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CONTENTS

FOAMING AND EMULSIFYING CHARACTERISTICS OF FRACTIONATED WHEY PROTEIN

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ABSTRACT

The foaming and emulsifying characteristics of whey protein concentrates and of β-lactoglobulin and α-lactalbumin enriched fractions derived therefrom were measured. The spray dried WPCs and the α-lactalbumin enriched fraction derived from acid whey protein concentrate produced foams comparable to those produced from egg whites. The freeze dried WPCs and β-lactoglobulin enriched fractions exhibited little foaming ability. The WPC and β-lactoglobulin enriched fractions produced acceptable emulsions although they do not appear to have as good emulsifying properties as egg yolks do. The α-lactalbumin enriched fraction had poor emulsifying properties. These results indicate that fractionations of WPC into β-lactoglobulin and α-lactalbumin enriched fractions results in products with improved foaming and emulsifying properties.

INTRODUCTION

Foaming or whipping is an important functional property of proteins in foods such as angel food cakes, sponge cakes, divinity type confections, candy, meringues, souffles and whipped toppings. Foams are formed by the dispersion of a gas of liquid and the envelopment of the gas cells by surface active agents which cause entrapment of the gas in the liquid phase. The ease with which a protein forms is related to the ability of the protein to lower the interfacial tension at the

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gas-liquid interface. The protein does this by unfolding and aligning itself between the two phases. Coagulation of these unfolded proteins promotes foam stability by creating strong surface films at the solution-air interface. Two approaches can be used to form foams: (1) bubbling air through protein solutions; and (2) incorporating air into protein solutions by mechanical agitation. Whipping ability is determined by measuring such factors as foam capacity and foam stability. The foam capacity or overrun refers to the maximum increase in foam volume which can be obtained through whipping. Foam stability is a measure of the ability of the foam to maintain its maximum volume for a certain period of time.

Several researchers have studied the foaming abilities of whey protein concentrates (WPC)(deWit and deBoer 1975; Haggett 1976; Jelen 1973; McDonough et al. 1974; Morr et al. 1973; Morr 1979; Richert et al. 1974). The foaming ability of a WPC is affected by pH, calcium ion concentration, protein solubility (as influenced by fractionation, concentration and heat denaturation), residual lipids (as influenced by whey separation, addition of phosphates, surfactants) and the viscosity of the final formulation (as influenced by the concentrations of protein, sugar and other added ingredients). In general, a WPC with a high protein concentration of 25% (W/V), very little lipid content and minimum whey protein denaturation should be the most suitable for foaming applications. Foam stablity of WPC formulations is significantly better at high pH values which are often achieved by the addition of calcium hydroxide. The hydroxide ions increase the pH while the calcium ions facilitate the unfolding of the protein chains.

Proteins with good emulsifying properties have many applications in foods which contain both water and fat phases, e.g., minced meats and salad dressings. Emulsions are heterogeneous systems consisting of one or more immiscible phases intimately dispersed in a continuous phase. Emulsion stability can be improved by the addition of surface active agents and finely divided solids. These stabilizers are termed emulsifiers. They act by reducing the liquid-liquid interfacial tension enough to cause the dispersed phase to form smaller droplets and by forming a film at the interfacial boundary which prevents or retards coalescence of the droplets of the disperse phase (3). Factors which influence the conformation of the proteins (allowing them to unfold and to be adsorbed at the fat-water interface) improve the emulsifying abilities of the whey proteins (Hayes et al. 1979; Shimuzu 1981).

MATERIALS AND METHODS

The whey protein samples tested were derived from fresh water Cheddar cheese whey or cottage cheese whey which was ultrafiltered to 90% volume

reduction. Portions of these ultrafiltered cheese wheys were then fractionated into β-lactoglobulin and α-lactalbumin enriched fractions. This fractionation procedures consisted of demineralization via electrodialysis or diafiltration, pH adjustment of the demineralized WPC to 4.65 followed by centrifugation. Centrifugation resulted in a β-lactoglobulin enriched solid and an α-lactalbumin enriched supernatant. The β-lactoglobulin enriched solid was then further purified by resuspending or washing the solid in a solution buffered at pH 4.65 followed by recentrifugation to remove some of the lactose and ash. Lactose and ash were removed from the α-lactalbumin enriched supernatant via diafiltration with distilled water. The samples were then freeze dried or spray dried.

Table 1 summarizes the compositions of the samples tested. Details of the procedures used to prepare the enriched fractions have been discussed by Slack *et al.* (1986a).

Table 1. Sample compositions (dry basis)

No.	Sample	% Protein	% Fat	% Ash	% Residual*
1	Spray Dried Acid Whey Protein Concentrate	27.92	1.95	7.94	62.19
2	Spray Dried Sweet Whey Protein Concentrate	34.63	6.04	6.03	53.30
3	Freeze Dried Acid Whey Protein Concentrate	33.94	2.99	5.93	57.14
4	Freeze Dried Sweet Whey Protein Concentrate	38.91	9.16	5.22	46.71
5	ß-Lactoglobulin Source: Electrodialyzed AWPC	67.34	4.45	1.38	26.83
6	ß-Lactoglobulin Source: Diafiltered AWPC Sp. D. SWPC	75.31	11.44	2.05	11.02
7	ß-lactoglobulin Source: Electrodialyzed SWPC	37.98	35.93	0.95	25.14
8	ß-lactoglobulin Source: Diafiltered SWPC	51.96	26.28	2.41	19.35
9	α-lactalbumin Source: Electrodialyzed AWPC	27.79	1.38	0.51	70.32
10	α-lactalbumin Source: Diafiltered AWPC	52.36	2.05	2.68	42.91
11	α-lactalbumin Source: Electrodialyzed SWPC	39.00	0.57	1.42	59.01
12	α-lactalbumin Source: Diafiltered SWPC	64.49	2.41	1.34	31.76
13	Freeze Dried Egg Yolks	31.70	63.80	3.58	0.92
14	Freeze Dried Egg Whites	68.80	1.90	5.04	24.26

AWPC = Acid Whey Protein Concentrate
SWPC = Sweet Whey Protein Concentrate

The foaming characteristics of the samples were determined by measuring the volume of a suspension of the sample after whipping. The suspensions were made up at concentrations of 10.0% (W/V) on a dry weight basis in distilled water. The pH of each suspension was then adjusted to 9.0 with $Ca(OH)_2$. Fifty ml samples were whipped in a Sun Beam Mix Master 5 speed blender at high speed for 10 min. After whipping, the slurries were poured into a 500 ml graduated cylinder and the total volume was measured. The percent overrun was expressed as the amount of volume increase after whipping divided by the original sample volume, multiplied by 100.

% overrun $= 100 [(V_F - V_S)/V_S]$
where V_F = volume of foam
V_S = original volume of sample

The foam stability was determined by transferring the whipped sample to a funnel and allowing it to drain into a graduated cylinder. The amount of liquid in the cylinder was measured immediately and after standing for 15, 30, 45 and 60 min. For these times the percent drainage was expressed as:

% Drainage $= 100 [V_D/V_S]$
where V_D = volume of liquid drained

The foam capacity and foam stability tests were each performed in duplicate.

The emulsifying capacities of the samples were determined by finding the volume of oil needed to collapse an oil and water emulsion made from a suspension of the sample and Mazola corn oil. The sample suspensions were made up in distilled water to contain 1.0% solids (W/V) on a dry weight basis. The pH of each suspension was then adjusted to 7.0 with HCl or NaOH. A mixture of 50 ml of the sample suspension and 25 ml of Mazola corn oil was blended for 30 s in a 2 speed Osterizer blender at high speed. If the emulsion remained intact after 30 s of mixing, oil was added at a rate of 5 ml/10 s until the emulsion collapsed, as measured by the change in conductance registered by a microammeter. The emulsion capacity was expressed as the volume of oil emulsified per gram of sample. All tests were performed in duplicate.

The emulsion stabilities of the samples were determined by measuring the volume of liquid that separated from an emulsion made from suspensions of the samples and Mazola corn oil. Suspensions of these samples were made up as described above for the emulsion capacity studies. The results of the emulsion capacity tests indicated how much oil was needed to break each emulsion. Hence, 90% of this quantity of oil was added to 50 ml of the sample suspension at a rate of 5 ml per 10 s in an Osterizer blender (blending at high speed). The emulsion was then transferred to transparent 50 ml graduated centrifuge tubes. The volume of the emulsion and the volume of the separated phase were recorded immediately and after standing for 0.5, 1.0, 3.0 and 24 h at room temperature. The emulsion stability was expressed at the volume of emulsified

layer after standing, divided by the original emulsion volume. The emulsion stability was calculated for each emulsion after standing 0.5, 1.0, 3.0 and 24 h. All tests were performed in duplicate.

RESULTS AND DISCUSSION

Studies of Foaming Characteristics

Table 2 summarizes the foam capacities and foam stabilities measured for foams formed from solutions of whey protein concentrates (WPC),β-lacto-globulin-enriched samples and α-lactalbumin-enriched samples at concentrations of 10% (W/V) and at pH 9.0. Error analysis of the method gives an error limit estimate of 100% for overrun values and an error limit estimate of 5% for the drainage values. As the data indicate, the spray dried whey protein concentrate samples (#1 and #2) and theα-lactalbumin enriched samples (#9 and #10) derived from acid whey formed foams with the greatest overruns with an average of 784% overrun (std. dev. = 236%). These foams retained some stability over a 1 h period. These foams were also comparable to foams made from a sample of freeze dried egg whites sample (sample #14) which had an average overrun of 633% and which also retained some stability over a 1 h period. The remaining samples exhibited little foaming ability (average overrun = 74% std. dev. 95%) and no stability over time.

The source of the whey, i.e. cottage cheese or Cheddar cheese did not appear to influence the foaming ability of WPC samples or theβ-lactoglobulin enriched samples in a consistent manner. In contrast, theα-lactalbumin enriched samples derived from cottage cheese whey (acid whey) exhibited good foaming properties whereas theα-lactalbumin enriched samples derived from Cheddar cheese whey (sweet whey) exhibited very poor foaming properties. Theα-lactalbumin enriched samples derived from both types of whey were fractionated under identical conditions and their compositions are not radically different from one another; hence there is no readily apparent reason why theα-lactalbumin enriched samples derived from sweet whey did not foam. The method of demineralization (electrodialysis or diafiltration) also did not appear to affect the foaming properties of the enriched samples.

