1.6% MeO). Both materials came from Fluke Co. (lot numbers 76282 and 76280, respectively) in the form of a dry powder with the general molecular type  $(C_6H_{10}O_6)_{130-150}$ .

The pectin gel was made from the apple (HM) pectin with the addition of sugar and citric acid at a pH of 3.0, which is the optimum value for gel formation. The system has a high content of solids (60%), i.e., 3% HM pectin, 57% sugar and 0.3% citric acid. The gels were prepared by a method similar to that of Moy *et al.* (1971).

### Sorption Apparatus

The standardized static method of measurement of water activity in foods, developed by the European Cooperation Project COST 90 (Spiess and Wolf 1983) was used for the experiments. The experimental apparatus, described in detail elsewhere (Tsami *et al.* 1990) consisted of a thermostatically controlled water bath, containing ten 1-L air-tight glass jars. Each glass jar contained a saturated salt solution corresponding to a specific water activity. The samples were placed on Petri dishes above the saturated salt solution.

The accuracy and reliability of the experimental apparatus were checked by measuring the sorption isotherm of the reference material (microcrystalline cellulose, MCC), as described in the above reference (Tsami *et al.* 1990).

### Experimental Procedure

Representative samples were taken from the bulk of the pectin powder (400–500 mg) and were placed in 25 × 25 mm weighing bottles to form a layer of thickness about 2 mm. The samples were then predried (for the measurement of the adsorption isotherm) in decanters containing P<sub>2</sub>O<sub>5</sub> for 15 days at room temperature.

The gel samples were prepared by cutting thin slices of 1 mm thickness (about 900 mg) from the mass of the gel. These samples were then placed in weighing bottles and predried in decanters for 24 days, until they reached a constant weight of about 500 mg. The mild conditions used for the initial dehydration of the samples (P<sub>2</sub>O<sub>5</sub>) and room temperature) were necessary, in order to prevent thermal degradation of the pectins (e.g., depolymerization) and changes of the physical structure of the gels (loss of elastic texture, shrinkage, etc.). The sample was weighed every three days and the dehydration was considered to be complete after 24 days.

To perform the sorption measurements, each sample was distributed in three weighing bottles for each glass jar (three replications for each experimental point). The required equilibration time was found to be 15 days for the pectins and 22 days for the gels, in accordance with the literature (Saravacos *et al.* 1986). The equilibrium moisture content was determined using the vacuum oven method at 70C and 50 Torr for 6 h (AOAC 1980).

### Mathematical Analysis

The moisture sorption isotherms of foods can be described by numerous mathematical models with two or more parameters (Van den Berg and Bruin 1981). However, models having more than three parameters are too complicated for straightforward interpretation or use (Schuchmann *et al.* 1990). The most successful three parameter model is probably the GAB equation (Van den Berg 1984; Maroulis *et al.* 1988), the parameters of which have a physical meaning:

$$X = \frac{X_m C k a_w}{(1 - k a_w) (1 - k a_w + C k a_w)} \quad (1)$$

where  $X$  is the moisture content of the material,  $a_w$  is the water activity,  $X_m$  is the monolayer moisture content, while  $C$  and  $k$  are parameters related to temperature effects.

A number of semi-empirical two parameter equations were also tested in this study, regarding their ability to describe the experimental data. These models are (Iglesias and Chirife 1982):

The Halsey model

$$a_w = \exp(-b_2/x^{b_1}) \quad (2)$$

The Henderson model

$$1-a_w = \exp(-b_2 x^{b_1}) \quad (3)$$

The Bradley model

$$\ln(1/a_w) = b_2 b_1^x \quad (4)$$

The Kuhn model

$$\ln a_w = b_1/(x-b_2) \quad (5)$$

The Oswin model

$$x = b_2 \left[ \frac{a_w}{1 - a_w} \right]^{b_1} \quad (6)$$

The Iglesias and Chirife model (Iglesias and Chirife 1978)

$$x = b_1 \left[ \frac{a_w}{1 - a_w} \right] + b_2 \quad (7)$$

The Iglesias and Chirife model (Iglesias and Chirife 1981)

$$\ln \left[ x + (x^2 + x_{0.5})^{1/2} \right] = b_1 a_w + b_2 \quad (8)$$

The BET model

$$\frac{x}{x_m} = \frac{C a_w}{1 + C a_w} + \frac{a_w}{1 - a_w} \quad (9)$$

In all the above equations,  $X$  is the moisture content (dry basis),  $b_1$ ,  $b_2$  are the parameters of the model and  $X_{0.5}$  (Eq. 8) is the moisture content at  $a_w = 0.5$ . In the case of the BET equation,  $X_m$  is the monolayer moisture content and  $C$  is a constant related to thermal effects.

In this study, we tried to modify Eq. (8), in order to improve the adequacy of the fit. This modification was based on the fact that when  $X_{0.5}$  was estimated

from the experimental data, its value was slightly negative. Placing  $X_{0.5} = 0$ , the modified equation takes the form:

$$\ln X = b_1 a_w + b_2 \quad (10)$$

The parameters of the equations were estimated by fitting the mathematical models to the experimental data. This procedure minimizes the sum of the squares of the residuals between the experimental and the calculated values (SST), using nonlinear regression analysis. Although some of Eq. (1)–(10) can be linearized by a transformation of the dependent variable (X), this procedure can give highly erroneous results and should be avoided (Maroulis *et al.* 1988).

The comparison of the goodness of fit for the various equations was made on the basis of the mean square error,  $s_E$ , calculated by:

$$s_E^2 = SST/(n - p) \quad (11)$$

where  $n$  is the number of experimental points and  $p$  is the number of parameters.

## RESULTS AND DISCUSSION

The experimental moisture sorption data of the three materials (LM pectin, HM pectin and pectin gel), measured at 25°C, are presented in Table 1, and the corresponding isotherms are given in Fig. 1 and 2. The isotherms of the two pectins gave the characteristic s-shaped curve of water adsorption isotherm (type II according to the BET classification), while that of the gel exhibited a different behavior (type III according to the BET classification, if the point 0,0 is not taken into account).

A result, which is immediately evident from the examination of Fig. 1, is that the HM pectin appears to sorb more water than the LM pectin up to a water activity value of 0.8, beyond which this trend is reversed and the LM pectin sorbs more water than the HM pectin. Such a behavior must be attributed to the complex structure of the pectin molecule, in which some of the carboxyl groups are esterified with methyl alcohol, some are neutralized with cations and some are free acids. The carboxyl groups show a great affinity for water, while the methoxyl groups are hydrophobic; therefore, from a first point of view, the LM pectin should be more hygroscopic than the HM pectin over the whole range of water activities. However, this is in contrast to our experimental results, as well as to older results of Palmer *et al.* (1948), who found that the equilibrium moisture content was practically independent of methyl ester content for the range investigated. This is an indication that the mechanism of water sorption by long chain polymers

TABLE 1.  
EXPERIMENTAL MOISTURE SORPTION DATA FOR THE THREE SAMPLES. THE  
MEAN VALUES OF THREE REPETITIONS FOR EACH EXPERIMENTAL POINT ARE  
REPORTED

| $a_w$ | Moisture content (kg H <sub>2</sub> O/kg d.s.) |           |            |
|-------|--|-----------|------------|
|       | LM pectin                                      | HM pectin | Pectin Gel |
| 0.112 | 0.016  | 0.037     | 0.023      |
| 0.226 | 0.022  | 0.062     | 0.023      |
| 0.327 | 0.047  | 0.082     | 0.023      |
| 0.438 | 0.056  | 0.100     | 0.029      |
| 0.529 | 0.074  | 0.127     | 0.053      |
| 0.577 | 0.084  | 0.146     | 0.088      |
| 0.708 | 0.119  | 0.175     | 0.187      |
| 0.753 | 0.131  | 0.208     | 0.260      |
| 0.843 | 0.290  | 0.254     | 0.472      |
| 0.903 | 0.422  | 0.328     | 0.793      |

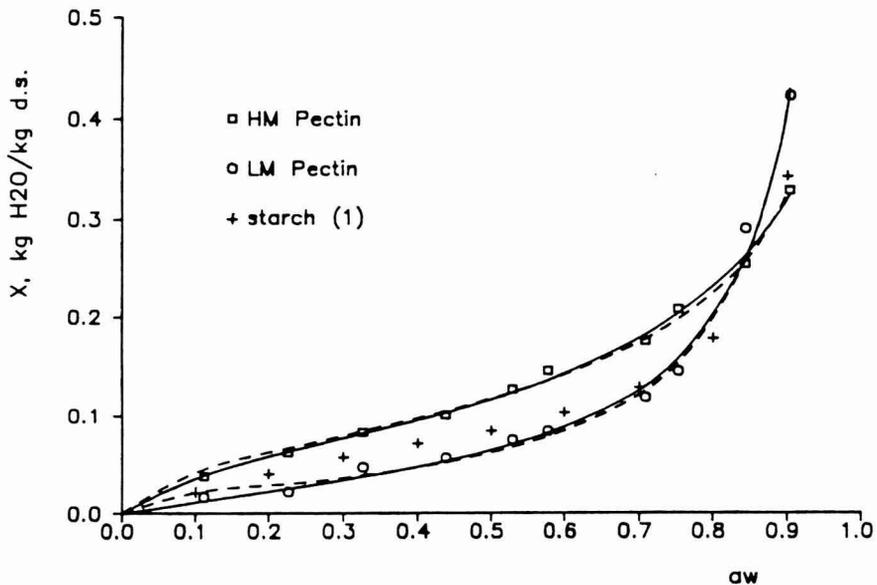


FIG. 1. MOISTURE SORPTION ISOTHERMS OF PECTINS AND STARCH AT 25C  
Continuous line: GAB equation, dashed line: Oswin equation.  
(1): Chilton and Collison (1974)

involves factors other than the hydration affinity of the characteristic groups. These are the availability of the polar groups in the molecule of the polysaccharides, the distribution and arrangement of these groups in the molecule, the secondary structure (interchain bonds, etc.) of the biopolymer, its conformation, the degree of crystallinity, the packing of the chains, etc. (Chen *et al.* 1984).

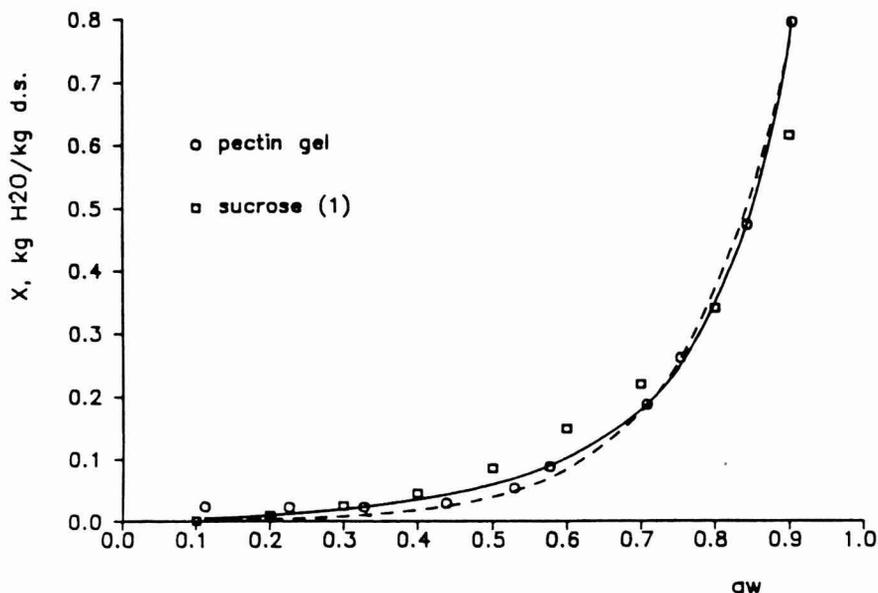


FIG. 2. MOISTURE SORPTION ISOTHERMS OF PECTIN GEL AND SUCROSE AT 25°C  
 Continuous line: GAB equation, dashed line: Eq. (10). (1): Makower and Dye (1956)

The molecule of the LM pectin contains a great number of free carboxyl groups (hydrophilic). However, the carboxyl groups can form interchain bonds with adjacent carboxyl groups, strong enough to resist the dissociating effect of water, even if the water penetrates between the chains. The existence of interchain hydrogen bonds between two adjacent carboxyl groups has been proved by X-ray investigation (Palmer *et al.* 1948). The X-ray photographs showed that the molecule of the LM pectin has a three-dimensional symmetrical structure, due to the interchain bonds and presents a high degree of crystallinity. As a result, the carboxyl groups are no longer available to water and the sorption ability of this pectin is limited. On the contrary, the higher the degree of methylation, the number of interchain bonds becomes smaller and the remaining carboxyl groups are available for the sorption of water. Besides, the presence of the methoxyl group increases the intermolecular distance and leads to a more loose packing of the biopolymer, which allows the penetration of the water molecules. As a result, the HM pectin sorbs more water for water activity values less than 0.8.

However, as the water activity increases, the sorbed moisture causes a subsequent swelling of the biopolymer, the degree of crystallinity decreases, and there is an increasing availability of the polar groups to the water molecules. Finally, the swelled polysaccharide goes into a solution. In this case, all the polar groups of the LM pectin are available for the sorption of water, and the LM pectin sorbs more quantities of water than the HM pectin. This behavior is observed for water

activity values higher than 0.8. Wallingford and Labuza (1983) reported that the LM pectin is more hygroscopic than the HM pectin for high values of the water activity.

In comparison to other polysaccharides, the pectins seem to be more hygroscopic than starch, sodium alginate, casein and cellulose (Leung and Steinberg 1979; Bandyopadhyay *et al.* 1980). The results of Bandyopadhyay *et al.* (1980) for apple (HM) pectin are very similar to those found here.

The moisture sorption isotherm of the pectin gel (made from the HM pectin) exhibits a different behavior, which is characteristic of sugars (due to the high sugar content of this gel). For low water activities ( $a_w < 0.6$ ) the sorbed moisture is very low, but for higher water activities ( $a_w > 0.6$ ) there is a steep increase of the sorbed moisture, due to the dissolution of sugars. The sorption isotherm of this gel is similar to the sorption isotherms of dried fruits (Weisser 1985; Tsami *et al.* 1990), which also have a high sugar content. The texture of the gel changes with the moisture content and for  $a_w < 0.6$ , when the sorbed moisture is low, the appearance is poor and the material is very hard, due to the dehydrating effect of the added sugars. For  $0.6 < a_w < 0.85$ , the gel mixture has all the desired characteristics in appearance and texture, being clear and soft, yet steady. In this region, the sugars still hold the largest portion of water, but the pectin molecules have sorbed enough water molecules, in order to lose their initial rigidity. For  $a_w > 0.85$ , there is a dissolution of sugars in water, and the appearance of the material resembles that of a concentrated solution.

The fitting of the mathematical equations on the experimental moisture sorption data gave the results of Table 2. Most of the equations gave an acceptable fit; the GAB equation (a three-parameter equation) gave the better fit, however, using the criterion of the mean square error ( $s_E$ ), the Oswin, Halsey and Henderson equations, as well as Eq. (10), gave similar results. The fit to these equations

TABLE 2.  
STANDARD DEVIATIONS ( $s_E$ , kg H<sub>2</sub>O/kg d.s.) BETWEEN THE EXPERIMENTAL  
AND PREDICTED VALUES FOR THE VARIOUS MATHEMATICAL MODELS

| Model                    | LM pectin | HM pectin | Pectin Gel |
|--------------------------|-----------|-----------|------------|
| GAB                      | 0.017     | 0.006     | 0.012      |
| Oswin                    | 0.016     | 0.006     | 0.020      |
| Halsey                   | 0.016     | 0.013     | 0.027      |
| Henderson                | 0.022     | 0.009     | 0.012      |
| Bradley                  | 0.027     | 0.046     | 0.033      |
| Kuhn                     | 0.016     | 0.028     | 0.022      |
| Iglesias I <sup>1</sup>  | 0.016     | 0.029     | 0.020      |
| Iglesias II <sup>2</sup> | 0.019     | 0.026     | 0.027      |
| BET                      | 0.016     | 0.045     | 0.018      |
| Equation (10)            | 0.018     | 0.009     | 0.017      |

<sup>1</sup> equation (7)

<sup>2</sup> equation (8)

Table 3.  
MONOLAYER MOISTURE CONTENTS OF THE THREE SAMPLES ( $X_m$ , kg H<sub>2</sub>O/kg d.s.) ESTIMATED BY THE GAB AND BET EQUATIONS

| Material   | GAB   | BET   | BET (points 1-9) | BET (points 1-8) |
|------------|-------|-------|------------------|------------------|
| LM pectin  | 0.044 | 0.043 | 0.039            | 0.033            |
| HM pectin  | 0.080 | 0.039 | 0.038            | 0.037            |
| Pectin gel | 0.768 | 0.093 | 0.056            | 0.028            |

was better for the HM pectin than for the LM pectin and the pectin gel. The results of the GAB and Oswin equations for the three samples are also given in Fig. 1 and 2 as the continuous lines. The Iglesias II equation gave poor fitting in the case of pectins, although it has proved successful for other materials (Iglesias and Chirife 1981). When we considered  $X_{0.5}$  as an unknown parameter of the model and estimated its value by fitting the equation to the experimental data, it was found slightly negative in all cases. This observation led us to the formulation of Eq. (10), which was more successful.

The GAB equation could also be used for the estimation of the monolayer moisture content,  $X_m$ , a parameter that is very significant for commercial purposes (Iglesias and Chirife 1982). However, the estimated values of  $X_m$  were unacceptably high, as shown in Table 3. The explanation is that the parameters of the GAB equation,  $X_m$ ,  $C$  and  $k$  are highly correlated (Maroulis *et al.* 1988), and their absolute values can vary greatly, although their combination gives acceptable results. The conclusion is that the GAB equation should not be used for the estimation of the monolayer moisture content. On the contrary, the BET equation, which gave poor fitting, can give reasonable values of the monolayer moisture content (Table 3), especially if we remove the experimental data for the highest water activity values. In this case, we see that the estimated monolayer moisture contents is of the order  $X_m$  (HM pectin) >  $X_m$  (LM pectin) >  $X_m$  (pectin gel), which agrees with the sorption behavior of the three samples.

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# CHANGES IN THE MOISTURE AND CYANIDE CONTENTS OF BITTER CASSAVA DURING ARTIFICIAL AND SOLAR DRYING

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## ABSTRACT

*Bitter cassava (Manihot esculenta Crantz) dice (2 × 1 × 1 cm) were sun-dried as well as dehydrated at 30, 45 and 60C with an air velocity of 300 ± 10 cm/min. The effect of drying on moisture removal and on the elimination of total cyanide was studied. Dehydration reduced the cyanide content at all temperatures. Rates of cyanide and moisture removal were fastest at 60C and decreased with dehydration temperature. Air circulation during dehydration facilitated cyanide reduction. Final moisture content depended on the drying temperature.*

## INTRODUCTION

Cassava is a major food source for a large part of world population, particularly in the countries of South America, Africa and Asia where it is primarily a major source of energy for about 300–500 million people (Okezie and Kosikowski 1982). Brazil is the world's leading producer of cassava, which has a significant preference in the human nutrition in the northeastern region, especially among the rural and the low income urban population (Mota 1978).

Cassava contains two cyanogenic glucosides: linamarin and lotaustrin, which on hydrolysis, yield hydrogen cyanide (Conn 1969), a strong protoplasmatic poison

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for all forms of life (Bruijn 1973). Traditional processing of cassava is unlikely to remove all the cyanide, which may be responsible for chronic toxicity associated with continued ingestion of cassava products (Coursey 1973). Osuntokun (1981) attributed cassava as the main dietary cause for chronic cyanide intoxication and neuropathy in Nigerian Africans. Drying of cassava has been reported to reduce the cyanide level in the cassava product. Gomez *et al.* (1984) studied the effect of sun drying and oven drying with forced air on total cyanide content of the chips of 10 varieties of cassava and observed that sun drying (48 h) and oven drying (60C, 24 h) reduced the initial cyanide content to approximately 15–30%. Loss of cyanide content in cassava from 77% (Gomez and Valdivieso 1984) to 85% (Paula and Rangel 1940) during oven drying has been reported, while loss of 56% (Paula and Rangel 1940) to 94% (Gomez *et al.* 1984) was observed during sun drying. Smaller cyanide losses varying from 26 to 33% at 60C and 18 to 21% at 80C were observed by Joachim and Pandittesekere (1944). Sun drying was reported to be more effective than artificial drying in the removal of total cyanide (Gomez and Valdivieso 1984; Mahungu *et al.* 1987). Cooke and Maduagwu (1978) observed that in earlier reports important process parameters such as piece size, drying rate and temperature were often not specified. Nambisan and Sundaresan (1985) reported that cyanide retention during dehydration is governed by chip thickness.

A mathematical model for the moisture transfer in cassava during dehydration was reported by Igbecka (1982). Correlation coefficients between the predicted and experimental values ranged between 0.97 to 0.99. However, no study on mathematical modelling for the total cyanide content during drying of cassava dice has been reported.

The present work was proposed with the objectives to: (1) quantify the losses in cyanide and moisture contents in bitter cassava (*Manihot esculenta* Crantz) during artificial and solar drying, and (2) process the experimental data and propose appropriate equations for the calculation of moisture and cyanide contents at different drying conditions.

## MATERIALS AND METHODS

### Bitter Cassava

Bitter cassava (*Manihot esculenta* Crantz) tubers of the cultivar ‘‘Osso Duro’’ of 12 months of age were obtained from the experimental station of EMATER — ‘‘Empresa de Assistencia Tecnica e Extensao Rural,’’ in the state of Paraiba, Brazil.

### Sample Preparation

The tubers were washed with water to remove the sand and were peeled. The tubers were cut radially into discs of about 2 cm thickness then cut into dice  $2 \times 1 \times 1 \text{ cm}^3$ .

### Drying

For solar drying, aluminum trays containing a single layer of diced cassava were put directly in the sun. At the end of each daily 8 h exposure to the sun, dice were collected and sealed in plastic bags and again exposed to the sun next day until the drying was complete. The ambient temperature was  $29 \pm 2\text{C}$  during drying.

Artificial drying of the cassava dice was carried out at 30, 45 and 60C for different time periods in a forced air dryer. The dice were spread on trays in layers, not exceeding 5 cm in thickness. The velocity of air was maintained at  $300 \pm 10 \text{ cm/min}$ .

During drying, cassava dices were removed periodically for the determination of moisture and total cyanide content.

### Analytical Methods

The moisture content of the samples was determined using AOAC (1984) method and the total cyanide content was determined by the method of Wood (1965). The HCN was liberated by the action of endogenous linamarase enzyme on the cyanogenic glucosides for 2 h at 37C, followed by addition of 2N sulphuric acid. The HCN was collected by distillation in a solution of sodium carbonate and the concentration of HCN was determined colorimetrically after the development of color with alkaline picrate. Moisture determinations were carried out in duplicate. Total cyanide content was determined on five replicates.

### Mathematical Modelling

From the experimental data for loss in moisture during drying, a numerical differentiation for each discrete point was done and an equation was obtained to represent the derivative  $dM/dt$  (change in moisture content with time). This function was equalized to zero to determine the equilibrium moisture content for each experiment (Fioreze 1986).

From the equilibrium moisture content, the fractional moisture ratio (MR) at various time periods was calculated by using the equation:

$$MR = \frac{M(t) - M_e}{M_o - M_e} \quad (1)$$

where  $M(t)$  is the moisture content (%) of the sample at time  $t$ ;

$M_e$  is the equilibrium moisture content (%) of the sample at the end of drying;

$M_o$  is the initial moisture content (%) of the sample.

Linear regression, least square method was used to calculate the parameters  $a$  and  $b$  of the equation:

$$MR = \exp(-at^b) \quad (2)$$

It was assumed that it will take a very long time of dehydration for total cyanide content to reach zero. The cyanide ratio (CR) was calculated by the following equation:

$$CR = \frac{C(t)}{C_o} \quad (3)$$

where  $C(t)$  is the concentration of total cyanide in the sample at time  $t$ ;  
and  $C_o$  is the initial total cyanide content of the sample.

Again, linear regression was applied to calculate the parameters of the equation:

$$CR = \exp(-ct^d) \quad (4)$$

The relative error (RE) between each experimental value and the calculated value was obtained from the equation:

$$RE = \text{ABS}(P_e - P_c)/P_e$$

where  $P_e$  represents the experimental value and  
 $P_c$  represents the calculated value.

The mean error between the points for each test furnishes the relative mean error (RME) given by:

$$RME = (RE)/n \quad (5)$$

where  $n$  is the number of points.

## RESULTS AND DISCUSSION

The results of the effect of temperature on removal of moisture and total cyanide content of diced dehydrated cassava under forced air circulation are shown in Tables 1-3, while Table 4 presents the data obtained on solar drying. The relative humidity of air in the dryer was same as that of the incoming air, since the product load was small. Tables 1-4 show the data obtained experimentally as well as the data calculated by applying the regression analysis. The necessity of applying regression analysis was felt due to the difficulty in obtaining representative samples for the determination of total cyanide in the cassava dice during dehydration. Because longitudinal and radial cyanide gradients exist in cassava roots (Bruijn 1973), the dice were not uniform with respect to total cyanide content. Although 5 replicates were carried out for each sample, a wide variation in cyanide contents was observed within these replicates.

TABLE 1.  
EFFECT OF DEHYDRATION (60 C; 16% RH) ON THE MOISTURE AND TOTAL  
CYANIDE CONTENT (HCN) IN 100 g OF BITTER CASSAVA

| Dehydration<br>time<br>(H) | Moisture (g) |       | HCN (mg)     |       |
|----------------------------|--------------|-------|--------------|-------|
|                            | Experimental | Calc. | Experimental | Calc. |
| 0.00                       | 61.80±5.05   | 61.80 | 3.20±0.72    | 3.20  |
| 1.50                       | 35.85±3.13   | 35.98 | 1.67±0.51    | 1.51  |
| 3.00                       | 20.50±1.72   | 19.52 | 0.73±0.11    | 0.88  |
| 4.50                       | 10.37±0.78   | 11.35 | 0.41±0.17    | 0.54  |
| 6.00                       | 7.52±0.53    | 7.64  | 0.26±0.13    | 0.35  |
| 7.50                       | 6.67±0.44    | 6.06  | 0.35±0.13    | 0.23  |
| 8.25                       | 5.47±0.41    | 5.66  | 0.23±0.02    | 0.18  |
| Correlation<br>coefficient | 0.9995       |       | 0.9939       |       |
| R <sup>2</sup>             | 0.9991       |       | 0.9878       |       |

TABLE 2.  
EFFECT OF DEHYDRATION (45 C; 31% RH) ON THE MOISTURE AND TOTAL  
CYANIDE CONTENT (HCN) IN 100 g OF BITTER CASSAVA

| Dehydration<br>time<br>(H) | Moisture (g) |       | HCN (mg)     |       |
|----------------------------|--------------|-------|--------------|-------|
|                            | Experimental | Calc. | Experimental | Calc. |
| 0.00                       | 57.00±4.35   | 57.00 | 4.56±0.89    | 4.56  |
| 2.00                       | 32.90±2.73   | 31.39 | 2.66±0.63    | 2.40  |
| 4.00                       | 20.97±1.57   | 21.45 | 1.28±0.27    | 1.58  |
| 6.00                       | 15.30±1.12   | 15.99 | 1.06±0.15    | 1.09  |
| 8.00                       | 11.50±0.81   | 12.72 | 0.68±0.08    | 0.78  |
| 23.50                      | 7.07±0.51    | 6.89  | 0.14±0.02    | 0.10  |
| Correlation<br>coefficient | 0.9988       |       | 0.9938       |       |
| R <sup>2</sup>             | 0.9977       |       | 0.9877       |       |

### Verification of the Model

A computer program was written to calculate the values for the constants a, b, c and d and RME values using the Eq. (2), (4) and (5) as described in the methodology. The values obtained for the equilibrium moisture contents and the constants of Eq. (2) and (4) for different experiments are presented in Table 5. Figures 1 and 2 show the difference in the experimental and calculated values of moisture and total cyanide ratio, respectively. Both, moisture and total cyanide removal from the cassava dice followed an exponential behavior. The good fit between the model and the data for moisture and cyanide ratio and the high correlation coefficients between the experimental and calculated values for the moisture and cyanide contents (Tables 1-3 and Table 4) support the adequacy of the equations. Within the experimental set of drying conditions, the RME values for the moisture contents were extremely low (less than 3.8%) while for the total cyanide content, the values varied from 10.5 to 14.2% for all drying temperatures except at 60C. A relatively higher RME value of 21.5% was found at 60C. These relatively higher values of RME for total cyanide content could be attributed to the factors such as the large variation in concentration within the same specie of cassava and to the precision of the experimental method of analysis for the total cyanide content at extremely low concentrations, expressed in mg/100 g.

TABLE 3.  
EFFECT OF DEHYDRATION (30 C; 74% RH) ON THE MOISTURE AND TOTAL  
CYANIDE CONTENT (HCN) IN 100 g OF BITTER CASSAVA

| Dehydration<br>time<br>(H) | Moisture (g) |       | HCN (mg)     |       |
|----------------------------|--------------|-------|--------------|-------|
|                            | Experimental | Calc. | Experimental | Calc. |
| 0.00                       | 59.35±4.71   | 59.35 | 5.60±1.38    | 5.60  |
| 3.00                       | 40.95±3.63   | 40.53 | 2.99±0.73    | 3.45  |
| 7.00                       | 24.85±1.91   | 25.03 | 2.69±0.47    | 2.24  |
| 11.00                      | 17.20±1.22   | 17.66 | 1.95±0.23    | 1.54  |
| 15.00                      | 14.50±1.14   | 14.38 | 1.24±0.17    | 1.10  |
| 26.00                      | 12.10±1.03   | 12.20 | 0.43±0.05    | 0.48  |
| 29.00                      | 12.10±0.98   | 12.10 | 0.51±0.13    | 0.39  |
| Correlation<br>coefficient | 0.9999       |       | 0.9865       |       |
| R <sup>2</sup>             | 0.9998       |       | 0.9732       |       |

### Losses in Moisture and Cyanide Contents

The final moisture contents of the dice were 5.5, 7.0 and 12.1% in the artificial drying at 60, 45 and 30C, respectively. Within the range (30 to 60C) of dehydration temperatures studied, the rate of removal of total cyanide was maximum during dehydration at 60C. To reduce the total cyanide content to a level of 10% of the original, nearly 6, 16, and 25 h of dehydration was required at temperatures of 60, 45, and 30C, respectively, for drying under a constant air flow rate of  $300 \pm 10$  cm/min (Fig. 2). Bourdoux *et al.* (1980) studied the effect of oven drying of cassava chips at 60, 105 and 165C and found that maximum cyanide elimination occurred at 60C. Among the three temperatures of oven drying, i.e., 45, 60 and 75C of cassava leaves, Padmaja (1989) reported maximum elimination of total and free cyanide at 60C.

Solar drying ( $29 \pm 2$ C) of cassava dice could not reduce the total cyanide down to the 10% level, probably due to the fact that there was no forced air circulation (Table 4). The importance of air circulation during dehydration of cassava for the removal of total cyanide can be judged by the fact that in order to reduce the total cyanide content to a level of 20%, only 15 h of dehydration with air circulation was required at 30C, while in solar drying, which employed almost

TABLE 4.  
EFFECT OF SUN-DRYING ( $29 \pm 2$  C) ON THE MOISTURE AND TOTAL  
CYANIDE CONTENT (HCN) IN 100 g OF BITTER CASSAVA

| Drying<br>time<br>(H)      | Moisture (g)     |       | HCN (mg)        |       |
|----------------------------|------------------|-------|-----------------|-------|
|                            | Experimental     | Calc. | Experimental    | Calc. |
| 0.00                       | 64.34 $\pm$ 5.87 | 64.34 | 4.61 $\pm$ 1.38 | 4.61  |
| 3.00                       | 52.50 $\pm$ 4.73 | 52.45 | 3.91 $\pm$ 0.83 | 3.78  |
| 6.00                       | 41.75 $\pm$ 3.54 | 40.75 | 3.02 $\pm$ 0.69 | 3.14  |
| 9.00                       | 32.26 $\pm$ 2.86 | 31.62 | 2.60 $\pm$ 0.53 | 2.63  |
| 12.00                      | 24.32 $\pm$ 1.92 | 25.00 | 2.04 $\pm$ 0.37 | 2.21  |
| 15.00                      | 19.75 $\pm$ 1.27 | 20.42 | 1.74 $\pm$ 0.22 | 1.86  |
| 18.00                      | 16.10 $\pm$ 1.17 | 17.34 | 1.13 $\pm$ 0.19 | 1.57  |
| 21.00                      | 14.70 $\pm$ 1.02 | 15.34 | 1.30 $\pm$ 0.20 | 1.32  |
| 24.00                      | 13.92 $\pm$ 1.11 | 14.05 | 1.27 $\pm$ 0.21 | 1.12  |
| 27.00                      | 13.55 $\pm$ 1.02 | 13.25 | 1.11 $\pm$ 0.27 | 0.95  |
| 30.00                      | 13.25 $\pm$ 1.11 | 12.75 | 1.08 $\pm$ 0.22 | 0.80  |
| Correlation<br>coefficient | 0.9993           |       | 0.9871          |       |
| R <sup>2</sup>             | 0.9987           |       | 0.9744          |       |

the same temperature ( $29 \pm 2$  C) but no forced air circulation, nearly 22 h were needed.

Although the present study is based on a limited volume of experimental data obtained under specific and well-defined drying conditions, the results of this study on the removal of moisture and cyanide contents are comparable with the results reported by Paula and Rangel (1940), Charavanapavan (1944), and Nambisan and Sundaresan (1985) for oven drying and with the results of Correia (1974) and Bruijn (1973) for sun drying of cassava chips. It can be concluded that, depending upon the initial cyanide concentration, temperature and time of dehydration the toxicity of cassava chips can be brought to a safe level.

TABLE 5.  
VALUES OF EQUILIBRIUM MOISTURE CONTENTS AND CONSTANTS  
FOR DIFFERENT DRYING CONDITIONS

| Temperature<br>(C) | $M_e$<br>(%) | a      | b      | c      | d      | Relative Mean Error |
|--------------------|--------------|--------|--------|--------|--------|---------------------|
| 60                 | 5.0          | 0.3774 | 1.1694 | 0.5473 | 0.7821 | 0.0352 0.2149       |
| 45                 | 6.5          | 0.4110 | 0.7831 | 0.3870 | 0.7287 | 0.0259 0.1378       |
| 30                 | 12.0         | 0.1507 | 1.1035 | 0.2116 | 0.7535 | 0.0043 0.1422       |
| 29(Solar)          | 12.2         | 0.6766 | 1.2172 | 0.0706 | 0.9431 | 0.0379 0.1057       |

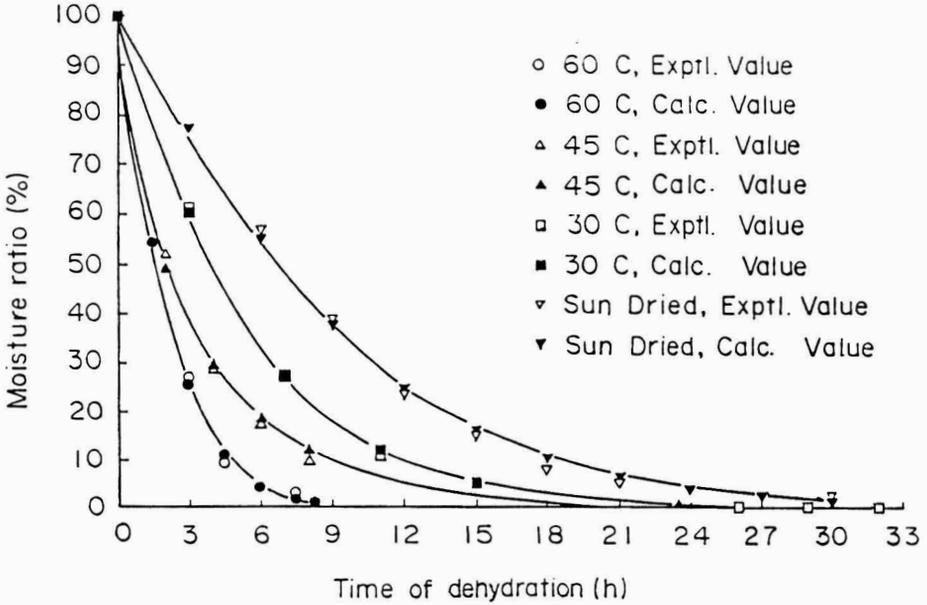


FIG. 1. EFFECT OF DEHYDRATION TEMPERATURE ON MOISTURE RATIO OF CASSAVA DICE

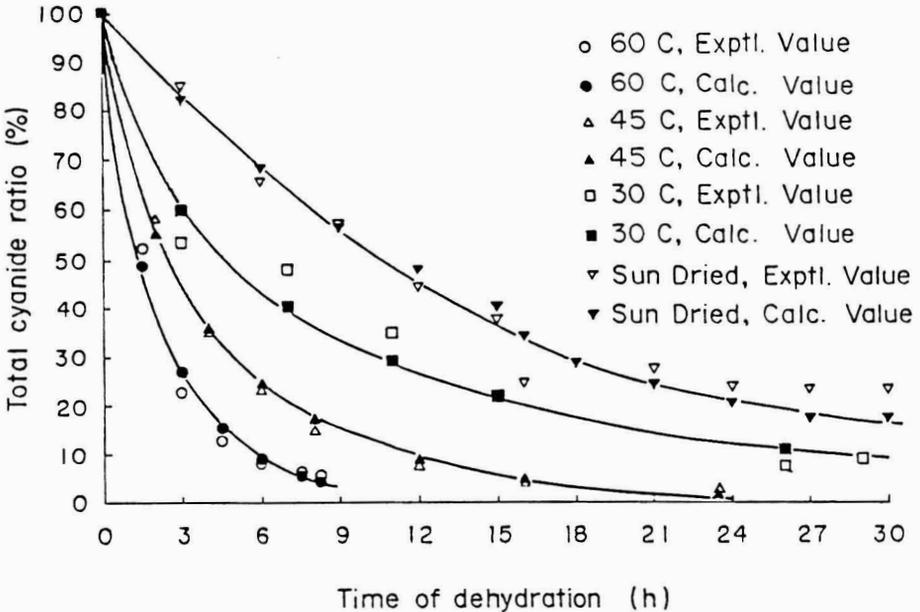


FIG. 2. EFFECT OF DEHYDRATION TEMPERATURE ON TOTAL CYANIDE RATIO OF CASSAVA DICE

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# DEVELOPMENT AND SENSORY CHARACTERISTICS OF EXTRUDED READY-TO-EAT PREBAKED POTATOES

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## ABSTRACT

*Small and off-grade Atlantic variety potatoes, which are normally not processed into products, were extruded and formed into precooked, ready-to-serve simulated baked potatoes. The potatoes were abrasion peeled, diced, steam blanched for 3 min, blended with 7% nonfat dry milk and extruded. The extruded product was filled into potato shaped plastic molds and stored at -20C for 24 h. Subsequently the frozen product was removed for the molds and dipped into a batter of wheat flour, water, glycine and dextrose to form skin and again stored in frozen conditions as above. Deep frying of the frozen products in corn oil at 190C for 3 min stabilized the skin coat. The microwave-baked simulated potatoes were evaluated for texture, flavor and color, and showed a high degree of acceptability.*

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## INTRODUCTION

Extrusion technology has been applied to a number of food products. The extrusion of cereals, which like potatoes are high in starch, represents an important segment of food processing. The extruder performs several important functions, including cooking and gelatinization of the starch and providing desired shape and texture to the processed product. In the case of potatoes, good quality extruded french fries have been prepared from dehydrated potato granules mixed with binders such as guar gum, high amylose corn starch and methylcellulose (Jadhav *et al.* 1976). The fabricated french fries based on 90% dehydrated potatoes compete with fries made from raw potatoes, since their compositional quality and frying time can be controlled (Nonaka *et al.* 1978). Other extrusion processed potato snacks, primarily based on dehydrated potatoes, are made by rehydrating the potato flakes, followed by extrusion, shaping and deep frying (Maga and Cohen 1978).