The WPC samples which were freeze dried (#3 and #4) had little foaming ability where as the WPC samples (#1 and #2) which were spray dried had foaming abilities comparable to those of the freeze dried egg whites. Marked improvement of the foaming properties of WPC by heat treatment is a patented procedure which results in reduced whipping time, increased overrun, and longer foam stability (Jelen 1973). This suggests that heat treatment of the α-lactalbumin enriched samples may further improve their foaming properties.

Table 2. Foaming and emulsifying properties

No.	Sample	% Overrun	% Drainage after 1 hr	Emulsion capacity ml oil/g protein	Emulsion stability after 24 hr ml solution/ml emulsion
1	Spray Dried Acid Whey Protein Concentrate	661	88	250	0.50
2	Spray Dried Sweet Whey Protein Concentrate	1026	94	270	0.44
3	Freeze Dried Acid Whey Concentrate	240	100	310	0.52
4	Freeze Dried Sweet Whey Protein Concentrates	0	100	255	0.33
5	ß-lactoglobulin Source: Electrodialyzed AWPC	73	100	165	0.34
6	ß-lactoglobulin Source: Diafiltered AWPC	53	100	135	0.38
7	ß-lactoglobulin Source: Electrodialyzed SWPC	23	100	250	0.45
8	ß-lactoglobulin Source: Diafiltered SWPC	0	100	190	0.45
9	α-lactalbumin Source: Electrodialyzed AWPC	933	80	180	0
10	α-lactalbumin Source: Diafiltered AWPC	517	90	95	0
11	α-lactalbumin Source: Electrodialyzed SWPC	0	100	130	0
12	α-lactalbumin Source: Diafiltered SWPC	203	100	75	0
13	Freeze Dried Egg Yolks	0	100	395	0.45
14	Freeze Dried Egg Whites	633	70	100	0.76

These results indicate that the α-lactalbumin enriched samples derived from acid whey protein concentrate are very good foaming agents and in this respect are comparable to dehydrated egg whites. The β-lactoglobulin-enriched samples proved to be ineffective foaming agents. When these two proteins are combined as in WPC's, the mixture in solution forms unstable foams the instability being attributed to the presence of the β-lactoglobulin proteins. However, spray drying markedly improved the foaming abilities of the WPC's. Haggett (1976) has theorized that the foaming improvement due to the heating of the WPC during spray drying may result from dissociation of β-lactoglobulin dimers. Mild denaturation of both types of proteins during heat treatment may also help the

proteins to unfold and align themselves at gas-liquid interfaces thereby enhanc-
ing foam formation. Some form of heat treatment may further improve the foam-
ing properties of the α-lactalbumin enriched samples making them good
substitutes for egg whites in foams.

Studies of Emulsifying Characteristics

Table 2 summarizes the emulsifying capacities and emulsifying stabilities of
oil in water emulsions stabilized with whey protein concentrates (WPC),
β-lactoglobulin-enriched samples and α-lactalbumin enriched samples. The
WPC samples and theβ-lactoglobulin-enriched samples had comparable emulsi-
fying properties. The WPC's formed emulsions which held an average of 271 ml
oil/g protein (S=27) and had an average emulsion stability of 0.45 ml solu-
tion/ml emulsion (S=0.09) after 24 h. Emulsions made with β-lactoglobulin
enriched fractions had an average emulsion capacity of 185 ml oil/g protein
(S=49) and an average emulsion stability after 24 h of 0.40 ml solution/ml
emulsions (S=0.06). Emulsions made with α-lactalbumin fractions had an
average emulsion capacity of 120 ml oil/g protein (S=46) but had no stability 30
min after formulation.

The similarities in the emulsifying capacities of the WPC's and β-lacto-
globulin enriched fractions indicate that the origin and the processing history of
the whey had little effect on the abilities of these samples to emulsify oil in
water. The tabulated values suggest that the WPC's are better emulsifiers than
the β-lactoglobulin enriched fractions. However, neither group could match the
emulsifying capacity of 395 ml oil/g protein measured for the freeze dried egg
yolks.

These results imply that the β-lactoglobulin enriched samples make adequate
protein emulsifying agents. However, these tests also indicate that they are not
superior to egg yolk in terms of ability to emulsify or stabilize large amounts of
oil in water. The lack of improvement in the emulsifying abilities of the β-lacto-
globulin enriched samples relative to the WPC samples may be due to a failure to
provide solution conditions which would allow the β-lactoglobulin proteins to
totally unfold and align themselves at the oil-water interface. Although the pro-
tein solutions were adjusted to pH 7.0, where the β-lactoglobulin samples prov-
ed to be very soluble, these conditions may not be conductive to unfolding to the
β-lactoglobulin molecules. If the globular nature of theβ-lactoglobulin proteins
still predominates at pH 7.0, their emulsifying abilities may be inhibited.

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RICE BRAN STABILIZATION BY DIELECTRIC HEATING

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ABSTRACT

Rice bran is an important by-product of rice milling as it contains about 15 to 25% oil. The rapid breakdown of the oil into free fatty acid (FFA) begins immediately after milling, rendering the oil nonedible by the action of a very active lipase enzyme present in it. To inactivate the enzymes responsible for the deterioration of the oil, rice bran samples were exposed to dielectric-heating for different durations. These samples were stored in sealed polyethylene bags for a period of six weeks. The increase in FFA in oil during the storage period of six weeks was found to be only 2% in the case of the samples exposed for 6 to 7 min, while for the untreated samples the FFA rise was observed to be 75% for the same storage period.

INTRODUCTION

Rice bran is a valuable by-product of rice milling due to its high industrial and nutritive potential. The most important utilization of rice bran is the production of oil, since rice bran when relatively free from hulls, contains about 15 to 25% oil. Because of its low linolenic acid content and its capacity to lower serum cholesterol level, rice bran oil is considered to be a high class edible oil. Owing to the acute shortage for edible oils being experienced in many countries, it is most desirable to exploit the possibility of extracting edible grade oil from rice bran.

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The main reason for the underutilization of rice bran oil for edible purpose is the time lag between the production of bran and its extraction. If the oil is not extracted from the rice bran immediately after its removal from rice, the oil contained in the bran is hydrolyzed into free fatty acid (FFA) and glycerol by the action of a very active lipase enzyme present in it. The rate of FFA release may be very high, sometimes as much as 5 to 7%. Splitting can occur in a single day and about 70% in a month under favorable conditions (Desikachar 1977). Spoilage may also occur as a result of oxidation leading to various odoriferous compounds including aldehydes, ketones, etc. Crude bran oil with FFA content of more than 10% is not generally suitable for edible oil production and hence such oil is used for manufacture of soaps and other industrial purposes (Enochian *et al.* 1981). In practice, however, the quick collection and extraction of oil from bran within a few hours of its production is not possible. This difficulty can be overcome by stabilization of rice bran, which is the process of inactivating the lipolytic enzymes in freshly milled bran so that the increase of FFA in rice bran oil is checked and the oil can be used for edible purposes after extraction.

Out of the different methods of stabilization, heat treatment either a wet or dry heat process, for inactivating the enzymes holds promise as a practical method and has been tried extensively at different places. Besides inactivating the enzymes, the heat treatment can simultaneously kill bacteria, molds and insect eggs which causes further spoilage.

The continuous type wet heat method of stabilization developed by Yokochi (1977) consisted of three stages: (1) steaming bran with direct steam injection (2) drying using steam jacket, and (3) cooling using forced cold air. In the batch type dry heat method reported by Pillaiyar *et al.* (1978), the rice bran contained in a cylindrical rotating vessel was heated at about 105 °C using hot air and held at this temperature for about 5 min. However, heating for a period of 50 min was required to bring the temperature of about 100 kg of bran to 105 °C. In both these methods, the treated rice bran could be stored safely for a period of four months keeping the FFA content within the 10% level.

However, inadequate stabilization under the wet heat treatment method may occur since the bran is so fine that steam penetration is difficult, and the bran clumps when dry centers form when agitation is inadequate (Barber and Benedito de Barber 1980). Besides, the most severe restriction on the use of wet heat stabilization is the requirement for a steam boiler. Most rice mills do not presently have equipment to generate steam. The capital required for installing a boiler, associated plumbing and satisfactory water supply would be high. The problems associated with the dry heat method are prolonged heating time and the difficulty of achieving uniform temperature with a material like rice bran which has a low thermal conductivity. Besides, high temperatures for long periods of heating can reduce the nutritional value of the bran proteins and vitamins (Barber and Benedito de Barber 1980).

In view of the drawbacks associated with the existing processes, one of the possible methods for rapid and most uniform heating of rice bran is the utilization of radio frequency (RF) dielectric heating. The dielectric heating has the unique advantage of generating heat internally in insulating materials which are not readily heated by other means. Therefore the objective of the present investigation was to study the effect of radio-frequency dielectric heating for inactivating the enzymes responsible for the deterioration of rice bran oil.

MATERIALS AND METHODS

A 1.5 KW capacity dielectric heater operating at 13.56 MHz was used. Two electrodes of size 40x30 cm were connected to the dielectric heater. The bottom electrode was made from 20 gauge aluminum sheet, while the top electrode was made from 20 gauge aluminum wire mesh, which facilitated the escape of moisture from the sample through the top electrode while heating.

A sample holder of size 14.4x14.4x1.5 cm was fabricated out of 3 mm thick teflon sheet. Teflon was used since it has a very low dielectric loss factor of 0.00008 (Mark 1976) and hence absorbs practically no energy from the radio frequency electric field.

The top electrode of the dielectric heater was placed above the sample holder on teflon spacers, leaving an air gap of 0.5 cm above the surface of the sample. The average electric field intensity applied was 0.5 KV/cm which was calculated using the following equation (Pour-El *et al.* 1981):

$$E = V/[d_1 + d_2(\epsilon_1/\epsilon_2) + d_3(\epsilon_1/\epsilon_3)] \text{ KV/cm} \ldots \ldots (1)$$

where E = electric field intensity, KV/cm
$\quad\quad V$ = RF electrode voltage, KV
$\quad\quad d_1$ = depth of bran sample in the sample holder, cm
$\quad\quad d_2$ = thickness of sample holder made of teflon, measured perpendicular to the plane of the electrodes, cm
$\quad\quad d_3$ = depth of the air space between the top level of the sample and the upper electrode, cm
$\quad\quad \epsilon_1, \epsilon_2 \& \epsilon_3$ = dielectric constants of rice bran, teflon and air, respectively.