In Michigan, potato production is important to the state's agricultural economy. Every year, small, extra large or off-grade potatoes, which have little acceptance in the existing processed potato products line, find their way into the fresh market, which lowers the consumer's perception of Michigan's potato quality. The objective of this study was to determine whether these small and off-grade potatoes unsuitable for existing processed products or fresh market can be extruded and formed into ready-to-eat prebaked potatoes.

## MATERIAL AND METHODS

### Preparation of Potatoes for Extrusion

Approximately 10 kg small and off-grade potatoes of Atlantic variety were abrasion peeled and diced to approximately 2 cm × 1 cm length and diameter, respectively. The potatoes were steam blanched for 3 min and subsequently cooled with cold water for approximately 30 s. The blanched potatoes were blended with 7% nonfat dry milk in a stainless steel kettle.

### Extrusion

The extrusion of the potato-nonfat dry milk blend was performed on a pilot scale Baker-Perkins MPF 50/25 extruder equipped with a twin-screw feeder (K-tron Corp, Glassboro, NJ). The extrusion conditions established in preliminary experiments were: screw speed 200 rpm; temperature in the nine barrel zones toward the die plate, 22, 40, 60, 59, 61, 71, 63, 60 and 60C. The product

temperature in the extruder ranged from 25–60C. The die configuration was 18 mm × 3mm and product feed rate was 0.70 kg/min.

### **Potato Reformation**

The extrudate was filled into potato-shaped plastic molds with length and diameter of 10 cm × 7 cm, respectively, and stored at –20C for at least 24 h. Subsequently, the frozen potatoes were removed from the molds and dipped (while still in the frozen state) into a batter containing 200 g wheat flour, 500 mg glycine, 500 mg dextrose and 300 mL cold water for 1–2 min to form a skin and then again stored at –20C for 24 h. The potatoes were then deep fried in corn oil at 190C for 3 min to develop the skin texture and color.

### **Sensory Analyses**

Sensory evaluations of the color (skin and interior), texture and flavor of the simulated baked potatoes (microwaved for 3 min and then held at room temperature for 5 min before presenting to the panelists) were done to determine consumer acceptability. A nonnumerical hedonic scale, which ranged from excellent (9) to extremely poor (1), was used (Larmond 1977). A panel of 64 judges, made up of students and staff members from Michigan State University, participated in this study. Each judge was presented with one sample and was asked to examine the color, flavor and the texture of the reformed potatoes.

## **RESULTS AND DISCUSSION**

### **Development of Prebaked Potatoes**

Figure 1 shows the schematic flow diagram for the development of the simulated prebaked potatoes. Prior to extrusion, the peeled and diced potatoes were treated with steam for 3 min. This partially cooked the potatoes and inactivated polyphenol oxidases responsible for enzymatic browning. It also caused some gelatinization of the potato starch and the absorption of the free water in the potato tissue by the starch, so that “water feed back” due to presence of free available water in the feed was controlled during extrusion. Steaming the potatoes for less than 3 min was found to be insufficient and steaming longer than 3 min produced a product with mushy texture.

During high pressure extrusion, a significant decrease in viscosity of the potato starch due to depolymerization of the starch molecule into smaller fractions has been reported (Kim and Hamdy 1987). Therefore the use of 7% by weight non-

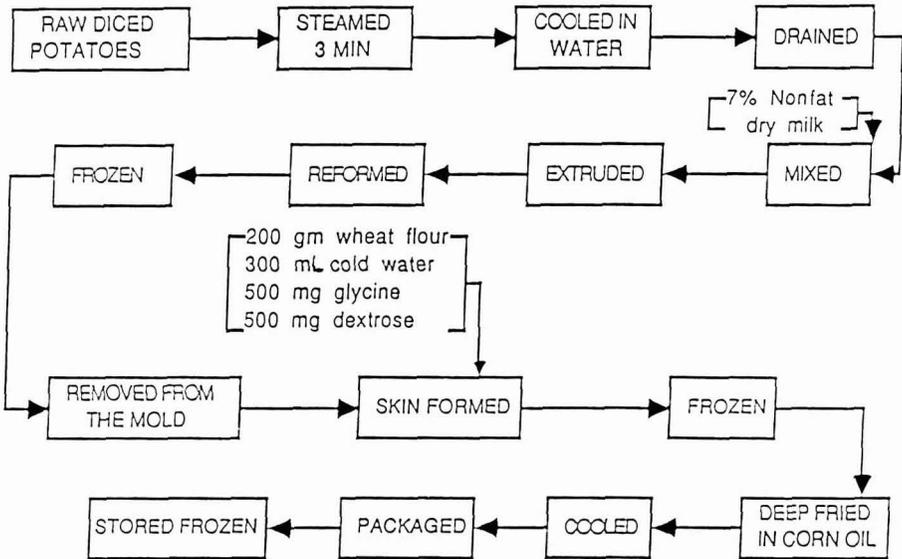


FIG. 1. SCHEMATIC OUTLINE OF THE PROCESSING OF SIMULATED BAKED POTATO PRODUCT

fat dry milk powder before the extrusion was necessary to bind the potato particles together. Besides its binding capacity, the use of powdered milk also influenced color and flavor of the final product.

The operating conditions for the extruder were determined in preliminary runs. It was observed that a product temperature of higher than 60C in the extruder produced an unacceptable gummy textured product. Excessive rupture of potato starch cells with concomitant escape of gelled starch has been suggested to cause a gummy texture in the cooked potatoes (Talbert *et al.* 1987). Temperatures lower than 60C produced a product with unacceptable flavor. Plastic molds were used for reforming the extruded potatoes, then simulated skin was developed. Deep-frying stabilized the skin coat and reproduced a desirable color.

### Sensory Analyses

Color, texture and flavor are three major components of food acceptability. Figures 2-5 show the evaluation of color, texture, flavor and overall acceptability of the simulated baked potatoes by the panelists.

Color is a readily discernible attribute for estimating the quality of potato products. The optimum color for steamed, boiled, baked, or mashed potatoes is creamy white. For fried potato products, a uniform golden crust is considered optimum.

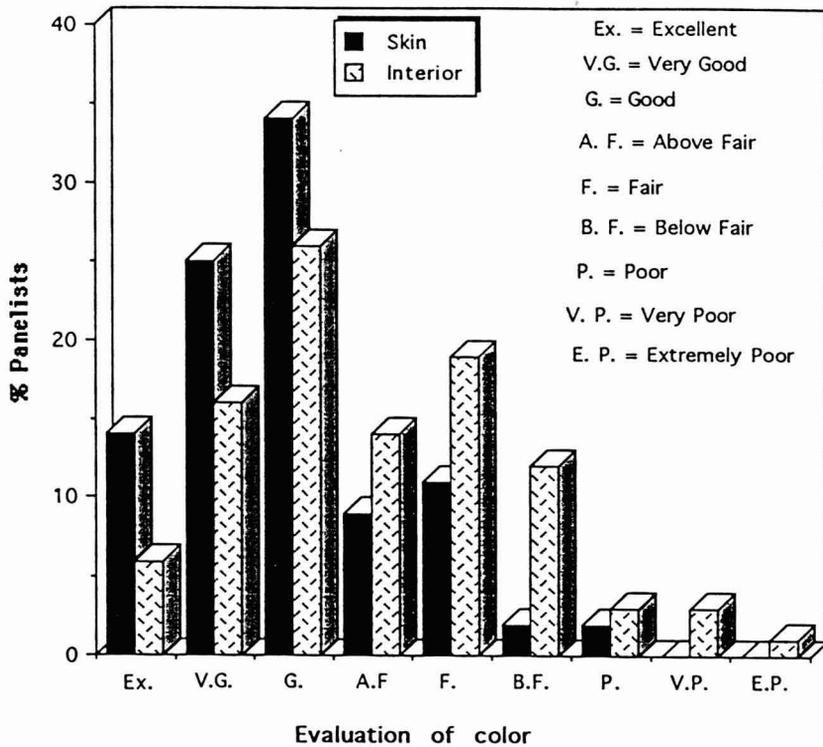


FIG. 2. SENSORY EVALUATION OF COLOR OF SIMULATED BAKED POTATO PRODUCT

Off-color is usually associated with after-cooking darkening of potatoes due to reaction of chlorogenic acid with metal ions, especially iron (Bate-Smith *et al.* 1958), or with nonenzymatic browning due to reaction between reducing sugars and amino acids during frying or dehydration (Shallenberger *et al.* 1959). In this study, the majority of panelists rated the skin and interior color of the microwave baked simulated potatoes (Fig. 2) as excellent to fair (92 and 83%, respectively) and only a few of them (8 and 17%) evaluated the color of skin and interior below fair.

The skin color formed by mixing into the batter of wheat flour, glycine, dextrose and water and deep frying received acceptance by most of the judges. The slightly lower rating for the interior color of the simulated product could be the result of nonenzymatic browning occurring during frying or microwave baking.

The texture of the cooked potatoes is considered to be one of the most important quality factors for consumer acceptance (Davis *et al.* 1983), and it is generally

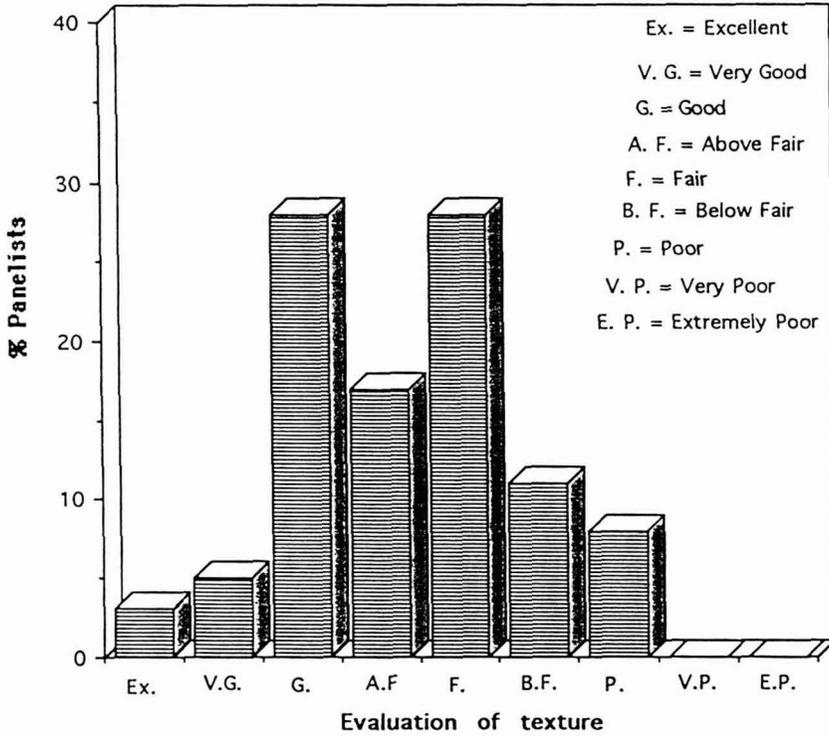


FIG. 3. SENSORY EVALUATION OF TEXTURE OF SIMULATED BAKED POTATO PRODUCT

agreed that good quality boiled, mashed, and baked potatoes should have a mealy texture. Leung *et al.* (1983) analyzed cooked potato samples of different cultivars and specific-gravity for textural and rheological properties by sensory evaluation, texture profile analysis (TPA) and stress relaxation tests. These authors found good correlation between hardness of cooked potatoes evaluated by sensory and TPA. Mealiness of cooked potatoes was found to correlate with the product of cohesiveness and adhesiveness by TPA. In this study most of the panelists rated texture of the simulated baked potatoes between very good and fair. Approximately 11 and 7% of the panelists' evaluated the texture as below fair and poor, respectively (Fig. 3).

Figure 4 represents the judges' evaluations of the flavor of the simulated potato product. Flavor denotes a complex sensation including odor or aroma and mouthfeel. Most of the panelist's evaluations (82%) were between the values fair to excellent. Only 8% of the panelists evaluated the flavor of the product as poor.

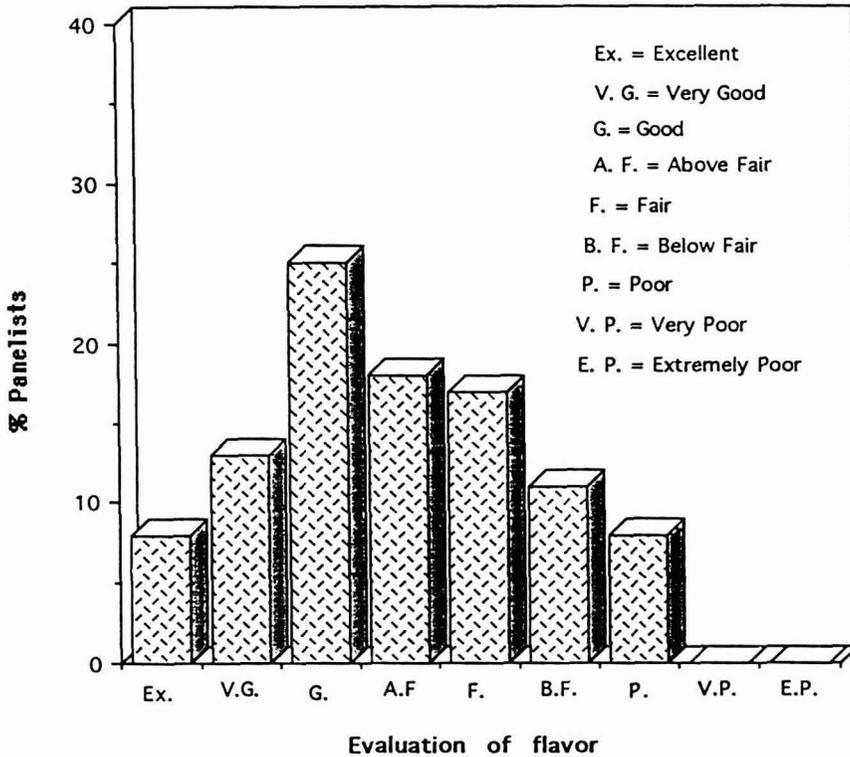


FIG. 4. SENSORY EVALUATION OF FLAVOR OF SIMULATED BAKED POTATO PRODUCT

In terms of overall acceptance (Fig. 5) of the simulated baked product through these discriminatory and preference tests, 84% of the panelists rated the product as fair to excellent. Approximately 8 and 6% panelist rated the overall acceptability of the product as below fair and poor, respectively.

In conclusion, this study demonstrated that manufacturing an acceptable simulated prebaked frozen potato product from small, extra large and off-grade potatoes through the use of extrusion processing is feasible, and may have potential for implementation in the market place. Steaming of the potatoes and addition of a binding agent was necessary to prevent problems during extrusion and to hold the extrudates together. Plastic potato shaped molds helped in reforming the extruded potatoes. Stable skin was developed by dipping in a specially formulated batter of wheat flour, glycine and dextrose, and subsequent frying. It must also be mentioned that in spite of care exercised prior to and during extrusion the texture of the simulated baked product received slightly lower ratings

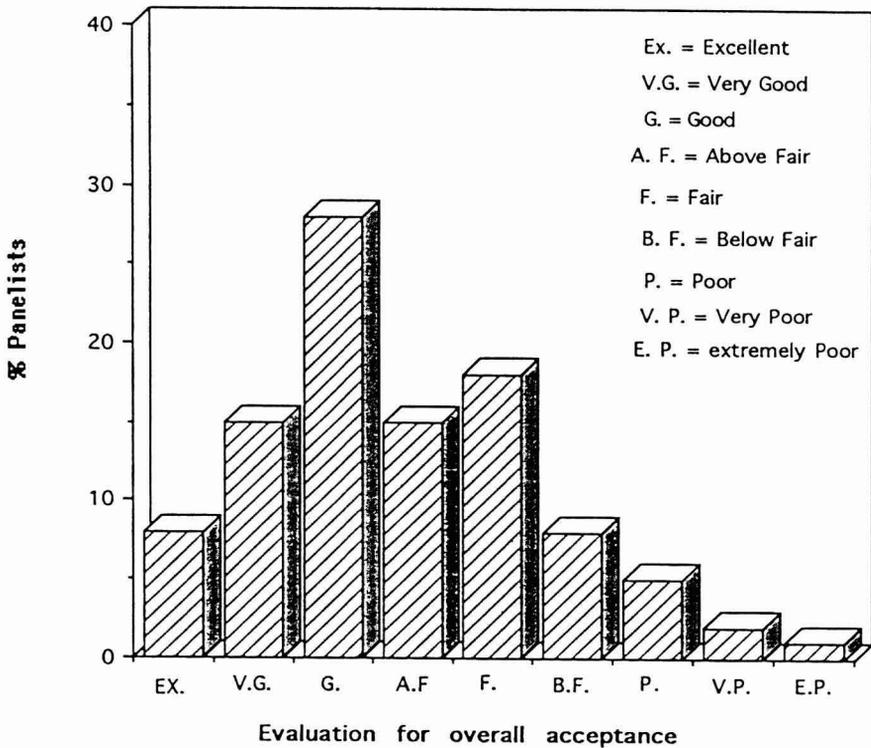


FIG. 5. SENSORY EVALUATION FOR OVERALL ACCEPTANCE OF SIMULATED BAKED POTATO PRODUCT

by the panelists in comparison to other quality attributes and needs further investigation.

### ACKNOWLEDGMENT

Acknowledgment is made to the Michigan Agricultural Experiment Station and the Michigan Potato Industry Commission for their support of this research.

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# ABSORPTION OF DISSOLVED ASCORBATE BY LIVE CHANNEL CATFISH (*ICTALURUS PUNCTATUS*)

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## ABSTRACT

*Live channel catfish were exposed to 0.3% sodium ascorbate solution at pH 5. Ascorbic acid concentration in the fillets was found to increase from 13 ppm initially (control group) to 51 ppm and 43 ppm for fish exposed to ascorbate for 8 and 24 h, respectively. For the group of fish exposed to 8 h of ascorbate followed by 16 h of dechlorinated water, pH 7, the fillets contained 27 ppm ascorbic acid. Fish which were exposed to water (without ascorbate) at pH 5 for 24 h showed a comparable ascorbic acid level in fillets to those fillets from the control group. The absorption of ascorbate via gills and/or intestines and distribution to required sites within the muscle tissue could serve to increase the stability of the fish fillet during subsequent processing and storage. Introduction of water-soluble antioxidants to aquacultured fish species by this approach requires further exploration.*

## INTRODUCTION

In the last decade, farm-raised catfish sales led the expansion of aquaculture in the United States, going from 46 million pounds in 1980 to 342 million pounds in 1989. In 1990, more than 360 million pounds of catfish were processed, making it the 5th most consumed finfish in the United States (USDA 1991).

One of the most serious problems facing the catfish processing industry is the short shelf-life of the frozen product (Ammerman 1985). Deterioration of catfish during frozen storage is most often characterized by the production of rancid odor and flavors from oxidation of unsaturated fatty acids. In addition to changes in

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flavor properties, lipid oxidation can lead to changes in texture, color, water holding capacity and nutritive properties of the muscle.

Antioxidants can be used to control lipid oxidation in fish muscle. Both multi-syringe injection and vacuum tumbling systems are being used to apply erythorbate-based antioxidants to catfish. Particularly with the injection system, localization of antioxidant at the injection site may occur leaving other sites within the muscle tissue unprotected. Additional drawbacks of the injection system are its associated sanitation problem and the addition of water to the fish tissue. While dipping of fish fillets in erythorbate has been proven to control rancidity (Bilinski *et al.* 1981), such treated product may develop a "granular" mouthfeel and "chalky" appearance (Palmer and Bowers 1972; Moledina *et al.* 1977). Dietary supplementation with tocopherol has also been shown to increase the oxidative stability of fish muscle (O'Keefe and Nobel 1978; Frigg *et al.* 1990); however, heat-labile antioxidants, such as tocopherol or ascorbic acid, are susceptible to degradation during extrusion and storage of fish feed. Although there is now a stable form of ascorbic acid (L-ascorbyl-2-polyphosphate) available for feed use (Grant *et al.* 1989), water-soluble vitamins such as ascorbic acid are not stored in appreciable quantities in the animal's body. Excesses of ascorbic acid are readily metabolized and excreted by the kidney (Robinson and Wilson 1985). Therefore, incorporation of ascorbic acid and its derivatives into feed is not a practical way to accumulate these compounds in the muscle tissue. On the other hand, absorption of dissolved ascorbate by live channel catfish via the gills and/or intestines immediately prior to processing could potentially be an effective method to apply water-soluble antioxidants. The transepithelial fluxes of ascorbate across the biological cell membrane would lead to cellular accumulation of this antioxidant, which could then be utilized to inhibit oxidative reactions during processing. While these early oxidative reactions are difficult to detect or measure, they may be of significance to later oxidation during storage (Lingnert 1991). Introduction of antioxidant to the live animal would also capitalize on the animal's metabolic system for distributing the antioxidant to required sites of utilization. The objective of this preliminary study, therefore, was to demonstrate the ability of live channel catfish to absorb ascorbate and erythorbate from the water and distribute them to muscle tissue.

## MATERIALS AND METHODS

### Stability of Ascorbate and Erythorbate

Sodium ascorbate (Sigma Chemical Co., St. Louis, MO) and sodium erythorbate (Pfizer Inc., New York) were dissolved separately in deionized water to yield a concentration of 0.1%. Each solution was subsequently adjusted to pH 5.0 with

acetic acid. The stability of these compounds at 25C and in the dark was assessed by high performance liquid chromatography (HPLC).

### **Fish Supply**

Live channel catfish (*Ictalurus punctatus*), approximately 300 g each, were obtained from a commercial fish farm in Thomaston, GA during late summer. Fish had been administered pelletized feed (Gold Kist Inc., Atlanta, GA) containing ascorbic acid as the source of vitamin C. Fish were transported to the laboratory (45–60 min) in plastic containers filled with pond water (21C, pH ~ 7) and aerated with battery-operated air pumps at a loading rate of 100–150 g fish per liter of water.

### **Variability of Endogenous Ascorbic Acid in Catfish Muscle**

Five channel catfish (without treatment) were sacrificed to examine the variability of endogenous muscle ascorbic acid. The ascorbic acid content of the fillets (white and dark muscle) was immediately determined in each fish.

### **Exposure of Live Catfish to Dissolved Ascorbate**

Fish were randomly divided into groups of three. Each group was held under dim light conditions in a well-aerated aquarium containing 30 L of dechlorinated water, sodium ascorbate or sodium erythorbate at 21C without feeding for up to 24 h. The dechlorinated water was prepared by adding 1 mL of dechlor-ease (Mydor, Pembroke Pines, FL) to 30 L of tap water. Appropriate amounts of ascorbate and erythorbate were dissolved in dechlorinated water to yield the desired concentrations. Upon completion of specific treatments, fish were sacrificed and muscle tissue analyzed for ascorbic acid and erythorbic acid content.

### **Sample Preparation**

Fish were asphyxiated with carbon dioxide saturated water for 3 to 5 min and then filleted. Fillets were washed in water and immediately placed in a –100C freezer until further analysis.

### **Extraction of Ascorbic and Erythorbic Acids.**

The modified protocol of Sapers *et al.* (1990) was used to extract ascorbic acid and erythorbic acid from catfish muscle tissue. Frozen muscle tissue was thawed and blended with a Black and Decker Handy Chopper (Shelton, CT). Using a

polytron mixer (Brinkmann Instruments Inc., Westbury, NY), the minced muscle tissue (4.0 g) was homogenized in 20 mL of cold extracting solution (40 mM sodium acetate buffer, pH 3.8, containing 15% methanol). An aliquot of the homogenate was filtered through Whatman No. 5 filter paper under vacuum. The filtrate was further filtered through a C<sub>18</sub> PrepSep column (Fisher Scientific, Norcross, GA) and a Gelman (Ann Arbor, MI) nylon acrodisc, 0.2  $\mu$ m, before quantification of the ascorbic/erythorbic acid content by HPLC.

### Quantification of Ascorbic and Erythorbic Acids by HPLC

The ion-pairing HPLC method of Tsao and Salimi (1982) was modified for the quantification of ascorbic and erythorbic acid from muscle tissue. A HPLC system (Micromeritics, Norcross, GA), equipped with a Brownlee Spheri-5 ODS analytical column (4.6  $\times$  220 mm, 5  $\mu$ m packing, Applied Biosystems Inc., San Jose, CA), a RP-C<sub>18</sub> silica guard cartridge (Applied Biosystems Inc., San Jose, CA), an injector (Rheodyne Inc., Cotati, CA) with a 20  $\mu$ L sample loop, an electrochemical detector (Model 656, Brinkmann Instruments Co., Westbury, NY) and an integrator (Model 3390A, Hewlett Packard, Avondale, PA), was used. The mobile phase, which consisted of 40 mM sodium acetate and 1 mM n-decylamine (Sigma Chemical Co., St. Louis, MO) in methanol-water (15:85), was adjusted to pH 4.0 and degassed before use. The flow rate was maintained at 0.9 mL/min while the oxidation potential of the electrochemical detector was set at +0.7 V versus the Ag/AgCl reference electrode.

### Statistical Analysis

Data collected from this study was analyzed by linear regression, the general linear model and Duncan's multiple range tests using SAS (1985) programs.

## RESULTS AND DISCUSSION

### Stability of Ascorbate and Erythorbate

Ascorbic acid was selected as an antioxidant because it is water soluble and it has been shown to function as a synergistic antioxidant with tocopherol in *in vitro* systems by regenerating tocopherols (Leung *et al.* 1981; Barclay *et al.* 1983; Sato *et al.* 1990). Selection of the ascorbic acid isomer for use in the subsequent study was based on the stability of the sodium salts of ascorbic acid (L-isomer) and erythorbic acid (D-isomer) over a period of 30 h (Fig. 1). Linear regression showed that ascorbate (slope = -0.44,  $r = 0.98$ ) was more stable than erythorbate (slope = -0.70,  $r = 0.98$ ) at room temperature water and pH 5.0.

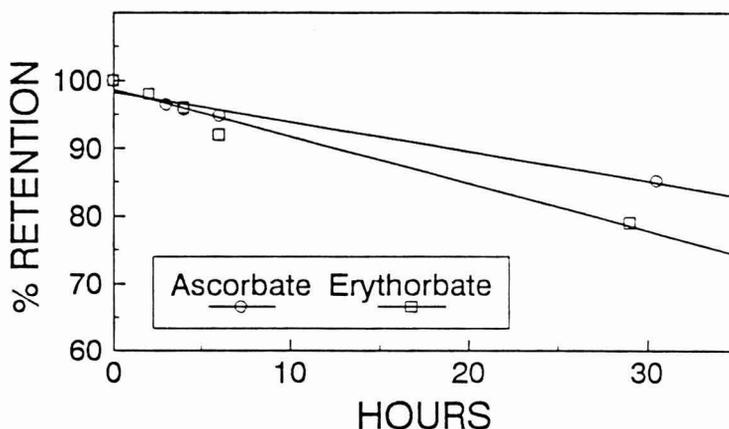


FIG. 1. STABILITY OF ASCORBATE AND ERYTHORBATE IN WATER AT 25C IN THE ABSENCE OF LIGHT  
Initial concentration 0.1% and pH 5.0.

### Variability of Endogenous Ascorbic Acid in Catfish Muscle

The initial ascorbic acid concentration in the muscle tissue of five channel catfish was  $12.4 \pm 2.9$  ppm. Three fish were, therefore, considered an adequate sample size for the subsequent absorption experiments.

### Exposure of Live Catfish to Dissolved Ascorbate

Table 1 demonstrated that the ascorbic acid concentration in muscle increased when live channel catfish were exposed to dissolved ascorbate. Following exposure of fish for 8 h to a solution of 0.3% sodium ascorbate, pH 5, the ascorbic acid content increased almost 4 fold compared to the control group (time 0). Exposure to the same solution for 24 h led to a significantly smaller increase. It would appear, therefore, that by 24 h, ascorbic acid removal exceeded the rate of ascorbic acid uptake into the muscle tissue either through increased catabolism or decreased absorption from the water. Even higher losses of ascorbic acid from muscle tissue were recorded when fish were transferred to dechlorinated water for 16 h following the 8 h exposure to the pH 5 ascorbate solution. When exposed to pH 5 untreated water for 24 h, however, ascorbic acid levels were comparable to levels in the control. While "stress" has previously been associated with depletion of ascorbic acid in fish tissue over a five-day experimental period (Tucker *et al.* 1987), the slight acidic pH encountered by the fish in these studies over 24 h did not appear to affect muscle ascorbic acid concentrations.

TABLE 1.

| MUSCLE ASCORBIC ACID CONCENTRATION FOR CHANNEL CATFISH |    |                   |                                  |
|--|----|-------------------|----------------------------------|
| % AA <sup>1</sup>                                      | pH | Exposure time (h) | Ascorbic acid (ppm) <sup>2</sup> |
| Control  |    | 0                 | 13.4 ± 2.1 D                     |
| 0.3  | 5  | 8                 | 50.8 ± 5.8 A                     |
| 0.3  | 5  | 24                | 43.2 ± 5.2 B                     |
| 0  | 5  | 24                | 14.3 ± 0.5 D                     |
| 0.3  | 7  | 24                | 23.7 ± 9.9 C                     |
| 0.3  | 5  | 8                 |                                  |
| followed by  |    |                   |                                  |
| 0  | 7  | 16                | 27.2 ± 2.4 C                     |

<sup>1</sup>AA = sodium ascorbate

<sup>2</sup>Each value is the mean of 3 fish ± standard deviation.

Means with the same letter are not significantly different ( $p \leq 0.05$ )

Ascorbic acid levels in fillets were lower when fish were exposed to a pH 7 solution than a pH 5 solution (Table 1). Differences in uptake could be explained by the degree to which ascorbic acid was ionized. Ascorbic acid ( $pK_a$  at 4.2 and 11.8) exists predominantly with a single negative charge at the physiological pH of 7 (Andrews and Crawford 1982). At pH 5, however, approximately 14% of the ascorbic acid will be nonionized and might be capable of passive transport through the hydrophobic cell membranes.

Erythorbic acid is not naturally present in fish tissue. The detection of 2 ppm erythorbic acid following an 8 h exposure to a solution containing 0.05% of both ascorbate and erythorbate at pH 5 supports the idea that live catfish can absorb these compounds from water. This observation also implied that the increase in muscle ascorbic acid did not arise solely from the redistribution of ascorbic acid from storage organs (spleen, kidney and liver) but could, at least, partly be attributed to absorption.

## CONCLUSIONS

This preliminary study demonstrated that live channel catfish had the ability to absorb ascorbate and distribute the antioxidant into the muscle tissue. Further

research is warranted to explore optimal conditions for ascorbate absorption and to test the effect of the absorbed ascorbate on oxidative stability. Introduction of antioxidant by this new approach could be potentially applied during live hauling of catfish to the processing plant.

### ACKNOWLEDGMENTS

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# CHARACTERIZATION OF THIN LAYER SUSCEPTORS FOR THE MICROWAVE OVEN

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## INTRODUCTION

Heating and cooking in the domestic microwave oven has become very popular, especially in the USA, in the last decade. Although the microwave oven was originally used primarily for defrosting and reheating of precooked foods, great efforts have been made by food manufacturers to expand and diversify its use.

Heating and cooking in a microwave oven involves conversion of electrical to thermal energy within the product. The temperature profile within the product is largely affected by internal heat generation due to absorption of electrical energy from the microwave field (Komolprasert and Ofuli 1989).

The most important electrical properties, determining the levels of absorbed power,  $P_A$ , by the product are: The dielectric constant,  $\epsilon'$ , the loss factor,  $\epsilon''$ , the attenuation factor,  $\alpha$ , and the intrinsic impedance,  $Z$ , of the product relative to free space in the cavity. These parameters depend on product composition and primarily on its moisture as well as ion content, and they may vary with temperature.

If the incident power in the microwave oven is considered to be unity, then:

$$P_A = 1 - P_T - P_R \quad (1)$$

where:

$P_A$  = Absorbed power by the product

$P_T$  = Transmitted power

$P_R$  = Reflected power

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The complex permittivity,  $\epsilon^*$ , of a product is related to the dielectric constant  $\epsilon'$ , and the loss factor,  $\epsilon''$ , by the following equation:

$$\epsilon^* = \epsilon' - i\epsilon'' \quad (2)$$

The intrinsic impedance,  $Z$ , of a product relative to free space is given by:

$$Z = Z_o(\epsilon^*)^{-1/2} \quad (3)$$

where:  $Z_o$  is the impedance of free space ( $Z_o = 377 \text{ Ohm}$ ).

The attenuation factor,  $\alpha$ , determines the amount of power absorbed at any depth,  $X$ , from the surface (Mudgett 1986; Von Hippel 1954):

$$P_x = P_o \exp(-2\alpha x) \quad (4)$$

$$\alpha = \frac{2\pi (\epsilon')^{1/2} [(1 + \text{tg}^2\delta)^{1/2} - 1]^{1/2}}{\lambda_o [2\epsilon_o]^{1/2}} \quad (5)$$

$P_x$  = Power level at depth  $X$  from the surface

$P_o$  = Incident power

$\lambda_o$  = Wavelength in free space

$\text{tg}\delta = \epsilon''/\epsilon'$

The impedance mismatch between the product and the environment determines the fraction of power transmitted at and reflected from the product's surface.

The greater the mismatch, the more incident power is reflected from the air-product interface, and the less is absorbed by the product (Komolprasert and Ofuli 1989; Mudgett 1986).

The magnitude of the reflection is determined by the extent of the impedance mismatch as given in the following equation:

$$P_R = \left[ \frac{Z \cos \phi - Z_o \cos \psi}{Z \cos \phi + Z_o \cos \psi} \right]^2 \quad (6)$$

$P_R$  = Fractional power reflected

$\phi$  = Reflection angle of wave from the dielectric

$\psi$  = Refraction angle of wave in dielectric

The heating characteristics of a product in a microwave oven depends, of course, also on its thermal properties, the most important one being the thermal conductivity.

One problem with the microwave oven is that it does not crisp and/or brown well and may leave the food soggy (Sacharow 1988). Although cooking is rapid, the relatively low food surface temperature and even lower-surrounding air temperature does not enable these processes to occur. To achieve significant browning and/or crisping reactions, the food surface has to be elevated to 150C at least (Anon. 1989; Sacharow 1988; Sheppard 1989).

One method to achieve browning or crisping of a product in the microwave oven is to use special browning dishes, which were developed especially for this purpose. The disadvantage of this method is, however, that this dish has to be preheated for several minutes before placing the product in it; thus, the major advantage of this type of food preparation, namely speed, is lost.

A second method involves special formulation or treatment of the product such as: coating its surface with oil or fat. This method, however, often does not work, is not applicable to many products and requires the costly and time-consuming different formulations for different products.

The third method that was developed in recent years and is the most efficient of the three, involves the use of a metallized thin layer susceptor (Sheppard 1989). Susceptors consist of a metallized (most commonly, aluminized) plastic film laminated to paperboard on top of which, or within which, the product is placed. They absorb microwave power (direct dielectric coupling) and convert it to heat, which is transferred to the product by conduction or radiation, creating localized areas of high temperature on the food surface. The temperature of the susceptor may reach 200–260C. This temperature depends upon the food and on the properties of the metallized film. The high temperature achieved, it evaporates the water and crisps and browns the food. The metallized film is responsible for the amount of the microwave energy absorbed and converted into sensible heat (Rozenkranz and Higgins 1987; Sacharow 1988). According to Lingle (1987) and Spaulding (1987), a susceptor that more closely resembles aluminum foil is more absorbent of microwaves than one that is clearer in appearance. The foil-like susceptor heats more quickly, but also reflects much more of the incident power. The thickness of the metal layer cannot be measured directly. Thus, electrical conductivity (Ohms per square) and optical density (O.D.) are measured and normally used to control the level and uniformity of this layer (Sacharow 1988; Holmgren 1987). A better method to describe and predict the performance of a susceptor is to use its electrical properties as a criterion.

The present study deals with the electrical properties of three susceptors (which differ in thickness of the metallized layer on the polyester carrier) and shows the effect of composition on the ability to heat a product.

## MATERIALS AND INSTRUMENTATION

The susceptors were prepared on an industrial vacuum metallizer followed by an industrial laminator and consisted of three PET (polyethylene terephthalate) films metallized to three different levels of aluminum, expressed as Optical Density (O.D.): 0.25, 0.30 and 0.35.

The metallized PET was laminated to a 300 g/m<sup>2</sup> paperboard using a polyurethane based adhesive. The size of each susceptor was 19 × 14 cm. A fiber-reinforced paper napkin wet by 100 g of distilled water was used to simulate a product. An Ammana Radarange R.S. 560A domestic microwave oven (rated power output 700 W, given by the manufacturer) was used for all experiments.

The maximum temperature of the susceptor's surface (reached at different heating times) was measured by Melting Point Standards (Omega Engineering, Inc., Stamford, CT 06907), which do not interfere with the heating process. Infrared (IR) thermography was also used to measure temperatures. In the latter case, a scanning IR sensitive detector with a color television display connected to a thermovision 880 (Aga Corp.) was used. Intrinsic impedance was measured, at 1.96 GHz, using a waveguide (WR 340, frequency range 1.7–2.6 GHz, cross section 86 × 43 mm). The results were collected by a Network/Spectrum Analyzer (MS 620J Anritsu, power output 1 mw) and corrected for the waveguide impedance (806 ohm). This instrument was also used to measure reflected and transmitted power.

## METHODS

The susceptor was placed at the center of the oven's floor on top of an insulating Teflon sheet. A fiber reinforced paper napkin wet by 100 g of distilled water was placed on top of the susceptor. The maximum temperature of the susceptor's surface, during heating, was measured using 0.01–0.03 g of Melting Point Standards. At least five replicates were tested at six points on each sample and the reported values are averages of these determinations. The oven was allowed to cool to room temperature between tests. Susceptors' temperature data, during cooling, was measured using an Infrared camera system. The thermographs were taken 15 s after the completion of heating (the susceptor was removed from the microwave oven). All reported data were corrected for target emissivity. The temperature at "0" (zero) time was measured by Melting Point Standards as mentioned above. For the simulated product (wet napkin) the reported temperatures are an average of the results collected by the IR camera system. The IR thermographs were taken immediately after the completion of heating. Duplicate tests were carried out with very good repeatability. The reported data are the average of the two measurements.

## RESULTS AND DISCUSSION

The temperature rise of three susceptors with different optical densities as a function of heating time is shown in Fig. 1. It can be seen that the susceptor metallized to the level of O.D. = 0.30 heated faster than the others. All susceptors reached a maximum temperature of about 200°C.

In Fig. 2 the susceptor's cooling is presented. It can be seen that the O.D. = 0.30 susceptor cooled down slower than the others. Thus, a product placed on top of this susceptor would heat and absorb more energy after the heating process is completed.

The percent of incident power and energy absorbed by the susceptor and by the food simulant placed on top of it during heating is shown in Fig. 3 and 4, respectively. The control in the latter figure refers to a napkin heated without a susceptor.