All the experiments were conducted with the bran milled from Japonica x Indica paddy variety. Rice bran was obtained by polishing the brown rice in an abrasive type polisher. The degree of polish given was about 5%. The bran obtained from the mill was sieved manually using a B.S. sieve No. 25, to remove all the rice husk and brokens contained in it. The average particle size of the bran was 0.0269 cm.

Since no standard method for the determination of moisture content of the rice bran was available, the standard method specified by AOAC (1970) for the determination of moisture content of soybean flour was followed. This method involved drying of about 5 g bran sample in the moisture box (provided with cover) for 2 h at 130 ± 3 °C. The initial moisture content of the bran immediately after milling was determined to be 10.93% (w.b.).

The sample holder was filled with 92 g bran in all the experiments. The sample was placed in between the electrodes of the dielectric heater leaving an air gap of 0.5 cm between the top surface of the sample and the top electrode. The sample was subjected to the dielectric heating treatment for various exposure times of 1,2,3,4,5,6 and 7 min by setting the process timer accordingly. It may be noted that no metallic object can be introduced for the measurement of the temperature of the sample kept in between the electrodes while the dielectric heater is in operation. Therefore at the end of heating, the sample was removed immediately and its temperature was measured using a Yellow springs Instrument (USA) Model 73-A temperature indicator kept near the dielectric heater. Since there was some time lag between the end of the heating and the temperature measurement, it was necessary to utilize an extrapolation technique (Nelson and Whitney 1960) to know the actual temperature attained by the sample at the end of the heating period. The elapsed time between the end of the treatment and the first temperature measurement was noted with a stop watch. Then for each degree drop of the samples temperature, the corresponding time was noted. A temperature-time curve was drawn and the actual temperature attained by the sample at the end of the treatment was obtained by exptrapolation as shown in Fig. 1.
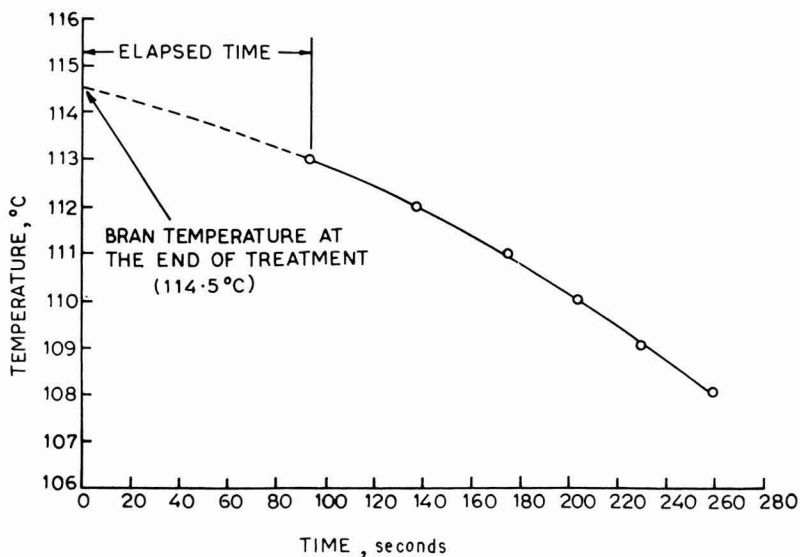
FIG. 1. A TYPICAL TEMPERATURE-TIME CURVE SHOWING ELAPSED TIME, BRAN
TEMPERATURE AND METHOD OF EXTRAPOLATION

At the end of each treatment, the final moisture content of the sample also was determined.

It is known that the bran has to be heated to above 100 °C for the inactivation of the enzymes (Loeb et al. 1949; Viraktamath and Desikachar 1971; Pillaiyar et al. 1978). Since it was found necessary to expose the bran for more than 4 min to obtain the temperature above 100 °C, the samples exposed for 4,5,6 and 7 min only were analyzed to study the efficacy of the treatment.

In order to find out whether there was any discoloration of the sample due to the treatment, the green tristimulus reflectance readings of the treated as well as untreated samples were measured using a Photovolt Company (USA) Model-670 reflection meter. About 250 g each of the treated and untreated samples were stored in sealed polyethylene bags at ambient conditions varying from 30 to 37 °C temperature and 50 to 85% relative humidity. Bran samples were taken periodically for a storage period of six weeks from each of the stored samples and analyzed for FFA content in oil. The extraction of oil from the bran was carried out using a water-bath shaker, in that 2 g of bran samples were taken in 50 ml conical flasks and extracted for 2 h with 25 ml n-hexane solvent at 60–65 °C. The solvent-oil solution was decanted through Whatman No. 1 filter paper into 250 ml flask. The excess solvent was then removed in a rotary thin-film evaporator. About 2 to 3 ml of chloroform was added to the flask and the oil present in the flask was dissolved in the chloroform. The oil-chloroform solution was then transferred into a 10 ml volumetric flask. Three to four washings with chloroform were necessary for removing all the oil contained in the flask.

In order to find out the FFA content in oil, 2 ml of oil-chloroform solution was pipetted into a clean alcohol rinsed 50 ml conical flask and diluted with 2 ml of pure chloroform. This mixture was titrated against 0.01N methanolic sodium hydroxide solution using 0.1% phenolphthalein as indicator. A blank titration with 2 ml of pure chloroform was also carried out. While titrating the oil-chloroform solution obtained from untreated samples, 0.1N methanolic sodium hydroxide solution was used, since the FFA content in the untreated sample was quite high as compared to the treated samples. FFA content in oil was expressed as percentage of oleic acid by using the following formula:

FFA (percent) = 100 VNF/1000 W
Where   V = volume of NaOH consumed, ml
            N = normality of NaOH solution
            F = equivalent weight of oleic acid (282)
            W = weight of oil contained in 2 ml oil-chloroform solution
            'W' was obtained by evaporating the solvent (chloroform) contained in 2 ml of oil-chloroform solution at 50 to 60 °C in an oven

RESULTS AND DISCUSSION

The final temperature and moisture content of fresh bran subjected to dielectric-heating treatment for different exposure periods of 1 to 7 min are presented in Fig. 2. The moisture contents reported are the mean of three replications. The standard error for the means in the measurements was found to be 0.074%. As the exposure time was increased, the final temperature increased resulting in the reduction of moisture content and the variations were found to be nonlinear.
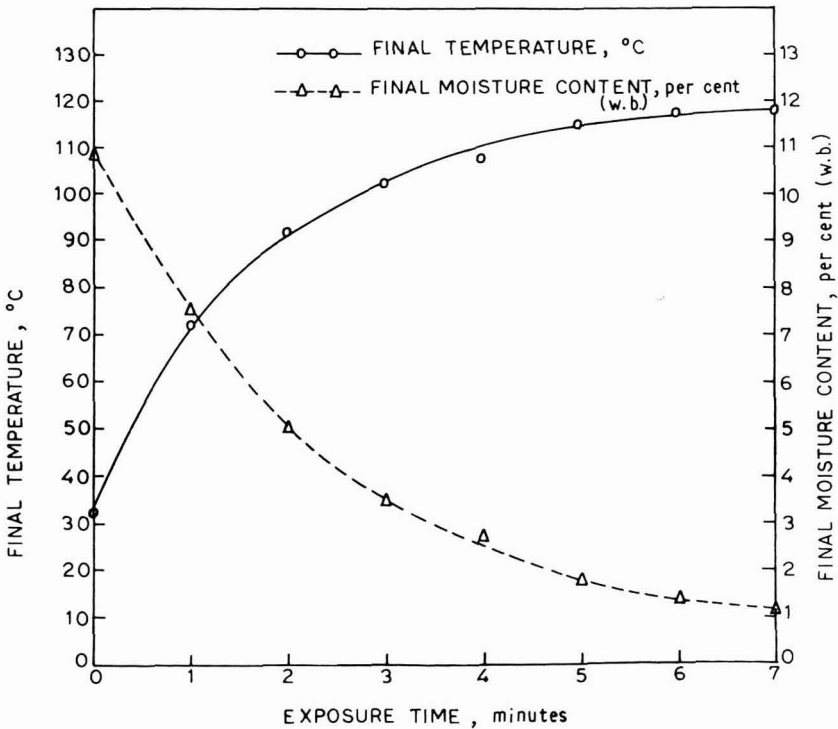
FIG. 2. EFFECT OF EXPOSURE TIME ON FINAL TEMPERATURE AND MOIS-
TURE OF RICE BRAN SUBJECTED TO DIELECTRIC HEATING

The absorption of energy from a RF electric field in a dielectric material is described (Nelson *et al.* 1966) by the following relationship:

$$P = KE^2\epsilon''f \dots\dots\dots\dots\dots\dots(2)$$

where K is 0.556 when P is the power absorbed in watt/cm^3, f is the frequency in megacylces, E is the electric field intensity in KV/cm as described by Equation (1) and ϵ'' is the dielectric loss factor of the material being exposed.

Equation (2) reveals that for a particular dielectric heating set-up the absorption of energy is dependent not only on the dielectric loss factor, but also on the dielectric constant of the material, since it influences the value of the field intensity.

It has been reported that the following second order models adequately describe the relationships between the dielectric properties of rice bran at 13.56 MH_z and its independent variables namely, moisture content, temperature and bulk density (Sreenarayanan 1983).

$$\epsilon' = 1.11 - 0.125M - 1.00 \times 10^{-3} T + 1.60 \times 10^{-3} \, \rho_b + 5.68 \times 10^{-3}M^2$$
$$+1.02 \times 10^{-3}M.T. + 2.40 \times 10^{-4}M.\rho_b \tag{3}$$
$$\epsilon'' = -0.279 - 7.3\times10^{-3}M + 2.01 \times 10^{-3}T + 7.47 \times 10^{-4}\rho_b$$
$$+ 2.03 \times 10^{-3}M^2 \tag{4}$$

Where ϵ' = dielectric constant
  ϵ'' = dielectric loss factor
  M = moisture content, percent (w.b.)
  T = temperature, °C
  ρ_b = bulk density, kg/m³

Further it may be noted that Eq. (3) and (4) hold good for the values of variables, namely moisture content of bran ranging from 4 to 14% (w.b.), temperature from 26.5 to 53.5 °C and bulk density from 300 to 400 Kg/m³. Examination of the above equations reveal that the dielectric constant and dielectric loss factor have positive nonlinear correlation with the variables within the ranges of the values of the variables mentioned above. Therefore, it is clear that for a particular dielectric heating set-up, the heating rate is mainly influenced by the amount of moisture available in the bran.