The advantage of the O.D. = 0.30 susceptor is evident ( $p < 0.001$ ). The effect of optical density on the intrinsic impedance of the different virgin susceptors is shown in Fig. 5. The real part of the impedance decreases linearly with increasing optical density. On the other hand, the imaginary part goes through

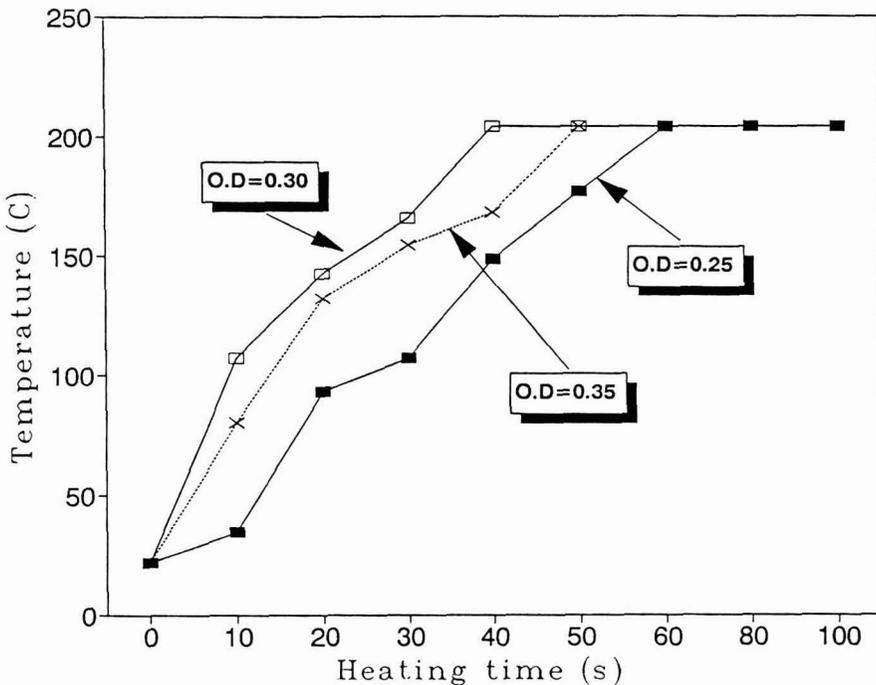


FIG. 1. EFFECT OF OPTICAL DENSITY ON SUSCEPTOR'S HEATING UP ( $P = 700$  watt)

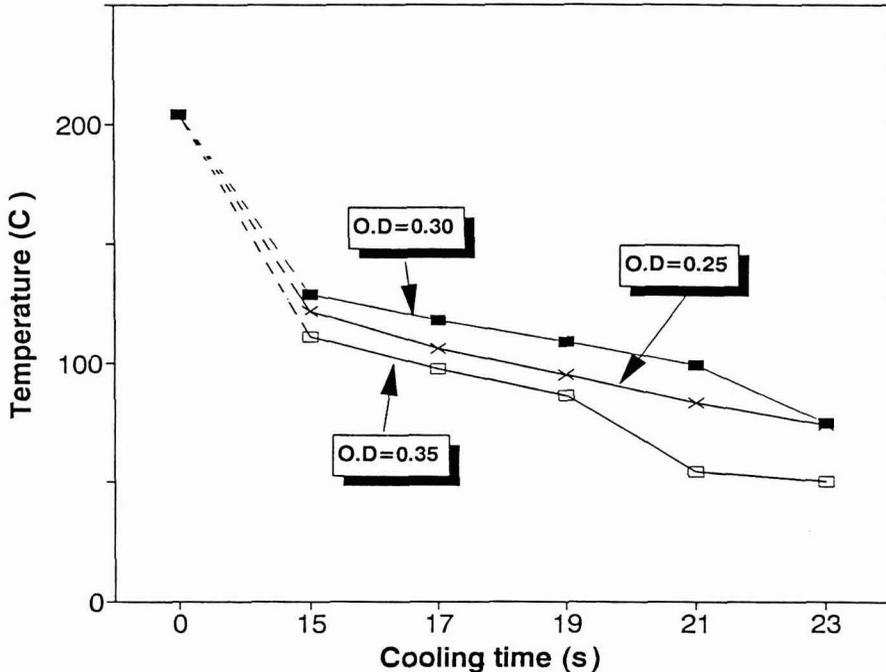


FIG. 2. TEMPERATURE OF SUSCEPTOR DURING COOLING AFTER 100 s OF HEATING

a minimum ( $t$ -test,  $p < 0.001$ ), as the optical density changes from 0.25 to 0.35. It is important to emphasize that this conclusion is based on three optical densities only.

The imaginary part of the intrinsic impedance increases during heating (Fig. 6). As the imaginary part represents capacitance, this increase probably reflects the increase in capacitance as a result of the formation of discontinuities in the metal layer of the susceptor. On the other hand, the real part did not change significantly during heating.

A closer inspection of Fig. 6 reveals that the imaginary part of the impedance of the O.D. = 0.35 susceptor is slightly lower than that of O.D. = 0.30 and significantly lower than that of O.D. = 0.25.

The mismatch between the impedance of the O.D. = 0.25 susceptor and that of air is about 700 ohms, whereas the mismatch of the O.D. = 0.35 and O.D. = 0.30 susceptors was about 200 ohms only (see Fig. 7).

These results correspond well with the results of energy absorbed by the load placed on top of the susceptor, namely, the smaller the mismatch, the greater was the efficiency of heating and the amount of energy absorbed by the susceptor. This result is in general agreement with the statement of Mudgett (1982) that

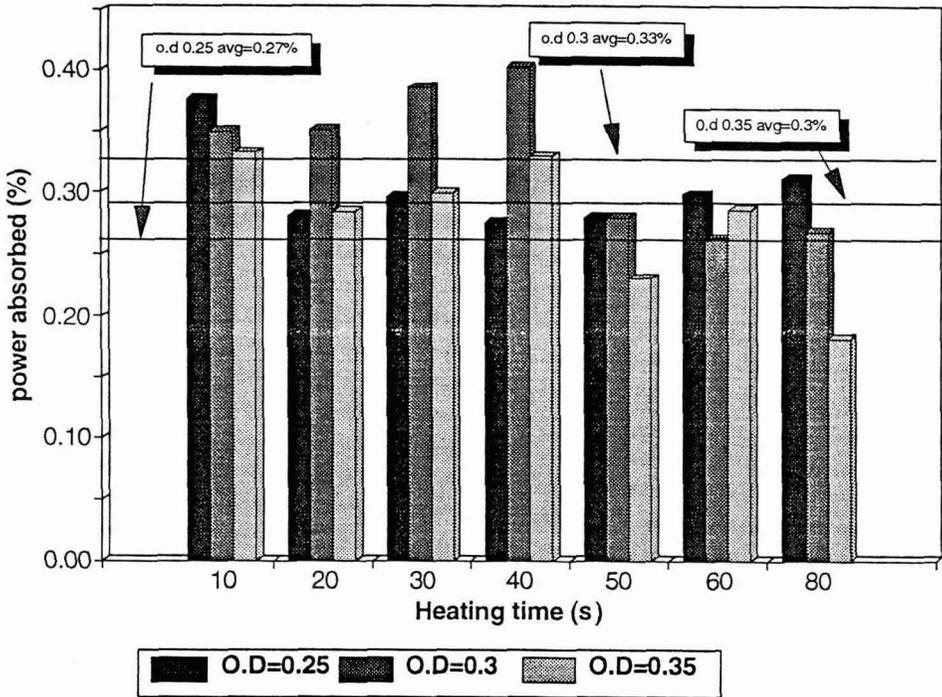


FIG. 3. RELATIVE ENERGY ABSORBED IN A SUSCEPTOR

the greater the mismatch between the impedance of a food and that of air, the more incident energy is reflected from the air food interface, and the less energy is transmitted to the product.

The changes in reflected, transmitted and absorbed power (as measured by the network/spectrum analyzer) of the susceptor with the O.D. 0.30 after heating for varying times in the microwave oven are shown in Fig. 8. It can be seen that the reflected and absorbed power change during heating.

Similar results were obtained with the O.D. = 0.25 and O.D. = 0.35 susceptors. These changes may be explained by metal dislocations during heating and PET film expansion and relaxation. Significant differences between the susceptors of the three optical densities were found in the reflected power, based on five measurements for each sample. Maxima of 80, 75 and 70% were found for the O.D. = 0.35, O.D. = 0.30 and O.D. = 0.25, respectively; namely, the more metal deposited on the PET film the greater the reflected power. The absorbed power by the O.D. = 0.30 susceptor was 40% of the incident one, whereas for the others, lower values were achieved. A significant difference between the absorbed power of the O.D. = 0.30 susceptor and those of O.D. = 0.25 and

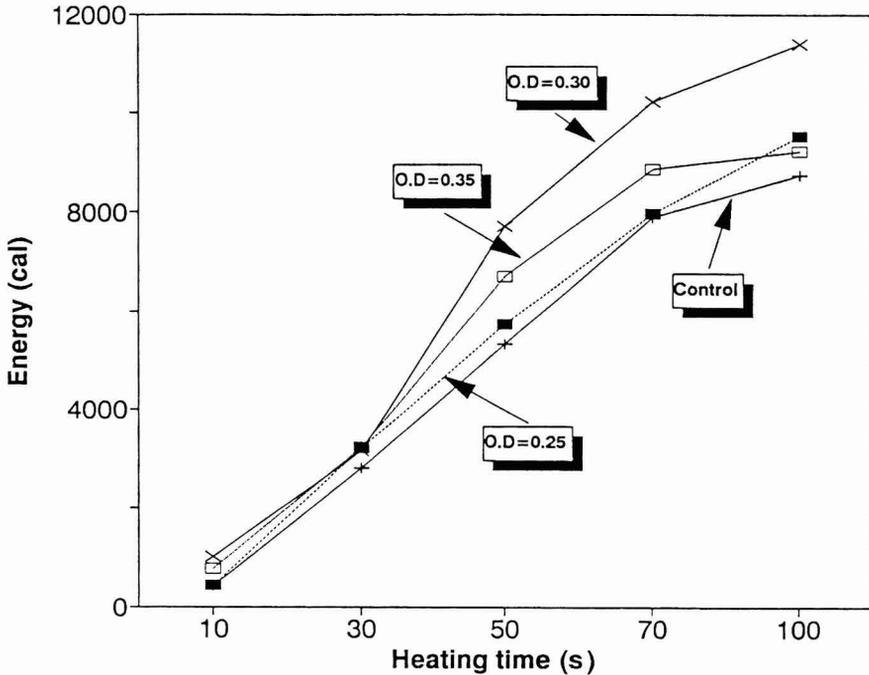


FIG. 4. EFFECT OF OPTICAL DENSITY ON THE ENERGY ABSORBED BY WATER  
( $P = 700$  watt)

O.D. = 0.35 ( $p < 0.025$  and  $p < 0.1$ , respectively) were found. These differences were insignificant, however, when comparing the O.D. = 0.25 and O.D. = 0.35 susceptors. The most efficient heating time interval was found to be 10–50 s; beyond 50 s of heating, the susceptors started to lose their ability to absorb microwave power and to heat up.

To summarize, it was found that the power absorbed and transmitted by a susceptor passed through a maximum when the optical density changed from O.D. = 0.25 to O.D. = 0.35. It was also found that the mismatch between the intrinsic impedance of the susceptor and that of air reflected its heating capability; the lower the mismatch, the faster and more uniform was the heating. Thus, the intrinsic impedance can be used as a criterion for the heating, browning and crisping capabilities of a susceptor.

### SYNOPSIS

The electrical properties and heating capabilities of three thin layer susceptors possessing three different optical densities (O.D.) were evaluated. It was found

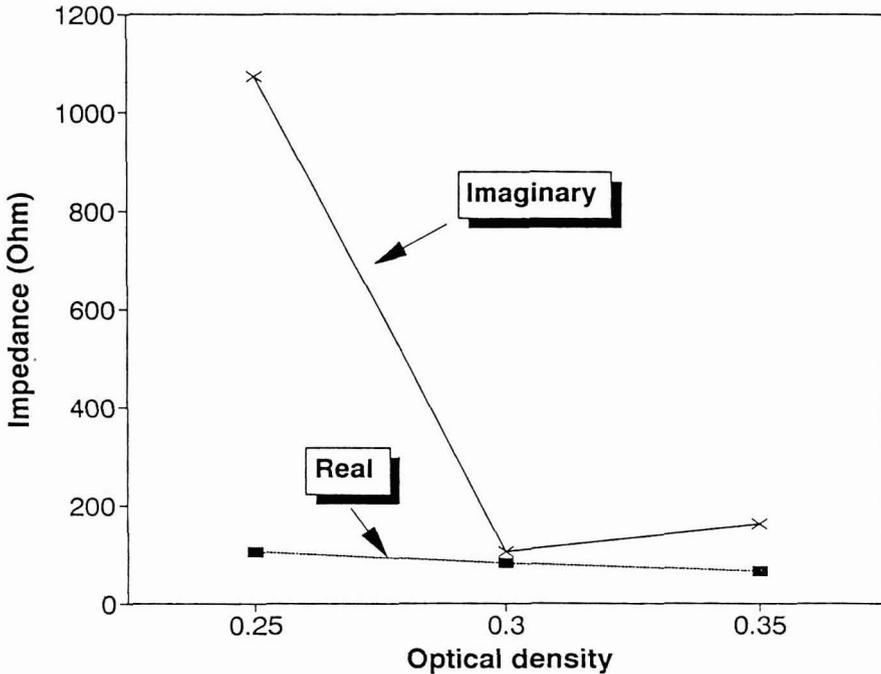


FIG. 5. RELATIONSHIP BETWEEN OPTICAL DENSITY AND INTRINSIC IMPEDANCE OF SUSCEPTOR

that the O.D. = 0.30 susceptor absorbed more of the incident microwave energy (40%) than the other two. The mismatch between the intrinsic impedance of the susceptor and that of air was found to be a good criterion for the heating capability of the susceptor: the lower the mismatch the better the heating.

### ACKNOWLEDGMENT

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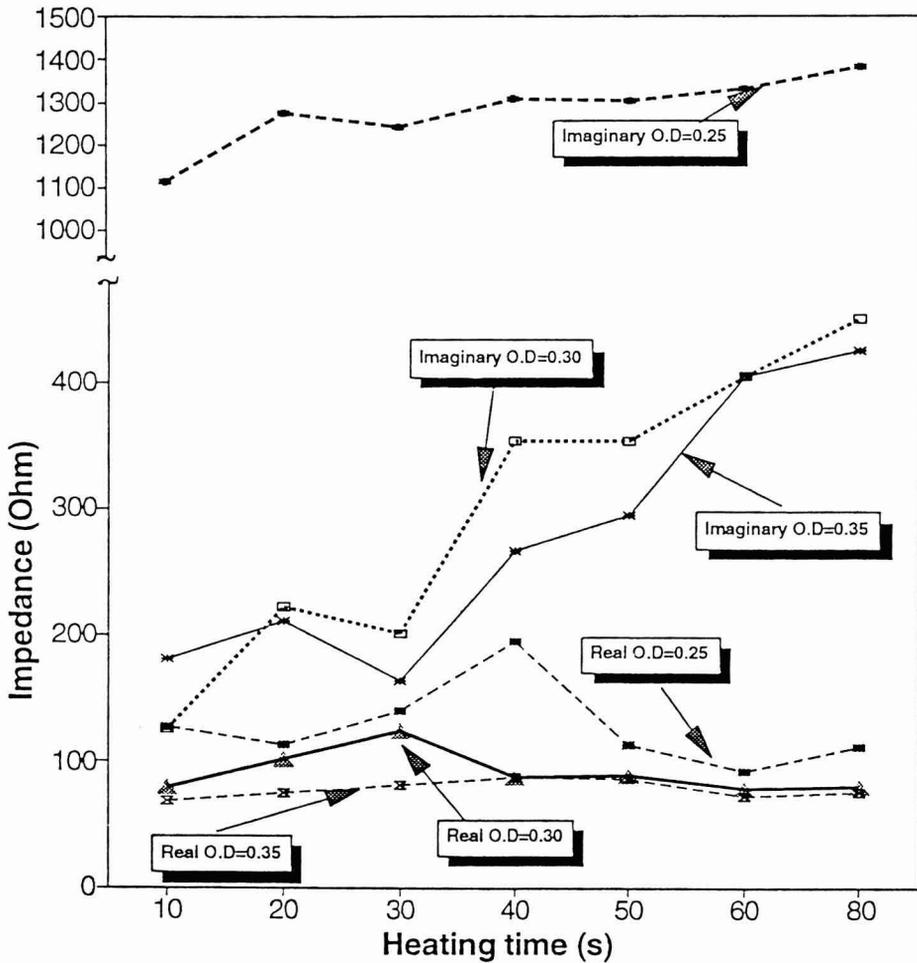


FIG. 6. EFFECT OF HEATING TIME ON SUSCEPTOR'S IMPEDANCE ( $P = 700$  watt)

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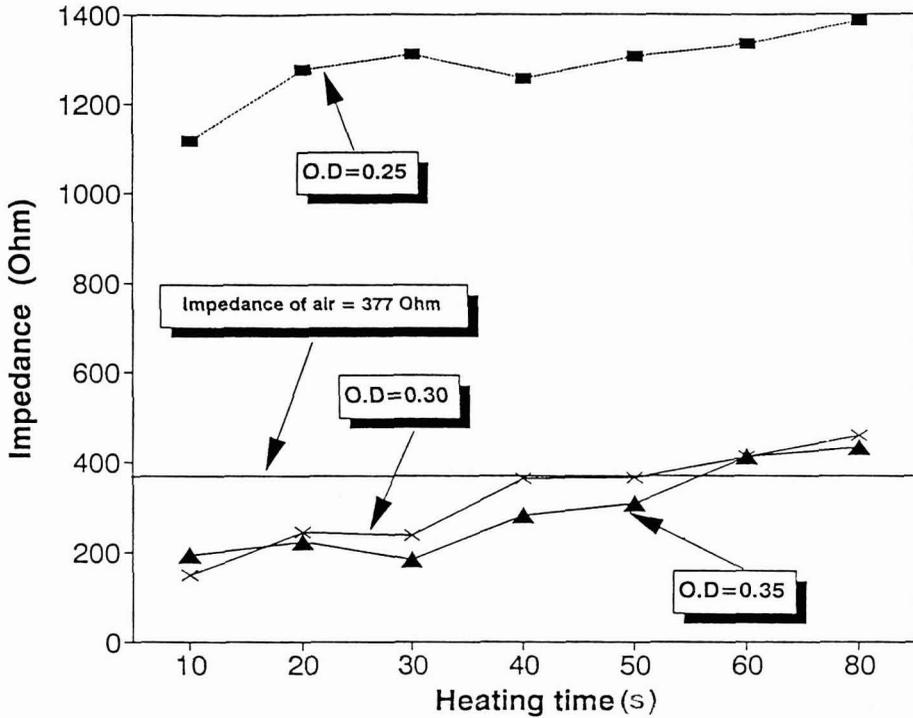


FIG. 7. INTRINSIC IMPEDANCE OF STUDIED SUSCEPTORS

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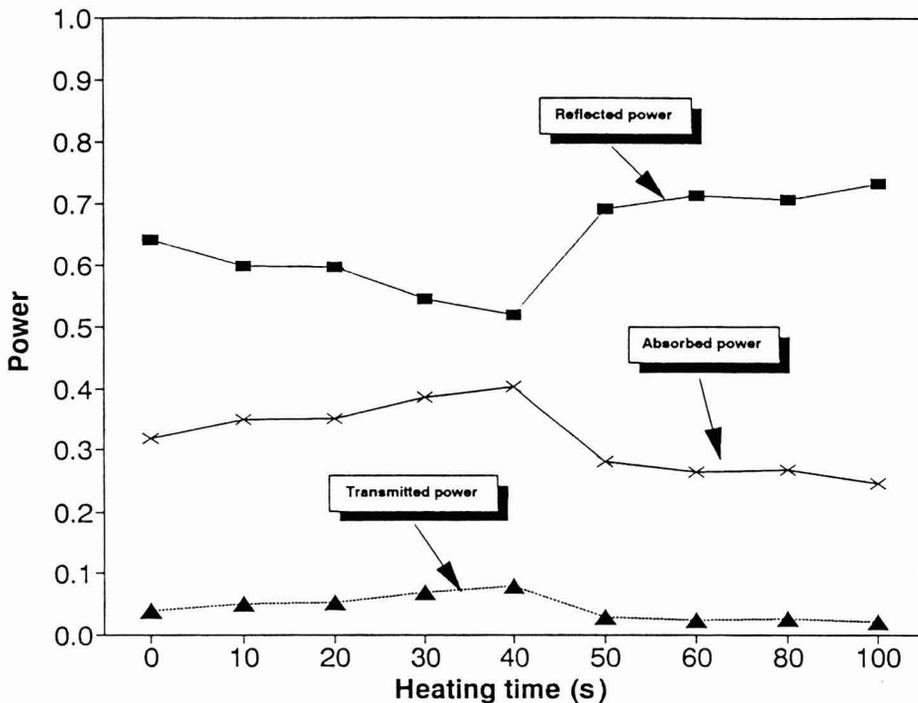


FIG. 8. EFFECT OF HEATING TIME ON REFLECTED, TRANSMITTED AND ABSORBED POWER (O.D. = 0.30, P = 700 watt)

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# EFFECTS OF FILM OVERWRAPPING, VACUUM PACKAGING AND VACUUM SKIN PACKAGING ON PSYCHROTROPHIC COUNTS AND CHEMICAL CHANGES OF ICED CHANNEL CATFISH

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## ABSTRACT

*Microbial and chemical changes during iced storage of fillets from channel catfish (*Ictalurus punctatus*) with film overwrapping, vacuum packaging, or vacuum skin packaging were determined by psychrotrophic plate counts, thiobarbituric acid (TBA), free fatty acid, pH, and ammonia production. Vacuum-packaged fish had significantly lower ( $p < 0.05$ ) psychrotrophic bacteria counts than overwrapped fish. A significantly higher free fatty acid content was also observed after week two in overwrapped samples. However, vacuum-packaged catfish had the same pH, TBA number, and ammonia production as overwrapped fish on the sampling days throughout three weeks of storage.*

## INTRODUCTION

According to USDA statistical reports (USDA 1991), 164 million kg of catfish were processed in 1990 compared to 21 million kg in 1980. In order to increase the growth rate in the market, the production of catfish with good storage characteristics in retail outlets will be very important to the industry. At present, overwrapping catfish with flexible film is the most common packaging method for retail display. However, overwrapping does not yield a leakproof package (Murray and Gibson 1971), and fish juice may accumulate in the package. The maintenance of quality of iced fresh fish by packaging system is one route to marketing success.