The green tristimulus reflectance readings of the samples are given in Table 1. These readings are the mean of three replicated measurements. The standard error found in the means was 0.07. It can be observed from Table 1 that the sample was slightly discolored due to the treatment and magnitude of discoloration was relatively more for longer duration treatments.

Table 1. Green tristimulus reflectance readings of bran exposed to dielectric heating

Exposure time minutes	4	5	6	7	Untreated sample
Green tristimulus reflectance reading	41.0	37.5	37.0	36.5	44

However, the extent of discoloration observed in the present study for the various exposure periods was quite negligible and hence the oil extracted from the treated bran was not analyzed to see the effect of RF heating on its discoloration. It may be pointed out that the discoloration of the stabilized bran is characteristic of any heat treatment. Barber and Benedito de Barber (1977) had also reported that the bran stabilized by the conventional heat treatment was darker as compared to the untreated bran.

The effect of dielectric heating for different exposure periods on FFA content of rice bran during storage in sealed polyethylene bags is given in Fig. 3.
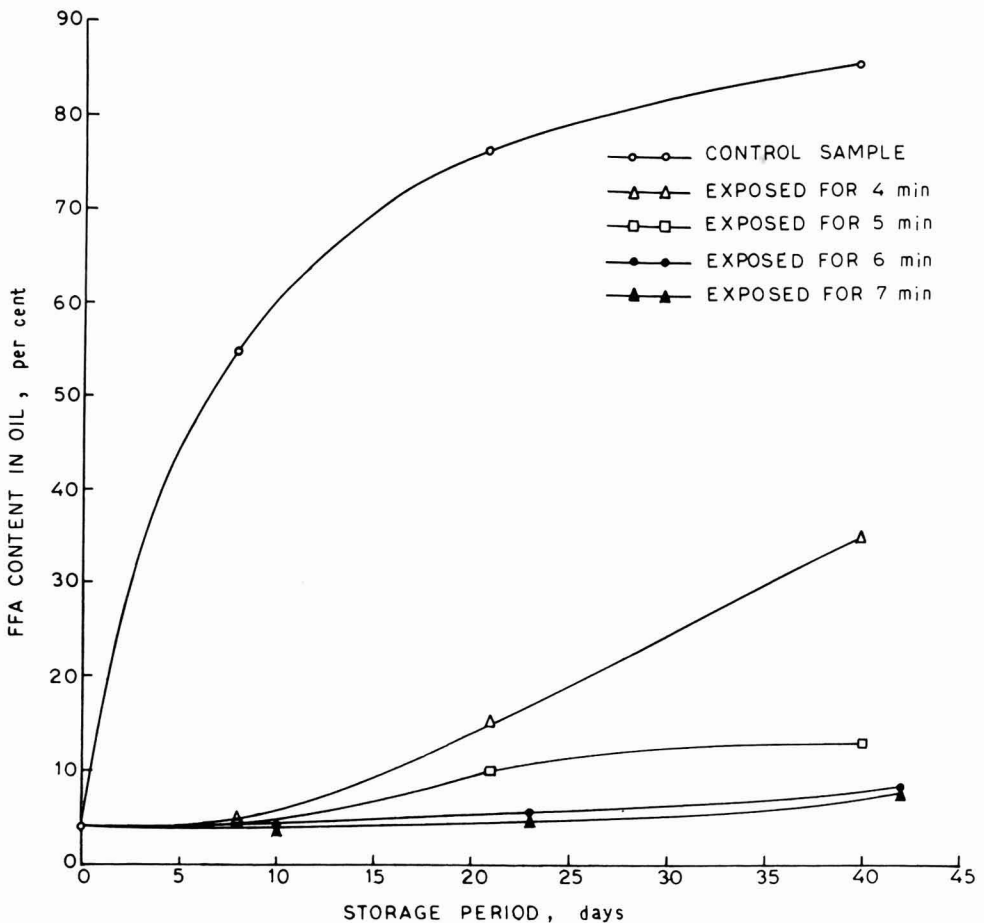
FIG. 3. EFFECT OF DIELECTRIC HEATING ON FFA CONTENT OF RICE BRAN DURING
STORAGE IN SEALED POLYETHYLENE BAGS

The FFA contents furnished are the mean of three replications. The standard error for the means of FFA contents was found to be 0.24%. The initial FFA content oil of the bran obtained immediately after milling was found to be 4.2%. It may be seen from the figure that the increase in FFA content of the bran exposed for 6 and 7 min was only about 2% during the storage period of six weeks while for the untreated samples the FFA rise was observed to be 75% for the same storage period.

From this study it can be concluded that RF dielectric heating is quite effective for the inactivation of the enzymes in rice bran and such stablized bran can be safely stored at least for a period of six weeks in sealed polyethylene bags keeping the FFA well below the limiting level of 10% for edible grade oil production. For stabilization of rice bran using RF energy, optimum exposure period required is 6 to 7 min at an average field intensity of 0.5 KV/cm for the dielectric heater operating at 13.56 MHz.

ACKNOWLEDGMENTS

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OPTIMIZATION OF ENZYMATIC HYDROLYSIS OF CANOLA MEAL WITH RESPONSE SURFACE METHODOLOGY

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ABSTRACT

Hydrolysis of Alcalase 0.6L of canola meal increased its soluble protein content, thereby reducing fermentation time necessary for production of a condiment similar to soy sauce. Using soluble nitrogen content of the hydrolysate as a criterion, and with the aid of response surface methodology, optimum conditions for the enzymatic reaction were: 69°C, pH 9, enzyme/substrate ratio of 0.31:1 (v/w), meal/solvent ratio of 1:10 (w/v) and reaction time of 2 h. The second order polynomial equation used to calculate the total soluble nitrogen yield (y) from a combination of temperature (x_1), pH (x_2) and enzyme/substrate ratio (x_3) was $y = 0.479 + 0.0083x_1 + 0.00077x_2 + 0.011x_3 - 0.015x_1{}^2 - 0.02x_2{}^2 - 0.023x_3{}^2 + 0.012x_1x_2 + 0.0051x_1x_3 + 0.00073x_2x_3$. The canonical form of the equation used to demonstrate the nature of the response surface was $y = 0.4813 - 0.01604w_1{}^2 - 0.02188w_2{}^2 - 0.02528w_3{}^2$ where w_1, w_2 and w_3 are the axes of the response surface. The experimental result of total soluble nitrogen obtained under optimum conditions for enzymatic reaction was 0.4882, which closely agreed with the calculated yield of 0.4813.

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INTRODUCTION

A condiment similar to soy sauce has been produced substituting canola meal for soybean as a substrate in the fermentation process (Ooraikul *et al.* 1980). They also demonstrated that an acceptable sauce could be produced in four weeks using HCl to hydrolyze canola meal prior to the usual microbial fermentation, instead of one year with microbial fermentation alone. However, acid hydrolysis, which requires refluxing for 10 h with 6% HCl, is rather expensive not only because of costly food-grade reagents but also because of the need to use expensive acid-resistant equipment. In addition, the so-called semichemical process produces substantial quantities of levulinic and formic acids which cause undesirable flavor (Fukushima 1981).

Aspergillus oryzae and *Aspergillus sojae*, the two mold cultures commonly used in the fermentation of soy sauce, are enzyme donors. Extracellular enzymes such as protease, sucrase, amylase, cellulase, lactase, lipase and phosphatase occur during sporulation of the molds in koji (Oba 1974; Yong and Wood 1977a, 1977b; Goel and Wood 1978; Kuninaka *et al.* 1980). Proteases and carbohydrates are by far the most imporant considering the nature of soy sauce, essentially a solution of solubilized proteins, sugars, organic acids and some minor flavoring constituents.

The extent of protein hydrolysis is one of the most important factors governing the quality of soy sauce. Soluble low molecular weight peptides and amino acids which contribute to the palate fullness of soy sauce are produced through the enzymatic breakdown of proteins. Yokotsuka (1977) reported that about 80–90% of the proteins were solubilized during soy sauce fermentation. Kunda and Manna (1975), Sekine (1976) and Impoolsup *et al.* (1981) have demonstrated that neutral and alkaline proteases predominate in the koji stage of fermentation, while Tsujita and Endo (1971) reported the presence of two acid proteases from *A. oryzae*, presumably predominate in the moromi stage.

Proteases may be effectively used to prehydrolyze substrates without the disadvantages associated with HCl. Alcalase 0.6L was chosen since it is a food-grade enzyme with optimum activity in the neutral and alkaline pH range (Novo Industri A/S, Alcalase 0.6L Technical Data). Enzyme activity data were based on hydrolysis of pure proteins such as hemoglobin, wheat gluten or soy isolate. Canola meal, on the other hand, is a complex substrate consisting of proteins, fiber, sugars, phenolic compounds (CIGI, 1982), which may even inhibit enzymatic activity. It is, therefore, necessary that optimum conditions with respect to pH, temperature, time, enzyme/substrate and meal/solvent ratios for the Alcalase 0.6L/canola meal system be determined to maximize protein hydrolysis. However, a one-factor-at-a-time approach for such determinations may be too approximate and time consuming. A response surface methodology (RSM) described by Box and Wilson (1951), which allows calculation of maximum yield based on a few sets of experiments in which all the factors are varied

within chosen ranges was used as the experimental design. This paper describes experiments which lead to determination of optimal conditions required for hydrolysis of canola meal with Alcalase 0.6L.

MATERIALS AND METHODS

Experimental Designs

Before RSM was applied, approximate conditions for hydrolysis, meal/solvent (M/S) ratio, temperature, pH, enzyme/substrate (E/S) ratio and reaction time were determined by varying one factor at a time while keeping the others constant. An appropriate range for each factor was determined for the RSM. Table 1 contains regimes used in the experiment.

Table 1. Regimes used in experiments

	M/S (w/v)	Temperature, °C	pH	Time, h	E/S (v/w)
M/S	1:3-1:25	60	7	3	-
Temperature	1:10	50-80	8.8	3	0.2
pH	1:12	66	7-10	3	0.2
Time	1:10	66	9	1-4	0.2
E/S	1:10	66	9	2	0.1-0.55

The M/S ratio experiment was conducted without Alcalase 0.6L to determine the appropriate quantity of water necessary for maximum extraction of soluble nitrogenous compounds from canola meal. An M/S ratio of 1:10 was optimal and used in subsequent experiments, except when pH was variable. In such cases a more diluted ratio of 1:12 was used because a greater quantity of buffer was required to keep the pH in the system constant. As optimal values of other factors became known they were also applied in subsequent experiments. The E/S was arbitrarily fixed at 0.2 for most experiments based on results from preliminary trials.

RSM with central composite rotatable design was used to optimize temperature, pH and E/S. The M/S and reaction time were fixed at 1:10 (w/v) and 2 h, respectively, since the M/S is dictated by the ratio commonly used in soy sauce production, while beyond 2 h no further increase in soluble nitrogen was detected. Three variables were assessed at five levels around the optima found in the one-factor-at-a-time experiments. Table 2 shows coding for each level of the variables. The α value of 1.682 was obtained from Myers (1971). Table 3 shows coded and uncoded combinations of variables at different levels.

Table 2. Codings for variables used in the optimization experiments

	Code				
	-1.682 (-α)	-1	0	1	1.682 (α)
Temperature, °C (x_1)	61.0	63.8	68.0	72.2	75.0
pH (x_2)	8.0	8.4	9.0	9.6	10.0
E/S Ratio, v/w (x_3)	0.20	0.24	0.30	0.36	0.40

Procedure

Specific quantities of Alcalase 0.6L (Novo Industri A/S, Enzyme Div., Novo Alle, DK-2880 Dagsvaerd, Denmark) as indicated in the experimental design were pipetted into 75 mL centrifuge tube containing 5 g defatted canola meal (Western Canadian Seed Processor Ltd., Lethbridge, Alta.), except for the M/S studies in which 10 g canola meal was placed in 250 mL centrifuge tubes without the enzyme. Appropriate quantities of buffer solutions with the desired pH [0.4 M phosphate buffer prepared as in Sorensen (1909) for pH 7–8.3, or 0.4 M carbonate-bicarbonate buffer prepared as in Delory et al. (1945) for pH 8.7–10], or deionized water in the case of the M/S experiments were added and the tubes shaken thoroughly. Suspensions were then incubated in a temperature controlled-shaker (New Brunswick Scientific Co. Inc., Edison, NJ) for specified periods of time at preselected temperatures as indicated in the design. The enzyme hydrolysates, or meal-solvent suspensions, were centrifuged at 14,000 rpm (Beckman model L2-65B with rotor type 21, Palo Alto, CA), and the supernatants filtered under vacuum. Each filtrate was made up to its original volume

of added solvent with deionized water. Approx. 0.5 mL aliquots were analyzed for total soluble nitrogen, expressed as %TSN, using the micro-Kjeldahl technique described by Pearson (1976).

Table 3. Coded and uncoded variables of central composite rotatable design

Coded			Uncoded		
x_1	x_2	x_3	Temp (°C)	pH	E/S (v/w)
-1	-1	-1	63.8	8.4	0.24
1	-1	-1	72.2	8.4	0.24
-1	1	-1	63.8	9.6	0.24
1	1	-1	72.2	9.6	0.24
-1	-1	1	63.8	8.4	0.36
1	-1	1	72.2	8.4	0.36
-1	1	1	63.8	9.6	0.36
1	1	1	72.2	9.6	0.36
-1.682	0	0	61.0	9.0	0.30
1.682	0	0	75.0	9.0	0.30
0	-1.682	0	68.0	8.0	0.30
0	1.682	0	68.0	10.0	0.30
0	0	-1.682	68.0	9.0	0.20
0	0	1.682	68.0	9.0	0.40
0	0	0	68.0	9.0	0.30
0	0	0	68.0	9.0	0.30
0	0	0	68.0	9.0	0.30
0	0	0	68.0	9.0	0.30
0	0	0	68.0	9.0	0.30

RESULTS AND DISCUSSION

Alcalase 0.6L consists mainly of subtilisin A (subtilisin Carlsberg), a serine-type endoprotease which hydrolyzes most proteins, especially plant proteins, producing soluble low molecular weight peptides (Novo Industri A/S Technical

Data). According to the Technical Data, the enzyme activity is normally measured as degree of hydrolysis (DH) or % peptide bonds cleaved. Hydrolyses of heterogeneous systems such as canola meals, however, may involve many biochemical and chemical reactions. The use of DH as a measure of enzymatic activity may be inadequate or unsuitable in these cases. Therefore, in the canola/Alcalase 0.6L system total soluble nitrogen (TSN) was used as an index since it could be simply measured, yet adequately described the difference in the degree of enzymatic activity in various regimes. Furthermore, quality and price of soy sauce are determined by TSN and the ratio of amino nitrogen (AN) to TSN (Hesseltine and Wang 1979).

In the experiment where M/S was varied % TSN increased rapidly until the M/S ratio reached 1:5 (w/v), and rose gradually thereafter (Fig. 1). However, a more dilute ratio of 1:10 was chosen for other experiments since it was easier to study the system at lower concentration.
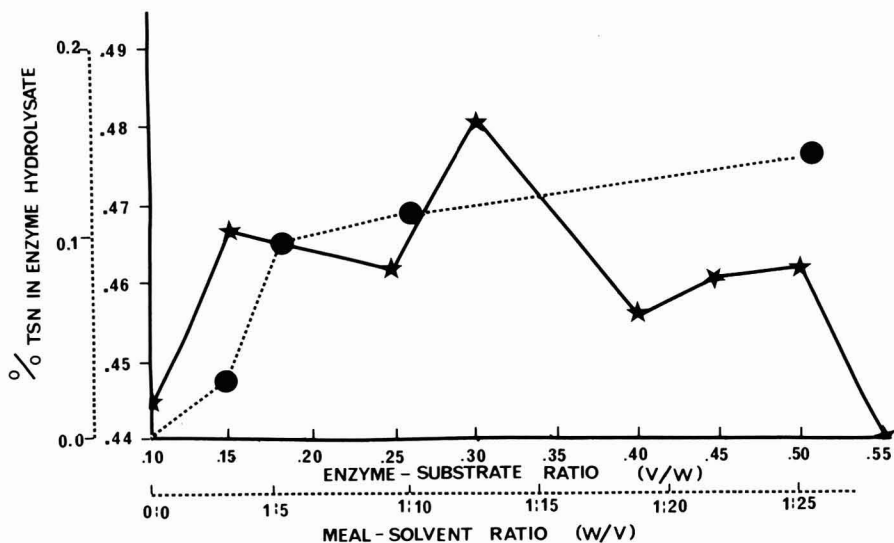
FIG. 1. EFFECT OF MEAL-SOLVENT (M/S) RATIO (W/V) AND ENZYME-SUBSTRATE (E/S) RATIO (V/W) ON TOTAL SOLUBLE NITROGEN YIELD (% TSN) DURING THE HYDROLYSIS OF CANOLA MEAL WITH ALCALASE 0.6L Temperature, pH and reaction time were fixed at 60 °C, 7 and 3 h, respectively, for the M/S experiment, and for the E/S experiment: 66 °C, 9 and 2 h, respectively, with M/S fixed at 1:10. Each value is an average of 2–4 replicates.

The temperature variation experiment showed a sharp increase in % TSN between 60° and 66°C followed by a rapid decline, while in the pH experiment maximum % TSN was obtained at pH 9 (Fig. 2). Note that the alkaline condition (pH 8–10) under which the hydrolysis took place was greater than the average pI value of the amino acids present in the hydrolysate (pK 6.0 at 25°C).
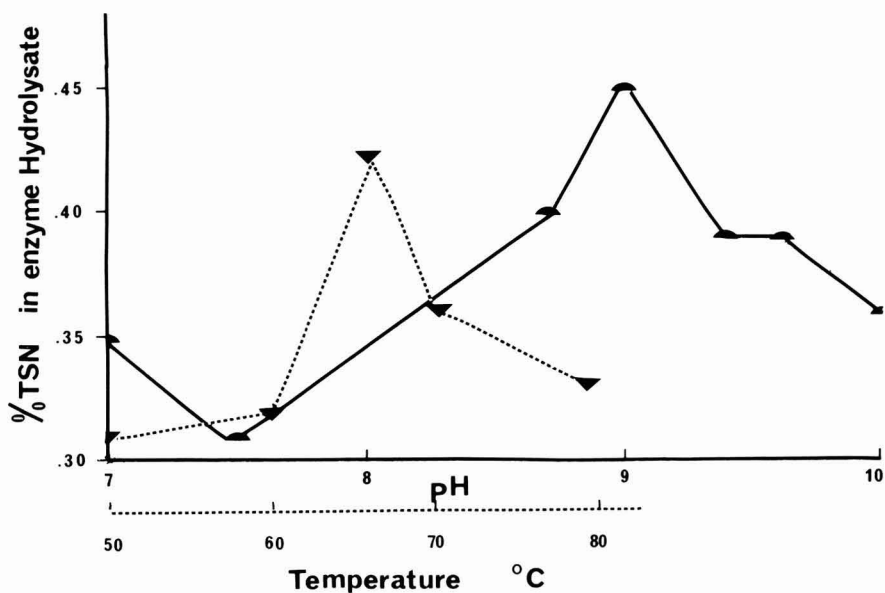
FIG. 2. EFFECT OF TEMPERATURE AND pH ON TOTAL SOLUBLE NITROGEN YIELD (% TSN) DURING THE HYDROLYSIS OF CANOLA MEAL WITH ALCALASE 0.6L M/S and E/S ratios, pH and reaction time were fixed at 1:10, 0.2, 8.8 and 3 h, respectively, for the temperature experiment. For pH experiment M/S and E/S ratios, temperature and reaction time were fixed at 1:12, 66°C and 3 h, respectively. Each value is an average of 2 – 4 replicates.