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Previous investigators have shown that an overwrap has little or no effect on refrigerated or iced shelf-life when compared to unpackaged fish (Huss 1971; Murray and Gibson 1971; Phillips and Cobb 1977), while vacuum packaging of seafood products has extended seafood shelf-life in many studies. Hansen (1972) stated that the storage life in wet ice of gutted, prepacked trout and herring greatly depends on the access of air to the fish surfaces during storage and that vacuum packaging in airtight films extended the shelf-life. However, vacuum packaging with barrier film may cause the retention of off-odors which may be objectionable to consumers and the potential risk of botulism (Daniels *et al.* 1986). In order to keep prepacked fresh catfish products safe and of good quality during iced storage, vacuum packaging with oxygen permeable materials might be advisable. Earlier studies focused on maintaining the quality of iced catfish (Heaton *et al.* 1972; Beauchat *et al.* 1973), but little information regarding the storage characteristics of iced vacuum packaged catfish is available. The objective of this investigation was to compare microbiological and chemical changes occurring in catfish fillets packaged by two commercial vacuum packaging methods to a traditional film overwrapped procedure during three weeks of iced storage.

## MATERIALS AND METHODS

### Collection of Samples

Channel catfish (*Ictalurus punctatus*), averaging 1.5 kg, were harvested by net from fish ponds of the Fisheries Research Unit, Auburn University, Auburn, Alabama, in late fall of 1989. Upon harvest, fish were deheaded, eviscerated, skinned, and filleted using pilot plant scale facilities and then stored on ice prior to packaging.

### Packaging Treatment and Storage

Fillets, averaging 350 g, were placed on polystyrene trays and were vacuum-packaged with E bags (Cryovac Co., Duncan, SC) using a Multivac AG 900 vacuum packaging machine (Multivac Sepp Haggemuller KG, West Germany) followed by a heat shrink process which required dipping in hot water (88C) for 2 s as reported by Teixeira *et al.* (1986). Fillets were also vacuum skin packaged with Intact® Skin Packaging film (ISPF, Trigon Packaging Co., Redmond, WA) using a Trigon Intact RM 331 Mark III Mini Intact machine. Saran® (polyvinylidene chloride, PVDC, Dow Chemical Co., Indianapolis, IN) was used to overwrap catfish fillets, which served as control. Specifications for the packaging materials are given in Table 1.

TABLE 1.  
SPECIFICATIONS OF PACKAGING MATERIAL<sup>a</sup>

| Packaging method | Composition                                      | Oxygen transmission rate <sup>b</sup> | Water transmission rate <sup>c</sup> |
|------------------|--|---------------------------------------|--------------------------------------|
| Overwrap         | Polyvinylidene chloride                          | 5 (at 20 C, 43% R.H.)                 | 1.00 (at 20 C, 85% R.H.)             |
| Vacuum bag       | Low density ethylene and vinyl acetate copolymer | 4,000 (at 22.8 C)                     | 10.08 (at 37.8 C, 100%R.H.)          |
| Vacuum skin pack | Surlyn (low density polyethylene)                | 930 (at 23 C, 75% R.H.)               | 4.00 (at 37 C, 95% R.H.)             |

<sup>a</sup> Specifications from technical literatures provided by manufacturers.

<sup>b</sup> Transmission rate = cc/m<sup>2</sup>/24 h/atm

<sup>c</sup> Transmission rate = g/m<sup>2</sup>/24 h/atm

All packaged fish samples were held with a 2:1 ice to fish ratio in ice chests (Thermos, Batesville, MS) provided with holes for drainage. Ice chests were housed in a refrigerated room at 4C to slow ice melt. Additional ice was added to chests as needed to maintain the same ice-to-fish ratio. Six replicate packaged fillets were randomly taken from each of three packaging treatments and evaluated at 1, 7, 14, and 21 days of storage for microbial count and chemical analysis. A complete randomized design was used for this study.

### Psychrotrophic Bacteria Count

Six replicates of flesh samples, 25 g each (5 × 5 cm<sup>2</sup>), from central portion of fillet surface were obtained using sterile disposable scalpels (Fisher Scientific, Pittsburgh, PA) and homogenized individually for 30 s with 125 mL sterile Butterfields phosphate buffer (Speck 1984) in Fisher brand polyethylene sample bags using a Tekmar Stomacher Lab-Blender 400. Appropriate serial dilutions were plated onto Standard Methods Agar by the spread plate method. Psychrotrophic plate counts were determined by counting the colony forming units (CFU) after the plates were incubated at 7C for 10 days (Speck 1984).

## Chemical Analysis

Chemical tests consisted of determining free fatty acid content, thiobarbituric acid (TBA) number, pH, and ammonia production. Whole fish fillet was ground and used for these analyses. Lipids were extracted from homogenized fillet by mixed solvent based on the AOAC method (AOAC 1984). The mixed oil was then titrated to determine the free fatty acid content, which was expressed as percent free fatty acid as oleic acid.

The distillation method of Tarladgis *et al.* (1960), with the following modifications, was used to determine TBA numbers. Antioxidant solution [200 mg of butylated hydroxytoluene (BHT) in 1.8 g of propylene glycol] was added to the sample before blending to prevent further oxidation as suggested by Yu and Sinnhuber (1967). Instead of glacial acetic acid, distilled water was used to make the 0.02M 2-thiobarbituric acid solution. Absorbances (A) of the TBA-malonaldehyde chromagens were read against a blank at 538 nm on a Bausch and Lomb Spectronic 20 Spectrophotometer. Results were expressed as TBA number (mg of malonaldehyde/kg of sample).

Minced fish flesh (20 g) was blended in a food processor (Regal La Machine I, Kewaskum, WI) with 80 mL of distilled water for 1 min. The homogenate was filtered through glass wool to collect 50 mL of filtrate. The pH of the filtrate was measured at room temperature with a glass electrode using a Fisher Accumet Model 950 pH Meter.

Millivolt readings from an ammonia ion selective electrode (Fisher Scientific, Pittsburgh, PA) coupled to a Fisher Model 950 pH meter were used to determine sample ammonia concentrations. Readings were taken after 1.0 mL of 10 N NaOH was added to 50 mL of filtered homogenate as previously described for pH determinations. Ammonia levels, expressed in millimolar concentration, were determined from an ammonium chloride standard curve and converted to mg NH<sub>3</sub>/g of fish.

## Data Analysis

Psychrotrophic count and chemical data for each period of storage were analyzed by analysis of variance using the general linear models procedure of PC SAS (SAS 1987). The Tukey's Studentized Range (HSD) Test at the 0.05 level was used to determine significant differences among samples packaged in three different materials and across sampling days.

## RESULTS AND DISCUSSION

After 7 days of iced storage, samples held in vacuum skin packaging and in vacuum bags showed significantly lower ( $p < 0.05$ ) psychrotrophic bacterial counts than Saran® overwrapped ones by 0.6 and 0.4 log CFU/g, respectively (Table 2). Vacuum packaging with high oxygen permeability materials slowed the growth of psychrotrophs on catfish fillets during iced storage. The lower rate of increase in psychrotrophic population in vacuum-packaged seafood products during cold storage was also observed by Harrison *et al.* (1991) and Leung (1991). Harrison and co-workers (1991) reported that the rate of increase in psychrotroph population was slightly lower in vacuum-packaged marine species including Atlantic spadefish, weakfish, and shrimp during 21 days of iced storage. Leung (1991) showed that overwrapped catfish fillets had a significantly higher population than the vacuum skin packaged ones during 16 days of storage at 4C.

Degradation of fish lipid may be caused by oxidation or enzymatic hydrolysis. Fat contents of catfish fillet samples ranged from 8.6 to 10.6%. However, TBA numbers of iced catfish ranged from 0.07 to 0.18, and no significant differences among iced catfish at the same sampling days during the entire holding period were found, regardless of packaging method (Table 3). Results indicated that

TABLE 2.  
EFFECT OF PACKAGING METHOD ON PSYCHROTROPHIC PLATE COUNT  
(LOG CFU/G FISH) IN ICE STORED CHANNEL CATFISH

| Packaging treatment  | Days of storage |       |       |       |
|----------------------|-----------------|-------|-------|-------|
|                      | 1               | 7     | 14    | 21    |
| Film overwrap        | 5.3aB           | 7.7aB | 9.3aA | 9.2aA |
| Vacuum bag           | 5.3aB           | 7.1bB | 8.7bA | 8.5bA |
| Vacuomy skin package | 5.3aB           | 7.3bB | 8.9bA | 8.7bA |

a,b

Means (n=6) in a column followed by the same letter are not significantly different at level of 0.05

A,B

Means (n=6) in a row followed by the same letter are not significantly different at level of 0.05

TABLE 3.  
EFFECT OF PACKAGING METHOD ON TBA NUMBER (MG MALONALDEHYDE/KG FISH)  
IN ICE STORED CHANNEL CATFISH

| Packaging treatment | Days of storage |         |         |        |
|---------------------|-----------------|---------|---------|--------|
|                     | 1               | 7       | 14      | 21     |
| Film overwrap       | 0.18aA          | 0.12aAB | 0.12aAB | 0.07aB |
| Vacuum bag          | 0.18aA          | 0.16aA  | 0.15aA  | 0.12aA |
| Vacuum skin package | 0.18aA          | 0.15aA  | 0.13aA  | 0.12aA |

<sup>a</sup> Means (n=6) in a column followed by the same letter are not significantly different at level of 0.05

A,B Means (n=6) in a row followed by the same letter are not significantly different at level of 0.05

vacuum packaging with high oxygen permeable film did not promote lipid oxidation. As stated by Sikorski and associates (1990), the oxidation of fish lipid is profound during frozen storage and is of minor importance under iced storage.

Lipid of iced fish undergoes enzymatic changes resulting in an accumulation of free fatty acids (FFA) (Dyer and Fraser 1959; Mazeaud and Bilinski 1976). About 20% of the lipid is hydrolyzed during the shelf-life of iced fish (Sikorski *et al.* 1990). Even though FFA production varies with species, muscle type, fat content, and storage temperature, FFA has been used as a quality index for fish (Dyer and Fraser 1959; Ke *et al.* 1977; Sikorski *et al.* 1990). Since FFA formation might affect protein denaturation (Dyer and Fraser 1959), retarding the lipid hydrolysis would help in maintaining a long-storing, high quality fish. Results of this study showed that vacuum packaging reduces lipid hydrolysis (Table 4). Catfish fillets held in vacuum skin packaging and vacuum bags had significantly lower FFA content than fish held in film overwrap on day 14. A lower bacteria population in vacuum-packaged samples (Table 2) may be one of the reasons for a lower FFA content. The lipid hydrolysis may be brought about by microbial or endogenous lipase (Huss 1988).

The vacuum-packaged fillets had the same pH and ammonia as film overwrapped ones at the same sampling days throughout the entire holding time. The pH of all fillets was 6.7 at day 1 and increased to 6.8 at the end of storage period. Ammonia content of all packaged samples increased sharply from 0.05 on day 1 to the range of 0.43 and 0.49 on day 7 and remained in the range of 0.38 and 0.54

TABLE 4.  
EFFECT OF PACKAGING METHOD ON FREE FATTY ACID CONTENT  
(MG/G FISH) IN ICE STORED CHANNEL CATFISH

| Packaging treatment | Days of storage |        |        |         |
|---------------------|-----------------|--------|--------|---------|
|                     | 1               | 7      | 14     | 21      |
| Film overwrap       | 0.04aC          | 0.05aC | 0.26aB | 0.44aA  |
| Vacuum bag          | 0.04aC          | 0.04aC | 0.17bB | 0.38abA |
| Vacuum skin pack    | 0.04aB          | 0.04aB | 0.12cA | 0.15bA  |

a,b,c

Means (n=6) in a column followed by the same letter are not significantly different at level of 0.05

A,B,C

Means (n=6) in a row followed by the same letter are not significantly different at level of 0.05

mg NH<sub>3</sub>/g for the rest of storage time. Results were similar to the report of Hearnberger *et al.* (1987). There were no significant differences ( $p < 0.05$ ) among samples with three packaging methods.

In summary, both vacuum skin packaging and vacuum bags did retard psychrotrophic bacterial growth in iced catfish. Although no effect on lipid oxidation was found, vacuum packaging did slow lipid hydrolysis in catfish. Considering the safety of vacuum packaging, the oxygen permeable packaging materials used in the study were tested by Anderson (1983) and by Garren and co-workers (Department of Food Science and Technology, University of Georgia, Athens, GA, unpublished), indicating that no botulinum toxin was detected prior to organoleptic spoilage occurring at 4C. Recently, Fujita (1990) indicated that fresh fish are a difficult area for packaging, and future studies should emphasize the selection of packaging material and the development of standardized packaging forms. Our study suggests that vacuum packaging with high oxygen permeable film may be a packaging alternative for improving the retail quality of iced fresh catfish but not for extending the shelf-life. It provides a better external appearance, and prevents leakage of fish juices.

#### ACKNOWLEDGMENTS

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# MASS TRANSFER KINETICS OF OSMOTIC DEHYDRATION OF MUSHROOMS

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## ABSTRACT

*A continuously circulated contacting reactor was designed and used for the osmotic process. Mushrooms were treated in two ways: (1) Sliced mushrooms were blanched and osmotically processed in 5, 10 and 15% salt solutions for 5, 10, 20 and 30 min at room temperature (called type 1 mushrooms); (2) Sliced and blanched mushrooms were osmotically dehydrated in 60% sucrose solution for 10 min at 50C and then transferred to the same processing conditions as mentioned in (1) (called type 2 mushrooms). The moisture content, sodium chloride and sucrose concentrations for both types of mushrooms were monitored with time. A mathematical model was used to describe the mass transfer kinetics for the two types of mushrooms. Contour plots of the mass changes in water and solutes were obtained and compared. The rates of mass changes were also determined. It was found that processing in a 15% salt solution results in the greatest ratio of rate moisture change to rate salt change for both type 1 and type 2 mushrooms. It is also the optimum condition for moisture removal and salt loading. The process method for type 2 mushroom was found the best in controlling sodium chloride penetration.*

## INTRODUCTION

Osmotic dehydration is an effective method to remove the water from food tissues while simultaneously introducing solutes such as sucrose, sodium chloride, glucose or other possible humectants. This will directly affect the water activity

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and produce a completely new environment, that may prevent microbial growth or chemical reactions from occurring. The main advantage of this dehydration practice is that it minimizes the damage to the cellular tissue and removes water in a short time period and without phase change as compared to drying. Because of incomplete dehydration, the osmotic dehydration method is usually followed by a further drying or freezing procedure in order to extend the shelf-life (Kim and Toledo 1987; Flink 1980; Hawkes and Flink 1978). Since the residues of the solutes in the food directly affect the flavor and taste. Controlling and regulating the osmotic process play an important role in attempting to improve the quality of the end products.

The shelf-life of mushrooms is only 3–7 days, even under the optimum conditions, 0C and 90% RH (Considine 1982). Canning is one of the common preservation methods (Lopez 1981). Freezing is another alternative (Gormley 1972). A blanching step prior to freezing, drying and canning is essential because the polyphenol oxidase in the mushroom is known to darken the surface color of mushrooms in a short time (Fujimoto *et al.* 1972). Quality of mushrooms is very much dependent on the color and texture (MacCanna and Gormley 1968). It has been reported that canning mushrooms will result in a shrinkage and loss of solids (McArdle and Curwen 1962). Because of the short shelf-life of mushroom, the development of a high quality processed mushroom plays an important role in the mushroom industry. Applying the osmotic dehydration method to fresh commodities has been extensively investigated. Studies emphasized the effect of various osmotic agents on the finished products (Tomasicchio *et al.* 1986; Lericci *et al.* 1985) and the effect on sensory quality of the end products (Dixon *et al.* 1976; Torreggiani *et al.* 1988; Pinnavaia *et al.* 1988). In this study, the research was aimed at two distinct objectives. The first, to investigate the kinetics of the sodium chloride, sucrose and moisture transfer during two types of osmotic processes. The second, to use a mathematical model to describe the soluble solids exchange.

## MATERIAL AND METHODS

### Material

The mushrooms (*Agarius bisporus*) used in this experiment were obtained from AMS mushroom grower located in Hamilton, Ontario, Canada. The mushrooms were transported to the experimental pilot plant for processing. They were sorted visually for color, size and contamination. A hand food slicer was employed to cut each whole mushroom into slices of approximately equal thickness (0.15 cm). Sliced mushrooms were blanched in a steam jacketed water bath for 3 min at 95C, to prevent the browning reaction by inactivation of the phenolase activity in the mushroom (Fujimoto *et al.* 1972).

### Osmotic Dehydration Apparatus

The schematic diagram of the batch recirculated osmotic dehydration system is shown in Fig. 1. The osmotic reactor is constructed of glass, with 1 cm in thickness, 20 cm inside diameter and 34 cm in height. Two perforated stainless steel screens (each with 19 cm in diameters and 420 perforated holes, 0.4 cm in diameter) were installed on the top and bottom of the chamber to prevent loss of the sliced mushrooms during processing. A 30 L tank was used for the osmotic solution. The osmotic solution was circulated using a volumetric pump (15 L/min).