Figure 3 illustrates the effect of reaction time on TSN yield. The greatest % TSN occurred after 2 h hydrolyses, followed by a substantial decrease. The abrupt decrease of TSN was attributed to incubation conditions of 66°C and pH 9 that were suitable for Maillard reaction between reducing sugars in canola meal and the available amino acids. The resultant amino-carbonyl compounds might subsequently polymerize to form melanoidins or color pigments. The foregoing hypothesis appeared to be corroborated by the color of the mash, which was darker after hydrolysis than before. Reduction of the TSN after 2 h may be caused by reaggregation of soluble proeins to insoluble complexes.
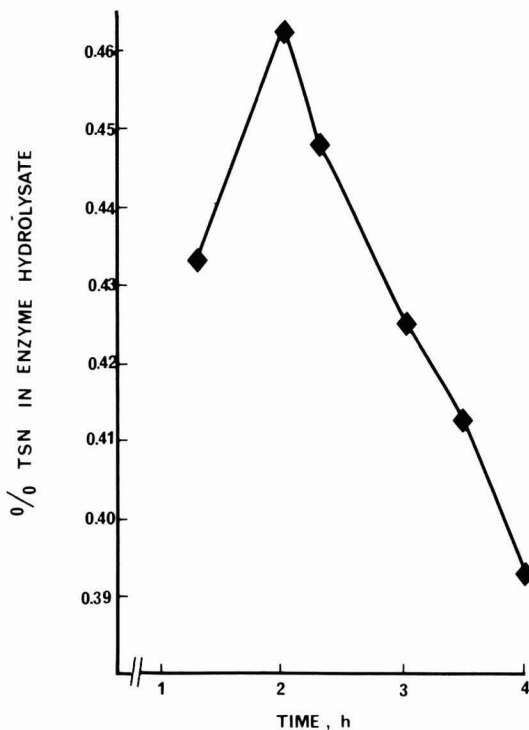
FIG. 3. EFFECT OF REACTION TIME ON TOTAL SOLUBLE NITROGEN YIELD (%TSN)
DURING THE HYDROLYSIS OF CANOLA MEAL WITH ALCALASE 0.6L M/S and E/S
ratios, temperature and pH were fixed at 1:10, 0.2, 66 °C and 9, respectively. Each value is an
average of 2 – 4 replicates.

No specific enzymatic activity trend, as measured by % TSN, indicated the ef-
fect of varying E/S ratio (Fig. 1). The results seemed to deviate from the general
rule of enzyme kinetics. Nevertheless, in a complex system such as canola meal
many factors may prevent a uniform rate of hydrolyses. For example, high fiber
content may prevent easy access of the enzyme to the substrate, and reactions
between proteins and other plant materials in the meal may inhibit hydrolysis.
Because of high substrate concentration, enzymatic activity might have reached
its plateau and, therefore, further increase in enzyme concentration may
decrease the activity.

The results of one-factor-at-a-time experiments do not reflect actual changes
in environment as they ignore interactions between factors which are present
simultaneously. More sophisticated experimental designs such as the RSM can
describe concomitant effects more fully and enable more accurate optimization
of factors. Practicality permitted the study of only a few rate controlling factors,
i.e., temperature, pH and E/S ratio, with RSM.

The Central Composite Rotatable Design was employed in the RSM. The technique was not used to obtain understanding of the physical mechanism of the system, although it might assist in gaining such knowledge, but to predict the response of the system to the changing environment. The experimental design is rotatable since the variance of the predicted response, y, at some points, x, is a function only of the distances of the points from the center (stationary point), and not a function of direction. This implies that the variance contours of y are concentric cirlces. Furthermore, such a design will leave the variance of y unchanged when the design is rotated about the centre (0,0,0), hence the name rotatable design (Montgomery 1984). There were five replicates at the central coding condition (0,0,0), i.e., temperature of 68 °C, pH 9 and E/S ratio of 0.3 (v/w). They were used to evaluate the reproduceability of the experiment.

Table 4 presents yields of TSN with corresponding temperature, pH and E/S ratio. The second order polynomial equation obtained from the analysis of multiple regression on the coded data (Table5) was:

$$y = 0.479 + 0.0083x_1 + 0.00077x_2 + 0.011x_3 - 0.015x_1^2 - 0.02x_2^2 - 0.023x_3^2 + 0.012x_1x_2 + 0.0051x_1x_3 + 0.00073x_2x_3$$

Examination of the fitted model with a t-test indicated that quadratic terms (x_1^2, x_2^2, x_3^2) with one linear term (x_3) and one interaction (x_1x_3) were significant at $p = 0.05$. The F-value and the correlation coefficient (r) of the overall model were also significant at $p = 0.05$, while the reproduceability of the experimental results was good. Therefore, the fitted model was appropriate for the description of the response surface. With the regression coefficients obtained, the stationary point of the fitted surface was computed using the equations suggested by Smith (1982). The predicted yield, y, together with the coded and natural variables of the stationary point, x_0, are shown in Table 6.

To characterize the stationary point, canonical analysis was performed on the second order polynomial equation to transform the fitted model to a new coordinate system with the origin at x_0. The axes of the system were then rotated until they were parallel to the principal axes of the response surface. The canonical form of the equation demonstrating the nature of the response surface was:

$$y = 0.4813 - 0.01604w_1^2 - 0.02188w_2^2 - 0.02528w_3^2$$

where w_1, w_2, w_3 are the axes of the response surface. Note that all the eigenvalues, i.e. -0.01604, -0.02188 and -0.02528, were negative, indicating that the stationary point was, in fact, a maximum with the surface slightly extended towards the w_1 axis (Myers 1971). Three-dimensional graphs of the response surface are shown in Fig. 4,5 and 6.

Using the conditions obtained at the stationary point, i.e. 69 °C, pH 9 and E/S ratio of 0.31 in an experiment with 2 h reaction time, the observed % TSN yield was 0.4882, which closely agreed with the calculated maximum yield of 0.4813. This yield is approx. 60% of the % TSN obtained by refluxing the meal with 6% HCl for 10 h.

Table 4. Observed and predicted yields of total soluble nitrogen (%)

Levels			Total Soluble Nitrogen (%, TSN)	
Temp, °C	pH	E/S (v/w)	Observed	Predicted
63.8	8.4	0.24	0.4252	0.4353
72.2	8.4	0.24	0.4167	0.4130
63.8	9.6	0.24	0.4026	0.4107
72.2	9.6	0.24	0.4241	0.4347
63.8	8.4	0.36	0.4398	0.4318
72.2	8.4	0.36	0.4352	0.4297
63.8	9.6	0.36	0.3980	0.4042
72.2	9.6	0.36	0.4560	0.4484
61.0	9.0	0.30	0.4401	0.4315
75.0	9.0	0.30	0.4450	0.4499
68.0	8.0	0.30	0.4335	0.4089
68.0	10.0	0.30	0.4131	0.4040
68.0	9.0	0.20	0.4494	0.4357
68.0	9.0	0.40	0.4341	0.4442
68.0	9.0	0.30	0.4882	0.4797
68.0	9.0	0.30	0.4861	0.4797
68.0	9.0	0.30	0.4716	0.4797
68.0	9.0	0.30	0.4746	0.4797
68.0	9.0	0.30	0.4774	0.4797

Table 5. Expected yield of total soluble nitrogen under various combinations of factors shown in the coded data, (x_1 = temp.; x_2 = pH; x_3 = E/S ratio)

x_1	x_2	x_3	x_1^2	x_2^2	x_3^2	$x_1 x_2$	$x_1 x_3$	$x_2 x_3$	% TSN/g Sample
-1	-1	-1	1	1	1	1	1	1	0.4252
1	-1	-1	1	1	1	1	1	1	0.4167
-1	1	-1	1	1	1	1	1	1	0.4026
1	1	-1	1	1	1	1	1	1	0.4241
-1	-1	1	1	1	1	1	1	1	0.4398
1	-1	1	1	1	1	1	1	1	0.4352
-1	1	1	1	1	1	1	1	1	0.3980
1	1	1	1	1	1	1	1	1	0.4560
-1.682	0	0	2.829	0	0	0	0	0	0.4401
1.682	0	0	2.829	0	0	0	0	0	0.4450
0	-1.682	0	0	2.829	0	0	0	0	0.4035
0	1.682	0	0	2.829	0	0	0	0	0.4131
0	0	-1.682	0	0	2.829	0	0	0	0.4494
0	0	1.682	0	0	2.829	0	0	0	0.4341
0	0	0	0	0	0	0	0	0	0.4882
0	0	0	0	0	0	0	0	0	0.4861
0	0	0	0	0	0	0	0	0	0.4716
0	0	0	0	0	0	0	0	0	0.4746
0	0	0	0	0	0	0	0	0	0.4774

Table 6. Coded and uncoded variables of the stationary point (point of maximum total soluble nitrogen yield)

Variable	Coded	Uncoded
Temperature (x_1)	0.232	69.0
pH (x_2)	0.216	9.0
E/S Ratio (v/w) (x_3)	0.131	0.31

Predicted yield - 0.4813 % total soluble nitrogen

Observed yield - 0.4882 % total soluble nitrogen

FIG. 4. A THREE-DIMENSIONAL RESPONSE SURFACE PLOT ILLUSTRATING OPTIMAL CONDITIONS FOR THE HYDROLYSIS OF CANOLA MEAL WITH ALCALASE 0.6L WHEN ENZYME-SUBSTRATE (E/S) RATIO WAS FIXED AT 0.3078 (v/w).