### Osmotic Dehydration Process

Flow chart of the mushrooms processing diagrams is presented in Fig. 2. For each treatment 1.5 kg of freshly sliced mushrooms was processed for each run and three replications were conducted for each treatment. Mushrooms were divided into two fraction. The first one was directly processed in 5, 10 and 15% sodium

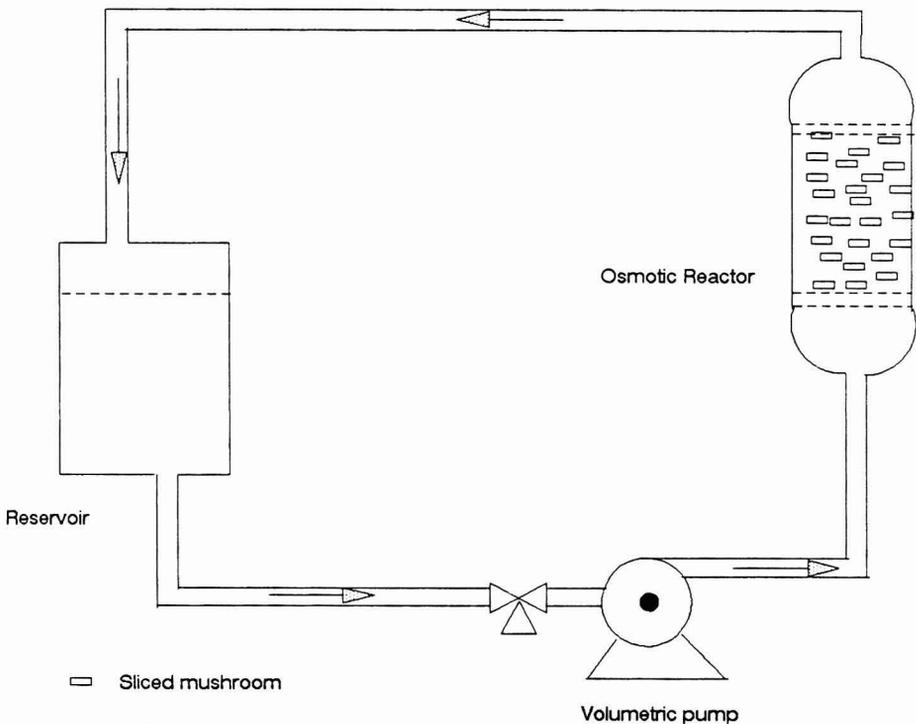


FIG. 1. SCHEMATIC DIAGRAM OF BATCH RECIRCULATED OSMOTIC DEHYDRATION DEVICE

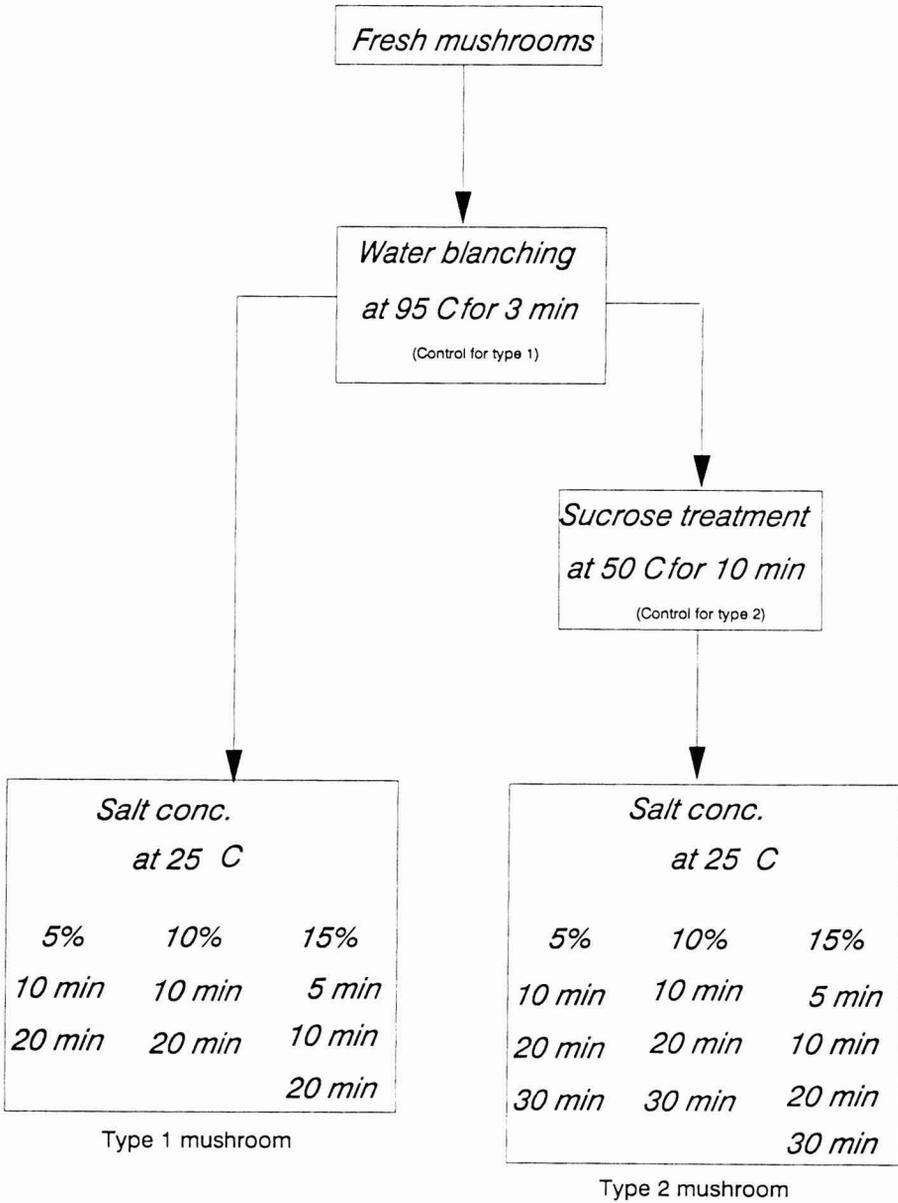


FIG. 2. FLOW CHART OF VARIOUS TREATMENTS OF OSMOTIC DEHYDRATION MUSHROOMS

chloride solutions at room temperature (named type 1 mushroom). The second was predehydrated in a 60% sucrose solution for 10 min at 50°C before being processed in the sodium chloride solution (type 2 mushroom). Two types of controls were obtained, the blanching for the type 1 and blanching and sucrose treated for type 2. The osmotically dehydrated mushroom slices were separated from each other and then stored in a walk-in freezer at -20°C.

### Moisture Content

The moisture content was determined by vacuum drying the mushroom sample at 75°C for 24 h (AOAC 1990).

### Sodium Chloride Analysis

Four grams of completely dried mushrooms were ground into powder form. Fifty mL of warm water (at 35°C) was added and the mixture shaken constantly for 15 min. The solution was then filtered through a Whatman No. 42 filter paper. The filtered solution was analyzed for the sodium content by the Varian Atomic Absorption Spectrophotometer (Model Spectr AA-300).

### Sugars Analysis

Twelve grams of mushrooms were blended with 25 mL of 80% methanol. The solution was allowed to sit at room temperature for 3 h with occasional shaking and then centrifuged for 20 min at 2500 rpm. The supernatant was filtered through Whatman No. 2 filter paper. The filtrate was passed through a 0.45 micron filter before the HPLC injection. The Waters Associates HPLC (Model 6000A, Milford, MA) equipped with a refractive index monitor was employed for the sugars analysis. It was equipped with a Na-exchange column (model: apex amino 5U) and run isocratically with a 75:25 Acetonitrile/water solvent system, at a flow rate of 2 mL/min.

## DATA REPRESENTATION

Calculation of the processing parameters such as moisture change (MC) weight change (WC), solutes concentration changes (sodium chloride  $SC_{(SA)}$  or sucrose  $SC_{(SU)}$ ) in the product, followed the formulae:

$$\text{WC (\%)} = \frac{M_{\theta} - M_o}{M_*} * 100 \quad (1)$$

$$\text{MC (\%)} = \frac{MW_{\theta} - MW_o}{M_*} * 100 \quad (2)$$

$$\text{SC}_{(SA, SU)} (\%) = \frac{MS_{\theta} - MS_o}{M_*} * 100 \quad (3)$$

where  $M_*$  represents the total mass of fresh mushrooms.  $M_o$  is the mass of the controls (they are either the mushrooms after blanching (type 1) or after blanching and sucrose treatment (type 2)).  $M_{\theta}$  is the mass of the mushrooms at the time of sampling.  $MW_o$  represent the amount of water in the controls and  $MW_{\theta}$  the amount of water at sampling. The  $MS_o$  represent the mass of the osmotic solutes in the controls;  $MS_{\theta}$  represent the amount of solute at sampling.

Although mass transfer in vegetable tissue can be quite complex, an unsteady state Fickian diffusion approach has been suggested by Hawkes and Flink (1978), Magee *et al.* (1983) and Biswal *et al.* (1991). They showed that mass transfer during osmotic dehydration can be represented by a linear relationship with the square root of contact time. While using the appropriate variables. The linearity of  $SC_{(SA \text{ or } SU)}$  and MC data verses the square root of time was tested and found to be applicable for both types of mushrooms. An example illustrating the linearity of moisture loss for type 1 mushrooms is shown in Fig. 3. Also Biswal *et al.* (1991) described the relationship between the pseudo mass transfer coefficient (K) and C (mass fraction of initial NaCl in the bath, %) and  $\Theta$  (time of contact, min) and t (temperature of contact, C). Using the previous concepts and satisfy the initial conditions that at  $\Theta = 0$ ,  $SC_{(SA \text{ or } SU)} = 0$  and  $MC = 0$ . The following equation was used for both solute and moisture changes:

$$\text{SC}_{(SA \text{ or } SU)} \text{ or } \text{MC} = \beta_o * C^{\beta_1} * \Theta^{0.5} \quad (4)$$

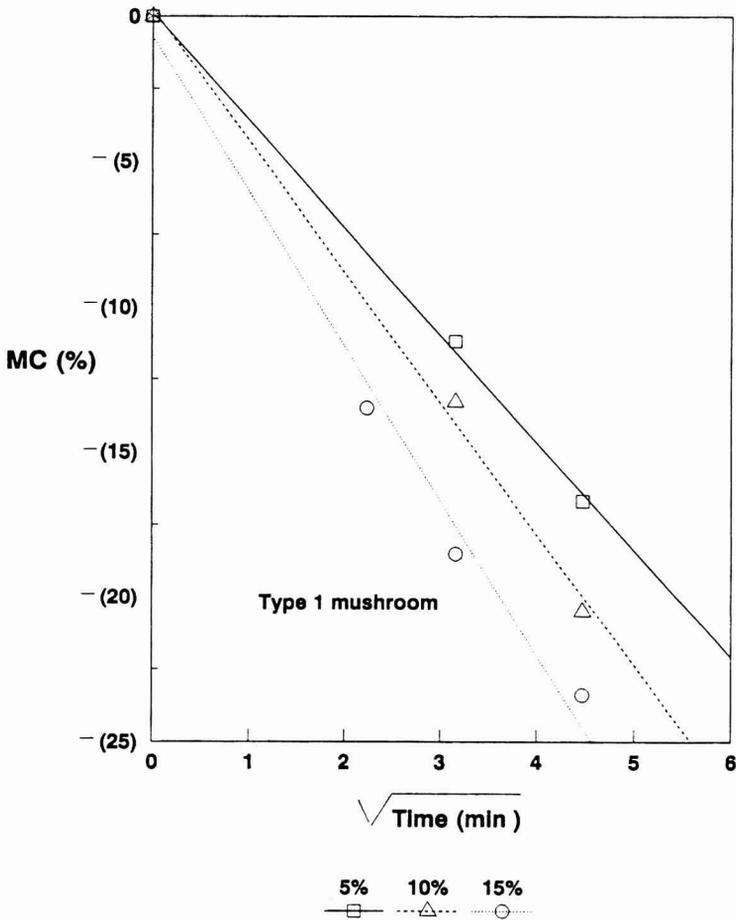


FIG. 3. MOISTURE CHANGES FOR TYPE 1 MUSHROOMS  
Origin of time is the time at which the mushrooms were introduced into the salt bath.

The nonlinear regression (PROC NIN) procedure through Statistical Analysis System (SAS 1988) was performed to find the best parameters for Eq. (4). The regression coefficients of the nonlinear model and their  $r^2$  for the fitting are listed in Table 1. As is shown, the predicted model fit the experimental values reasonably well. The values of rate change of the SC (sodium chloride, sucrose) and moisture (MC), are of great importance in analyzing the kinetics of mass transfer. These values can be obtained by taking the first derivative of Eq. (4). Rate ratio for salt or sucrose, defined as the rate of moisture change divided by rate of salt

TABLE 1.  
REGRESSION COEFFICIENTS AND R<sup>2</sup> OF EQ. (4)

| Mushroom | Coefficients        |                   | R <sup>2</sup> |
|----------|---------------------|-------------------|----------------|
|          | $\beta_0$           | $\beta_1$         |                |
| Type 1   |                     |                   |                |
| Salt     | 0.4258              | 0.4872            | 0.8426         |
| CI*      | (0.4013 - 0.4503)   | (0.4641 - 0.5103) |                |
| Moisture | -1.9021             | 0.3509            | 0.9792         |
| CI*      | (-1.9151 - -1.8891) | (0.3497 - 0.3521) |                |
| Type 2   |                     |                   |                |
| Salt     | 0.0975              | 0.9622            | 0.9555         |
| CI*      | (0.0982 - 0.0968)   | (0.9307 - 0.9937) |                |
| Sucrose  | -0.4582             | 0.2876            | 0.7765         |
| CI*      | (0.3670 - 0.5494)   | (0.2744 - 0.3008) |                |
| Moisture | 2.6041              | -0.2665           | 0.9430         |
| CI*      | (2.5224 - 2.6858)   | (0.2535 - 0.2795) |                |

Regression coefficients were obtained from SAS by fitting experimental data with replications.  
CI\* represent the Asymptotic 95% Confidence Interval.

or sugar change at the same time will be used to monitor the efficiency of water removal and salt loading.

## RESULTS AND DISCUSSION

### Mass Transfer During Osmosis

The results of the 19 different treatments were condensed on 5 contour plots (Fig. 4-6) generated using Eq. (4). It should be noted that origin of time in the figures is the time at which mushrooms were introduced into the salt bath. Contour plots present the changes in salt, sucrose and moisture from their value when they entered the salt bath. The salt, sucrose and moisture contents were 0, 0.2 and 90.5% for type 1 mushrooms and 0, 5.8 and 46.5% for type 2 mushrooms prior to entering the salt bath. During the sucrose treatment, type 2 mushrooms lost 44% of moisture (MC) and gained 5.6% of sucrose (SC). The actual magnitude of salt, sucrose and moisture content in the mushrooms after the salt bath treatment can be obtained directly by adding the mass changes from the contours (Fig. 4-6) to the initial mass.

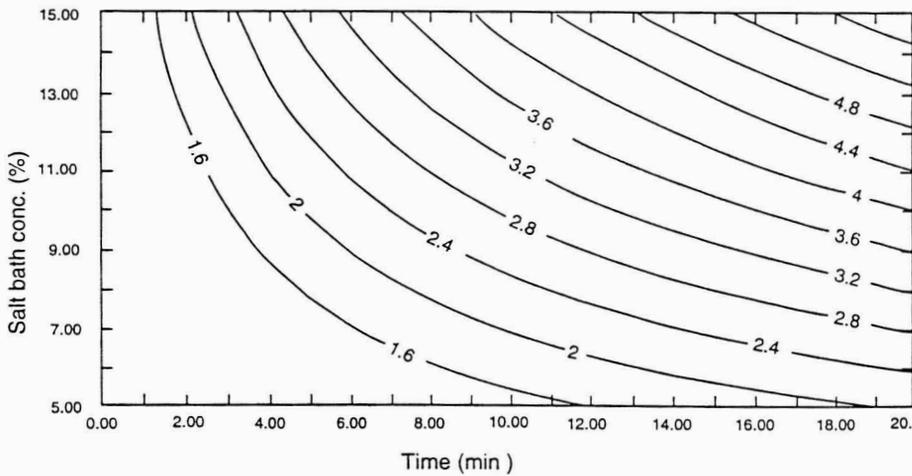
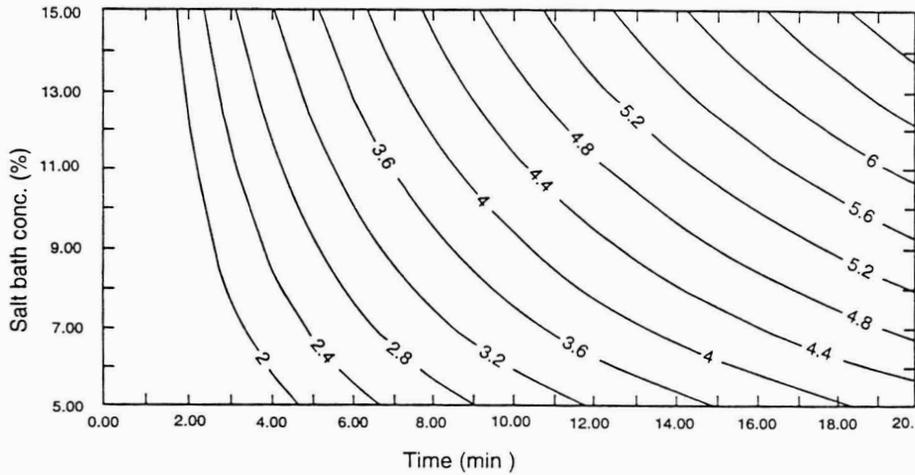


FIG. 4. CONTOURS OF SALT CHANGES (%) FOR TYPE 1 (TOP)

AND 2 (BOTTOM) MUSHROOMS

Origin of time for each type of mushrooms is the time at which the mushrooms were introduced into the salt bath.

### Kinetics of Mass Changes

The sodium chloride changes for type 1 and 2 mushrooms are presented in Fig. 4. It is apparent that the type 2 mushrooms achieve a lower salt residue com-

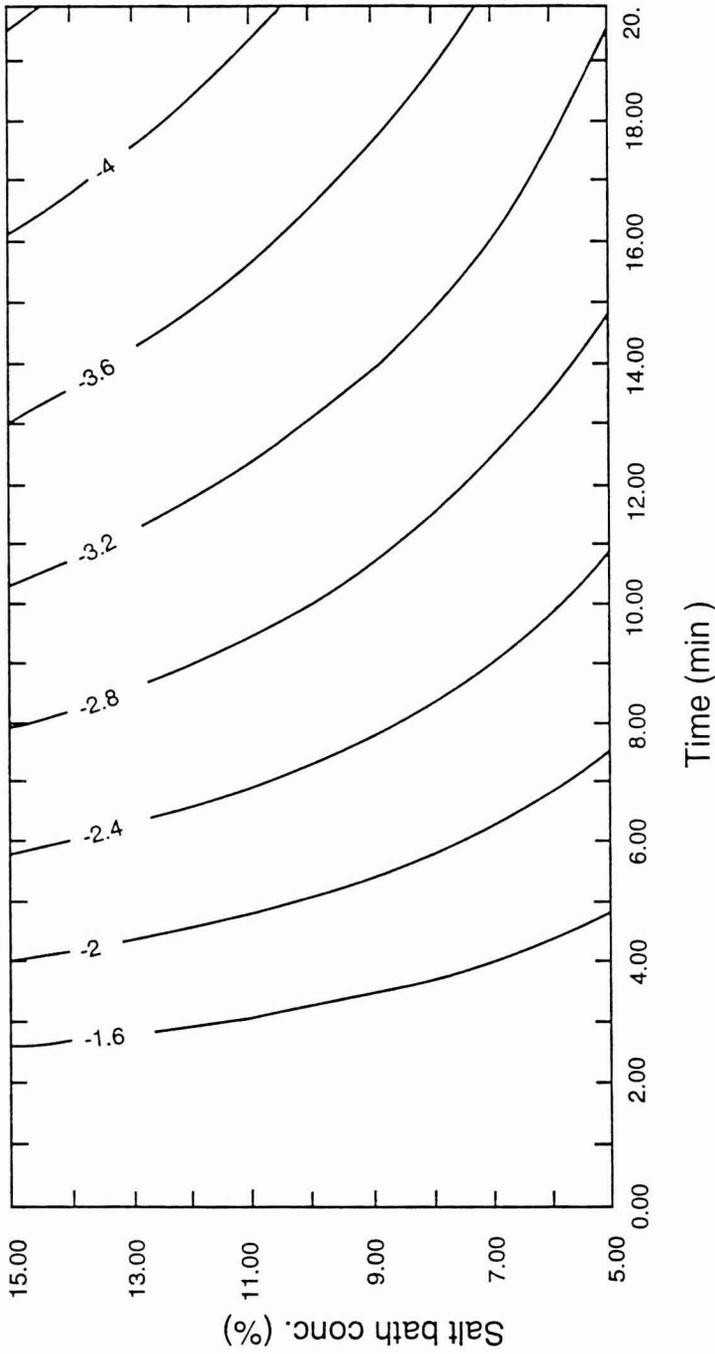


FIG. 5. CONTOURS OF SUCROSE CHANGES (%) IN TYPE 2 MUSHROOMS  
Origin of time for each type of mushroom is the time at which the mushrooms were introduced into the salt bath.