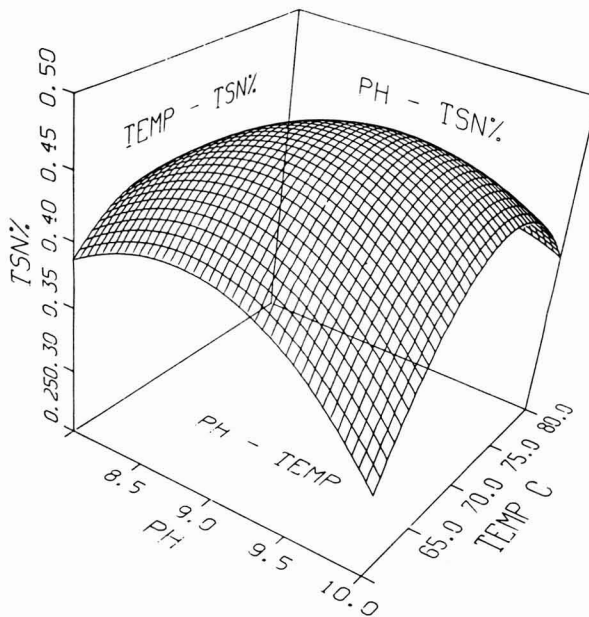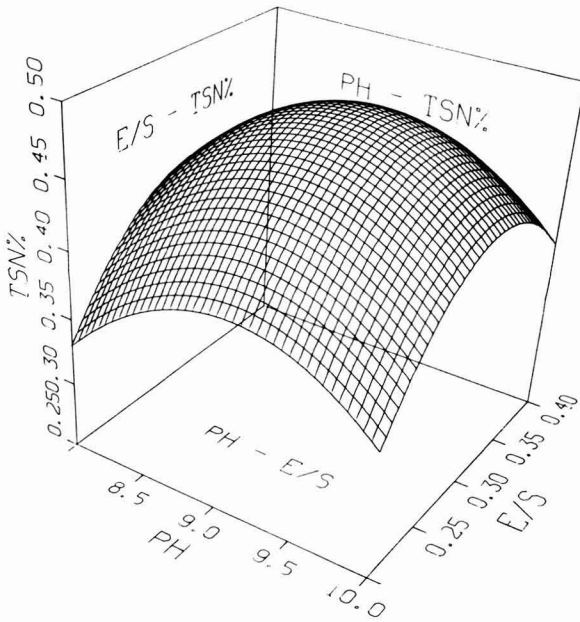FIG. 5. A THREE DIMENSIONAL RESPONSE SURFACE PLOT ILLUSTRATING OPTIMAL
CONDITIONS FOR THE HYDROLYSIS OF CANOLA MEAL WITH ALCALASE 0.6L WHEN
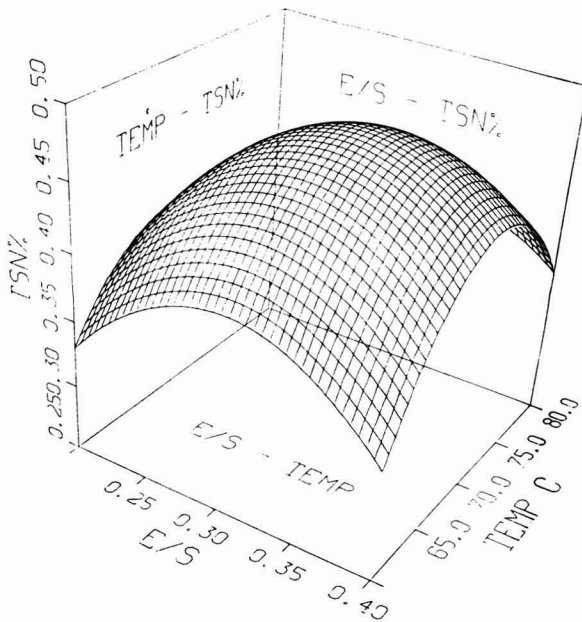TEMPERATURE WAS FIXED AT 68.96 °C.

FIG. 6. A THREE-DIMENSIONAL RESPONSE SURFACE PLOT ILLUSTRATING OPTIMAL
CONDITIONS FOR THE HYDROLYSIS OF CANOLA MEAL WITH ALCALASE 0.6L WHEN
pH WAS FIXED AT 9.02

The objective of this study was to obtain a high degree of protein hydrolysis in canola meal with Alcalase 0.6L in preparation for sauce fermentation, and RSM was used as a tool to determine optimum reaction conditions to attain that objective. No attempts were, therefore, made to understand kinetics of the enzymatic reaction or interactions of environmental variables involved. The variables chosen for the study were those most important in enzymatic hydrolysis. This study demonstrated that Alcalase 0.6L could be used effectively to hydrolyze canola meal and showed that RSM is a most efficient experimental design when several variables affecting the reaction are evaluated simultaneously. Optimum values of these variables for the reaction that would produce a highest TSN yield were obtained from a greatly reduced amount of experimental work. The effect of interactions between variables and reliability of the experiment could also be statistically evaluated when experimental results were fitted to the mathematical model.

The conditions considered optimum in this study apply, of course, only to the system herein described, i.e., defatted canola meal obtained from a canola oil extraction process hydrolyzed with Alcalase 0.6L. Other systems could be treated in a similar manner. Experimental design with RSM is a very valuable method of reducing work required for such studies.

ACKNOWLEDGMENTS

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EFFECTS OF SMOKING ON PROTEIN QUALITY OF ATLANTIC MACKEREL
(Scomber scombrus) [1]

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ABSTRACT

Atlantic Mackerel (Scomber scombrus) *fillets were hot-smoked using an AFOS-Torry Mini Kiln. The nutritional quality of both smoked and nonsmoked fish protein prepared from the same lot of fish, and of a standard protein casein, were evaluated by protein efficiency ratio (PER) in male rats. The hot smoking process caused a significant ($p \leq 0.05$) decrease in PER of protein from smoked mackerel muscle as compared with that from nonsmoked fish muscle. The change in protein quality was related to the loss of essential amino acids such as lysine and tryptophan and a reduced availability of lysine from smoked fish. Determination of lysine and tryptophan in three layers of the fillet (innermost, middle and outermost) showed that the maximum loss of these amino acids from smoking was in the outermost layer. Lysine and tryptophan were determined by high performance liquid chromatography and spectrophotometric methods, respectively. A small percentage of rats showed some evidence of pale liver on diets containing either casein or smoked mackerel.*

INTRODUCTION

Fatty fish such as Atlantic mackerel *Scomber scombrus* have been used as experimental dietary components to modify blood biochemistry through increased amounts of fish lipids (Singer *et al.* 1983, 1984). When some high fish diets are recommended variety is important, and smoked fish is an attractive variant. Smoked fish is also often eaten in the forms of appetizers and snacks. With modern distribution systems the need for the older "hot smoked" (part of the process<80 °C, part at 80 °C) product has lessened and "cold" smoking (≤ 33 °C) is more extensively used in technologically advanced countries. Cold-smoked products are cooked before being eaten. "Hot-smoked" products are more stable and suited for less advantaged countries, and as they require no further cooking are also very acceptable as snack items. Tabulated data for nutritional values of such products seldom includes the actual loss of nutritional value of the processed product compared to the unprocessed product.

Smoking of fish is a preservative procedure which as a whole combines the processes of salting (brining), heating and drying, and finally the addition of smoke itself. Therefore the overall effect of the smoking process on fish protein could result in amino acids being affected by any or all of these steps. When the ϵ-amino group of lysine is blocked by either aldehydes or by a Maillard reaction, the reacted lysine is not susceptible to enzymatic attack and thus becomes unavailable. Therefore, the measurement of lysine in processed food results in a value for total lysine which may not be appropriate for estimating nutritionally available lysine.

In smoking fish the loss of lysine and other essential amino acids is essentially proportional to the time and temperature of processing and may exceed 55% destruction in traditional hot-smoke tropical operations (Caurie *et al.* 1974). Other workers have recorded much smaller losses, in the range of 6 to 33% (Hoffman *et al.* 1977). Recent work on fish (Clifford *et al.* 1980) has confirmed that lysine is the most sensitive of the essential amino acids (a 25% destruction of lysine is accompanied by a 7% fall in other nutritionally critical amino acids, including histidine, arginine and N-terminal residues collectively), and that losses are much greater in the outermost 5–10 mm of the fish and decline rapidly toward the center. Accordingly one would expect lysine loss to be approximately inversely proportional to the thickness.

No information is available regarding the direct effect of smoking on tryptophan in fish, and this essential amino acid has also now been examined in detail. With these considerations, the objective was to evaluate the protein quality by both chemical and bioassay procedures, in Atlantic spring mackerel, a species commercially valuable in the North American market.

MATERIALS AND METHODS

Edible parts (muscle, skin on) of the fish were smoked using AFOS-Torry Mini Kiln according to the method described in Torry Advisory Note No. 82 by Bannerman (1980). The fish were obtained from the mackerel trap fisheries in Mill Cove, near Halifax, Nova Scotia, Canada, on June 17, 1983. During the dressing operation the head, viscera and backbone were removed. A black membrane lining the belly flap was also removed. Half of the lot of fish was used for salting and subsequent smoking. The remaining half was stored in a cold room at $-30\,°C$. All of these dressed and/or processed fish (dressed raw, dressed salted, and smoked) were vacuum packed in Cryovac B-620 multilayer barrier bags, and were stored at $-30\,°C$ for no more than three months prior to analysis. Full details of processing are given elsewhere (Bhuiyan *et al.* 1986a), but in brief the dressed fish were brined before smoking in an appropriate strength (relative to size) of NaCl solution, increasing the salt concentration from 0.45% in nonsmoked fish to 4.23% in smoked fish. Fish were smoked about 3 h; first at $30\,°C$ for about an hour, then at $50\,°C$ for about 45 min, and finally for the rest of the time from 50 to $80\,°C$. The decisions of the kiln operator are very much based on experience and vary with size of fish, etc.

The protein quality of both nonsmoked and smoked fish of the same batch was evaluated by identical chemical and bioassay procedures. Freeze-dried mackerel (nonsmoked and smoked) was prepared from three successive layers of skinned fillet, from the outside surface inwards. Each layer was about 4 mm thick. The proximate compositions of each layer were determined by methods given below. Both available and total lysine were determined according to HPLC methods (Peterson and Wartheson 1979). For total lysine (as didansyl lysine) HPLC conditions were: column, μ-Bondapak C_{18}; solvent, acetonitrile: 0.01 phosphate buffer, pH 7.0 (39:61, v/v); UV detector, 254 nm; flow rate, 1 ml/min; and attenuation, 1.0 AUFS. For available lysine (as DNP-lysine) HPLC conditions were: column, μ-Bondapak C_{18}; solvent, acetonitrile: 0.01 M acetate buffer, pH 4.0 (20:80, v/v); flow rate, 1 ml/min; UV detector, 436 nm; and attenuation, 0.2 AUFS. Trytophan was determined according to the spectrophotometric method of deVries *et al.* (1980). All amino acid standards used in this study were purchased from Sigma Chemical Co., USA.

Mackerel muscle was prepared for the feeding study by freeze-drying using a Stokes freeze-dryer (F.J. Stokes Corp., Philadelphia, PA). PER was determined, for the nonsmoked and smoked mackerel, and casein, according to the section 42.212 method of the AOAC (1980). Casein was used as a reference standard protein. Experimental animals were male weanling rats of Wistar strain from Charles River Canada Ltd., 21–25 days old and weighing approximately

35 g before the acclimation period. Corn starch was used as source of carbohydrate instead of glucose as suggested by the AOAC method. The levels of vitamins used in these diets were similar to the recommendations of the American Institute of Nutrition (Anon 1977). The composition of the experimental diets is shown in Table 1. The composition of food ingredients i.e., minerals, vitamins and carbohydrates, was essentially uniform in proportion and quality except for the protein supplements. The level of protein sources was adjusted to make all diets isonitrogenous.