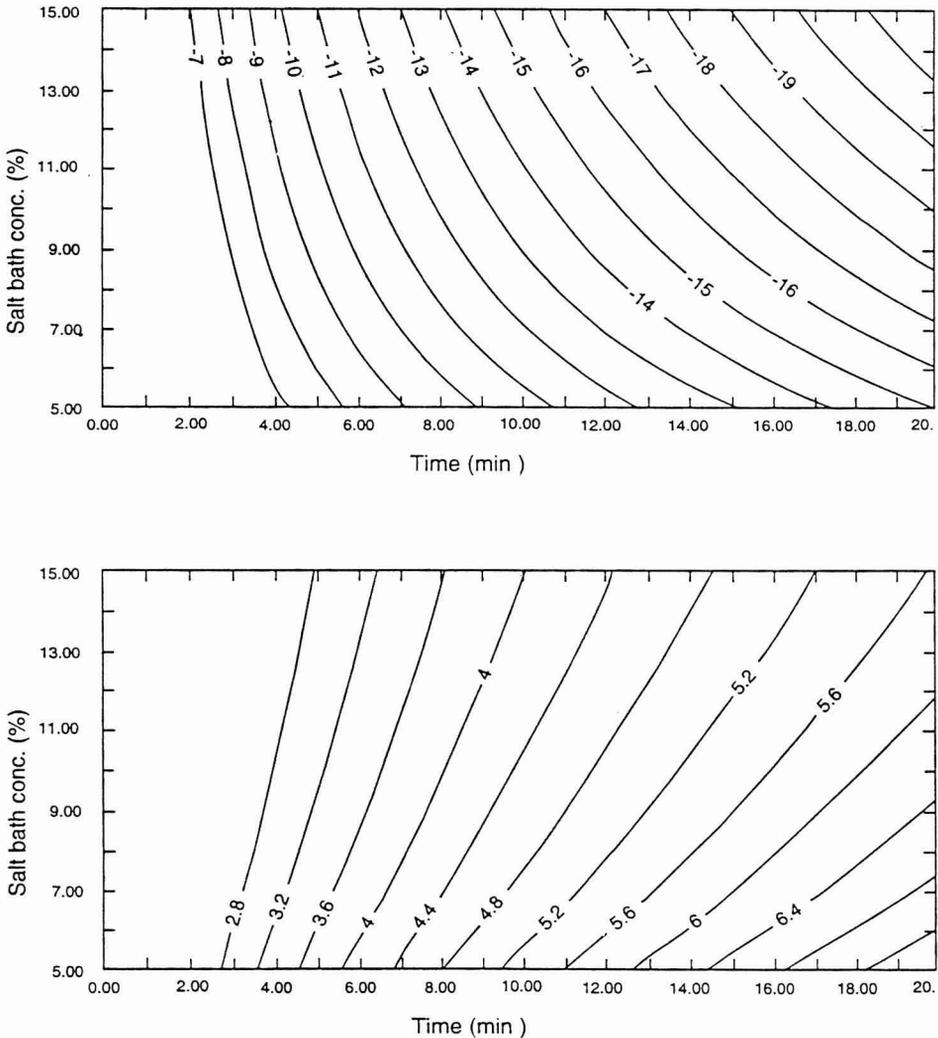


FIG. 6. CONTOURS OF MOISTURE CHANGES (%) FOR TYPE 1 (TOP) AND 2 (BOTTOM) MUSHROOMS

Time origin for each type of mushrooms is the time at which mushrooms were introduced into the salt bath.

pared to the type 1 processed at the same salt concentration for the same length of contact time. As expected, the higher the concentration of sodium chloride in the solution, the more sodium chloride penetrates into the mushroom. Conversely (Fig. 5), the sucrose content in the type 2 mushrooms, decreases during contact with the salt solution. Contour plots for the moisture changes in the two types of mushroom are shown in Fig. 6. As expected, the magnitude of water

loss in the type 1 mushroom is increasing with the salt concentration and the time of exposure. On the other hand, type 2 mushrooms, which have lost 44% of water during the sucrose treatment, regain a certain amount of water during the salt treatment. As shown the lower the concentration of the sodium chloride solution, the higher the amount of water regained at a given time. Notwithstanding this moisture regain the final type 2 mushrooms are still much lower in moisture than the equivalent type 1 (Fig. 6). The reason for this water gain is due to the fact that a 60% sucrose solution has a water activity of 0.894 which is almost the same as the one of a 15% sodium chloride solution (Table 2). Therefore, when exposed to a sodium chloride solution of less than 15% concentration the mushrooms would pick up water from the solution.

### Kinetics of Rate Change

From Eq. (4), it is possible to obtain values for the rates at which the transfer of water and solutes occurs as well as the direction in which they occur, and the results are presented in Fig. 7 and 8. A positive rate means a gain of mass and vice versa. It is clear that for the type 1 mushrooms the rate of water and salt movements are in opposite direction but as well significantly different. As one would expect the rate of water withdrawal increases as the concentration of salt in the outside solution increases but with a significant difference in their order of magnitude. This differential rate of movement of solute and water is one of the main features of osmotic dehydration which, properly controlled, will lead to desirable properties of the final product. As presented in Fig. 7, the rate of MC for type 2 mushrooms are all negative with value between 1.2 and 0.3 (%/min). Rates of salt change, as shown in Fig. 8, decrease with time. Data for the rate ratio of salt to water is presented in Fig. 8. Since the flow of moisture and salt are simultaneous during the process, knowledge of the rate ratio would assist one to recognize the relationship between rate of moisture and solute changes.

TABLE 2.  
OSMOTIC COEFFICIENT AND WATER ACTIVITY OF SUCROSE  
AND SODIUM CHLORIDE AT 25C

| Concentration   | Water Activity |
|-----------------|----------------|
| Sodium chloride |                |
| 5%              | 0.970          |
| 10%             | 0.935          |
| 15%             | 0.893          |
| Sucrose         |                |
| 60%             | 0.894          |

Source: Adapted from Robison and Stokes (1965)

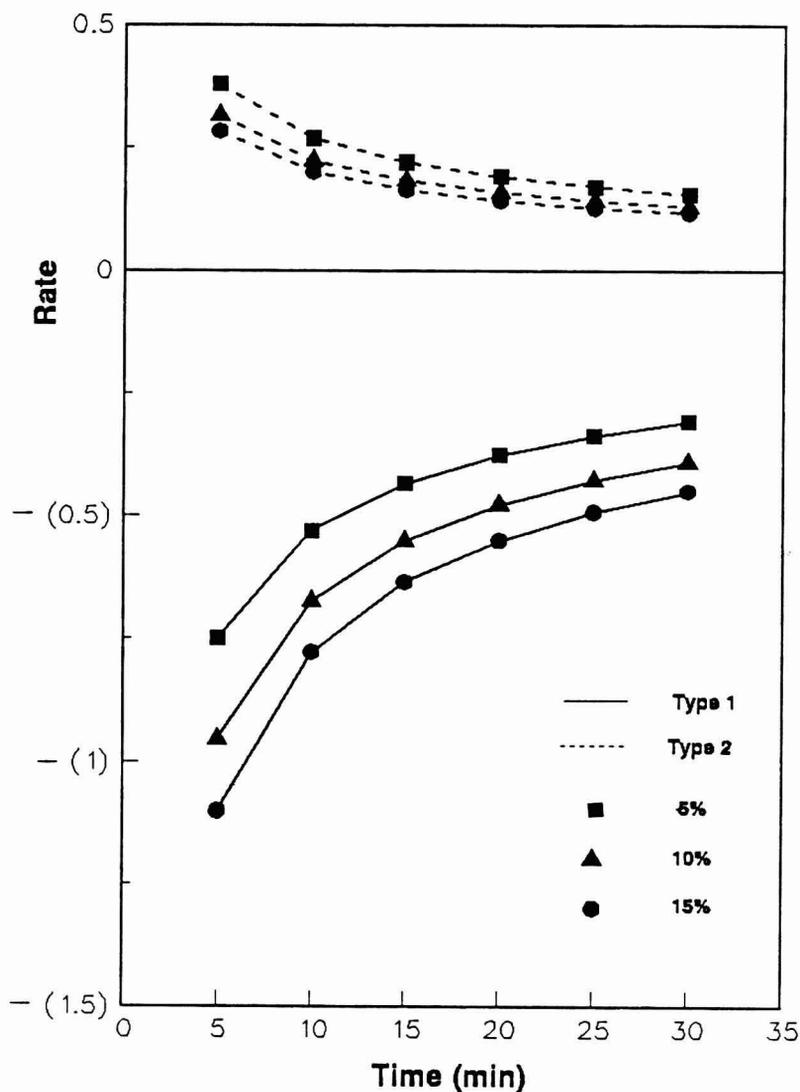


FIG. 7. RATE OF MOISTURE CHANGES (%/min) FOR TYPE 1 AND 2 MUSHROOMS

Time origin for each type of mushrooms is the time at which mushrooms were introduced into the salt bath.

Rate ratio for type 1 mushroom as shown maintained a value between  $-3.6$  and  $-3.1$ , which indicate the rate of moisture loss is at least 3 times faster than the salt gain. For comparison the rate ratio of sucrose during sucrose treatment has

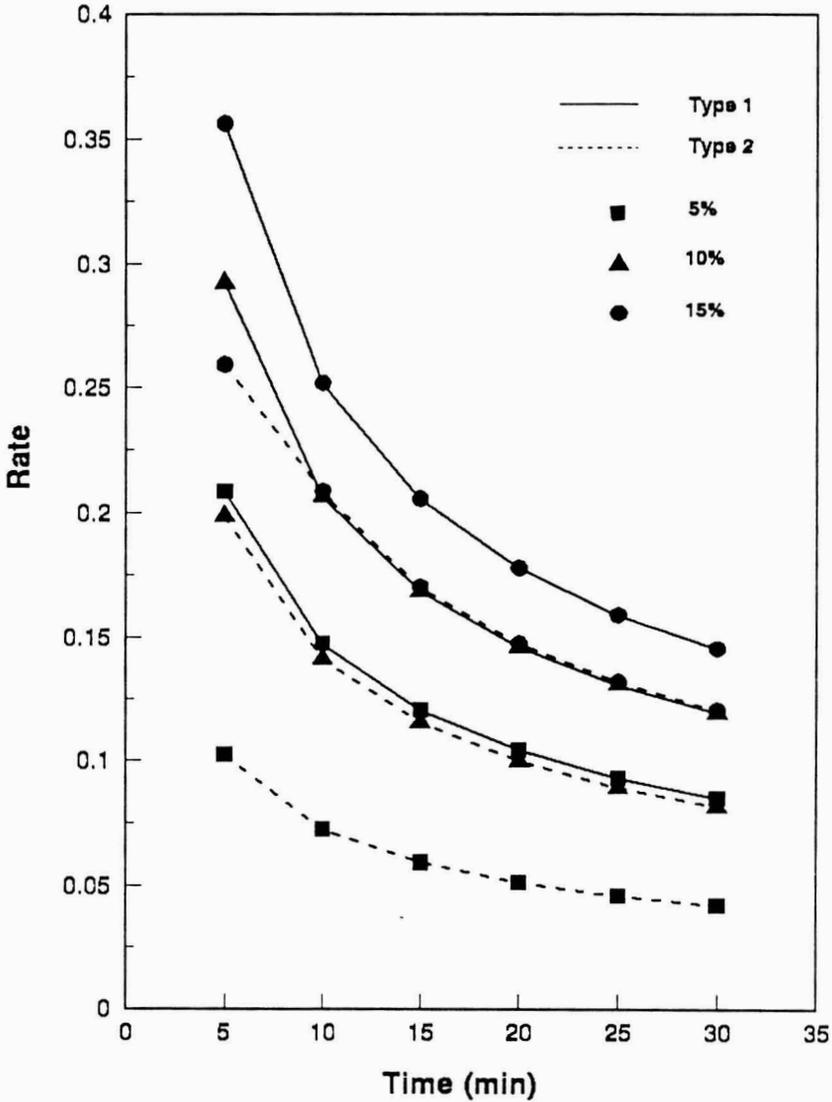


FIG. 8. RATE OF SALT CHANGES (%/min) FOR TYPE 1 AND 2 MUSHROOMS  
Time origin for each type of mushroom is the time at which mushrooms were introduced into the salt bath.

a calculated value of  $-7.3$ . Procedure for type 1 mushroom, as we have discussed above, is a combination of dehydration and salt loading, slightly in favor of dehydration. The magnitude of moisture loss will be restricted by the tolerance to the salt level determined by consumer acceptance. Type 2 mushrooms go through a two-stage procedure, during the first stage of sucrose treatment, dehydration predominates the entire process. During the second stage of salt treatment, sucrose is removed and salt added with a minimum of water changes. As shown in Fig. 9, since the rate ratio is around 1 for high salt concentration, this would create a high salt concentration at the surface of the material lowering the surface water activity effectively. The effect of sucrose treatment is clear, it provides a method of removing water effectively and assist in controlling the salt content in the final product.

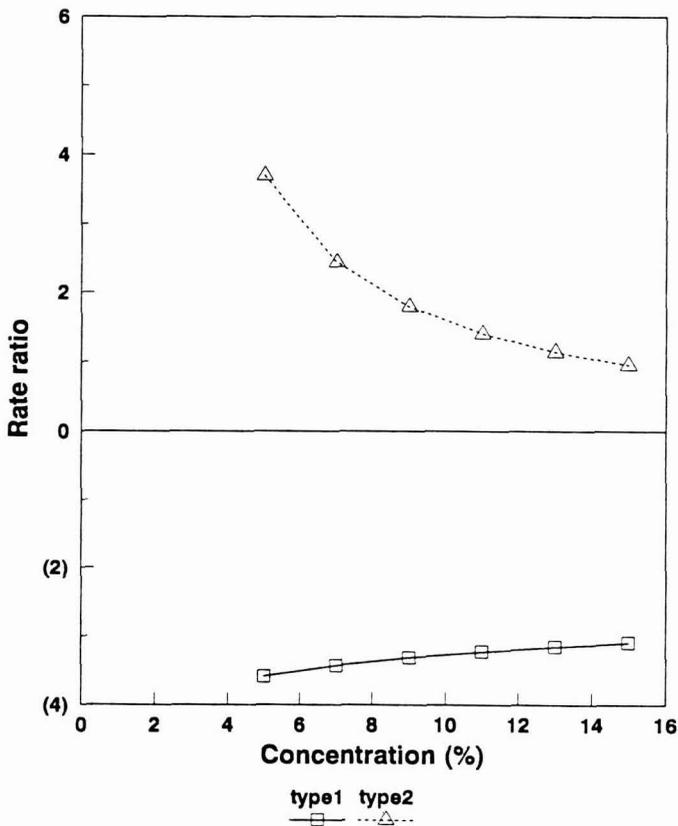


FIG. 9. RATE OF RATIO FOR TYPE 1 AND 2 MUSHROOMS  
Time origin for each type of mushroom is the time at which mushrooms were introduced into the salt bath.

## CONCLUSION

In this study, the changes in moisture and sodium chloride were shown to be linearly related to the square root of time. The mathematical relationships of the mass transfer kinetics were developed for the two types of processing methods. It was found that the model adequately explained the solutes exchanges during osmotic dehydration. The 15% sodium chloride solution was found to be the optimum for both types of processing conditions. Pretreatment of mushrooms in high concentration of sucrose solution followed by the treatment in the high salt concentration solution was found to be the most effective method to remove water and loading salt to further lower the water activity in the mushroom. Research continue to elevate other solutions with different solutes so as to understand more precisely the differential effect of water and solute movement.

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## GUIDE FOR AUTHORS

Typewritten manuscripts in triplicate should be submitted to the editorial office. The typing should be double-spaced throughout with one-inch margins on all sides.

Page one should contain: the title which should be concise and informative; the complete name(s) of the author(s); affiliation of the author(s); a running title of 40 characters or less; and the name and mail address to whom the correspondence should be sent.

Page two should contain an abstract of not more than 150 words. This abstract should be intelligible by itself.

The main text should begin on page three and will ordinarily have the following arrangement:

**Introduction:** This should be brief and state the reason for the work in relation to the field. It should indicate what new contribution is made by the work described.

**Materials and Methods:** Enough information should be provided to allow other investigators to repeat the work. Avoid repeating the details of procedures which have already been published elsewhere.

**Results:** The results should be presented as concisely as possible. Do not use tables *and* figures for presentation of the same data.

**Discussion:** The discussion section should be used for the interpretation of results. The results should not be repeated.

In some cases it might be desirable to combine results and discussion sections.

**References:** References should be given in the text by the surname of the authors and the year. *Et al.* should be used in the text when there are more than two authors. All authors should be given in the References section. In the Reference section the references should be listed alphabetically. See below for style to be used.

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HASSON, E.P. and LATIES, G.G. 1976. Separation and characterization of potato lipid acylhydrolases. *Plant Physiol.* 57, 142-147.

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Journal abbreviations should follow those used in *Chemical Abstracts*. Responsibility for the accuracy of citations rests entirely with the author(s). References to papers in press should indicate the name of the journal and should only be used for papers that have been accepted for publication. Submitted papers should be referred to by such terms as "unpublished observations" or "private communication." However, these last should be used only when absolutely necessary.

Tables should be numbered consecutively with Arabic numerals. Type tables neatly and correctly as they are considered art and are not typeset. The title of the table should appear as below:

TABLE 1.

### ACTIVITY OF POTATO ACYL-HYDROLASES ON NEUTRAL LIPIDS, GALACTOLIPIDS, AND PHOSPHOLIPIDS

Description of experimental work or explanation of symbols should go below the table proper.

Figures should be listed in order in the text using Arabic numbers. Figure legends should be typed on a separate page. Figures and tables should be intelligible without reference to the text. Authors should indicate where the tables and figures should be placed in the text. Photographs must be supplied as glossy black and white prints. Line diagrams should be drawn with black waterproof ink on white paper or board. The lettering should be of such a size that it is easily legible after reduction. Each diagram and photograph should be clearly labeled on the reverse side with the name(s) or author(s), and title of paper. When not obvious, each photograph and diagram should be labeled on the back to show the top of the photograph or diagram.

**Acknowledgments:** Acknowledgments should be listed on a separate page.

Short notes will be published where the information is deemed sufficiently important to warrant rapid publication. The format for short papers may be similar to that for regular papers but more concisely written. Short notes may be of a less general nature and written principally for specialists in the particular area with which the manuscript is dealing. Manuscripts which do not meet the requirement of importance and necessity for rapid publication will, after notification of the author(s), be treated as regular papers. Regular papers may be very short.

Standard nomenclature as used in the biochemical literature should be followed. Avoid laboratory jargon. If abbreviations or trade names are used, define the material or compound the first time that it is mentioned.

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