Table 1. Composition of the experimental diets

Ingredients	Casein group	Freeze-dried spring mackerel groups	
		Non-smoked	Smoked
Corn starch	68.4	67.9	66.2
α-celluose[a]	5.0	5.0	5.0
Vitamin mix[b]	1.0	1.0	1.0
Bernhart-Tomerelli Salt Mix (modified)[c]	4.0	4.0	4.0
Choline chloride	0.2	0.2	0.2
Corn oil[d]	10.0	10.0	10.0
Casein	11.4	-	-
Non-smoked mackerel	-	11.9	-
Smoked mackerel	-	-	13.6

[a]Purchased from ICN Nutritional Biochemicals, Cleveland, Ohio

[b]Vitamin composition identical to American Institute of Nutrition vitamin mixture 76A.

[c]Salt mixture USP XVII, US Biochemical Corp., Cleveland, Ohio.

[d]Mazola corn oil, Canada Starch Co., Inc., Montreal

The proximate compositions of the casein and of nonsmoked and smoked (both freeze-dried) mackerel, as well as of the experimental diets, were determined prior to diet formulation (Table 2). Protein was determined by the rapid micro-Kjeldahl method of Concan and Soltess (1973). Moisture was determined by keeping the sample in an oven at 110 °C until constant weight was obtained. Ash was determined by an AOAC method (1980). For lipid determination the method of Bligh and Dyer (1959) was used.

Table 2. Proximate composition of the experimental diets

Diet	Percentage[a],[b]			
	Moisture	Protein	Lipid	Ash
Casein	10.8 ±0.42A	10.3 ±0.38A	10.0 ±0.32A	3.9 ±0.08A
Non-smoked fish	10.5 ±0.52A	10.3 ±0.19A	9.9 ±0.29A	4.2 ±0.11A
Smoked fish	10.1 ±0.38A	10.0 ±0.46A	10.1 ±0.18A	5.0 ±0.13A

[a]Mean ± std. error

[b]Data within a column with the same letter are not significantly different (p ≤ 0.05)

The feeding experiment was of 28-day duration. At the end of the experiment, PER for each feed and for the reference standard casein were calculated by the following formula:

$$PER = \frac{\text{weight gain in grams}}{\text{weight of protein consumed in grams}}$$

After the termination of the experimental period all the rats were subjected to physical observation and gross examination of different organs. General appearance (behavior, coat) and eyes were checked individually. Animals were killed, after physical observation, by excess of CO_2 gas. Liver, kidney, heart, and spleen were removed, weighed, and physically examined.

The experiment was of a randomized complete block design. The randomization procedures were adopted after culling out excessively large and small rats. The method of analysis of variance as outlined by Steel and Torrie (1960) was applied to all data obtained from this experiment and the means were compared using Duncan's multiple range test.

RESULTS AND DISCUSSIONS

The total and available lysine and tryptophan contents of smoked and nonsmoked mackerel fillet are presented in Tables 3 and 4, respectively. All amino acid values are expressed as mg/g of protein. The total lysine (8.4%) found in mackerel muscle (Table 3) is comparable to the values reported by others(Neilands et al. 1949; Braekkan and Boge 1962). The available lysine content of smoked mackerel has not previously been reported.

Table 3. Total and available lysine content of nonsmoked (NSM) and smoked (SM) spring Atlantic mackerel in different layers of fillet and whole fillet

Location of sampling	Treatment	Total lysine mg/g protein[a,b]	Available lysine mg/g protein[a,b]	% lysine loss on smoking Total	Available
Innermost layer	NSM	90.3 ±1.16	82.1 ±0.73		
	SM	87.3 ±1.18	78.5 ±1.35	3.3	4.4
Middle layer	NSM	81.2 ±0.67	79.4 ±0.72		
	SM	77.6 ±0.88	74.8 ±1.12	4.4	5.8
Outermost layer	NSM	78.0 ±0.89	67.8 ±1.05		
	SM	72.0 ±0.75	61.8 ±1.73	7.7	8.8
Whole fillet	NSM	84.2 ±0.72	75.6 ±0.93		
	SM	79.9 ±1.22	70.6 ±0.91	5.1	6.6

[a]Mean ± std.error
[b]significant loss of lysine as tested by paired-difference 't' test (P≤0.05) between NSM and SM.

Both available and total lysine suffered comparable losses due to the smoking process. As expected, the magnitude of decrease was higher for available lysine than total lysine. The losses of total and available lysine in whole fillets were 5.1 and 6.6%, respectively. The outermost layer of the fillet showed the highest loss. Hoffman et al. (1977) reported a loss of approximately 11% when they smoked tilapia (Tilapia lidole) at 75 °C for 38 h. However, the smoking of

mackerel at a lower temperature has resulted in relatively smaller loss of lysine. It appears that some of the damage of lysine may be a function of temperature alone, or a combined function of temperature and other food constituents resulting in Maillard-type browning reactions. Thus lysine loss of this type is not directly attributable to the smoking process in the strict meaning of the words. Smoke constituents do have an adverse effect on lysine (Chen and Issenberg 1972), and this can occur even during cold smoking (Dvorak and Vognarova 1965). These changes are not restricted to lysine alone since during hot smoking other amino acids are also affected (Mauron 1970; Erylova et al. 1962). Thus the tryptophan in smoked fish must have been affected by the same factors. Information about the nature of the smoke constituents which react irreversibly with food is limited. For example it has been suggested that 4-substituted phenols do not react, although they are included in the phenols known to be important in smoke flavor (Toth and Potthast 1984).

The tryptophan content of the whole muscle is approximately 0.9% (Table 4), which is also comparable to the results of Braekkan and Boge (1962) and Neilands et al. (1949). Most tryptophan losses were observed in the outermost layer which is more exposed to heat and air than the successive inner layers. On a combined basis the total loss was 4.4% in whole fillet. The loss of tryptophan in fish protein as a result of the smoke processing has not previously been reported.

Table 4. Tryptophan content in whole and different layers of nonsmoked (NSM) and smoked (SM) spring Atlantic mackerel fillet

Location of sampling	Treatment	Total tryptophan mg/g protein[a],[b]	% loss on smoking
Innermost layer	NSM	9.5 ±0.35	
	SM	9.4 ±0.30	1.0
Middle layer	NSM	9.2 ±0.17	
	SM	8.7 ±0.27	5.4
Outermost layer	NSM	8.4 ±0.26	
	SM	7.8 +0.19	7.1
Whole layer	NSM	9.0 ±0.38	
	SM	8.6 ±0.32	4.4

[a]mean ± std.error
[b]Insignificant loss as tested by paired-difference 't' test (P≤0.05) between NSM and SM

Hydrolysates of nonsmoked and smoked mackerel fillet were reacted with dansyl chloride for HPLC determination. Using a standard of didansyl lysine the peak area response was linear with concentration for injections containing from 1.1 to 3.7μg($r^2 = 0.990$, significant at 5% probability level). Recovery of didansyl lysine from a known amount of lysine added directly to hydrolysate was 92.3 ± 1.73 and 93.1 ± 1.01% for nonsmoked and smoked samples, respectively. The ϵ-DNP-lysine peak for DNP derivatives of available lysine in nonsmoked and smoke samples came out separately from excess dinitrophenol in the system,

from the different α-DNP-lysine and from γ-DNP-ornithine. These were check-
ed separately using the same chromatographic conditions used for available
lysine determination. Using standard DNP lysine, the peak area response was
linear with concentration for injections containing from 1.08 to 5.40 μg DNP-
lysine (r^2 = 0.997, significant at 1% level). The recovery was 93.2 $\pm$ 0.98 and
93.4 + 1.68 for nonsmoked and smoked samples, respectively.   The spec-
trophotometric response at 590 nm for the tryptophan standards was linear over
the range 25.4 to 406.0μg tryptophan (r^2 = 0.996, significant at 1% level). The
recovery of tryptophan by this method for whole portions of nonsmoked and
smoked fish samples was found to be 95.0 $\pm$ 2.61 and 95.3 $\pm$ 3.38% respec-
tively.

The proximate compositions of different layers of mackerel fillets are shown
in Table 5. Both nonsmoked and smoked samples increase progressively in pro-
tein from the surface of the innermost layer, whereas lipid has an opposite trend.
Mackerel, in common with most other fish, have two types of muscle in the edi-
ble fillet. These are designated "red" and "white" muscle, the red muscle being
nearer the skin (Love 1970). Nova Scotia mackerel have been shown to have
much more lipid in the red muscle than in the white muscle, although in very fat
fish the white muscle fat content can approach that of the red muscle (Ackman
and Eaton 1971). This is an aspect of lipid distribution separate from the less
well-known subdermal fat (Ackman 1980) which is uniquely susceptible to
autoxidation (Ke et al. 1977). In most fish muscle the protein content is usually
constant and in the range of 18–20% on a wet weight basis. Triglyceride fat
merely displaces water in those species with variable fat contents. This effect is
confirmed in the nonsmoked mackerel of Table 5, since the greater ash content
of the innermost fillet layer to some extent reflects the natural salts present in the
higher water content. From Table 3 and 4 it is obvious that a similar trend of
progressive increase of lysine and tryptophan contents exist, maintaining a rough
proportionality with the increase of protein content. The ash content in the brin-
ed and smoked mackerel is nearly uniform, indicating that salt has penetrated the
whole of the fillet examined.

Table 5. Proximate composition[a] of different layers of freeze-dried nonsmoked (NSM) and smoked
(SM) spring Atlantic mackerel fillet

Treatment	Layer	% Moisture	% Protein	% Lipid	% Ash
NSM	Innermost	1.7 ±0.08	86.8 ±1.10	5.0 ±0.23	7.4 ±0.29
	Middle	1.5 ±0.09	82.0 ±0.76	9.2 ±0.44	6.8 ±0.45
	Outermost	1.4 ±0.09	81.2 ±0.52	12.2 ±0.57	5.6 ±0.26
SM	Innermost	1.4 ±0.02	76.2 ±0.76	5.2 ±0.08	17.9 ±0.41
	Middle	1.8 ±0.12	75.2 ±1.00	6.3 ±0.13	18.1 ±0.29
	Outermost	1.5 ±0.11	70.4 ±1.15	9.2 ±0.43	18.0 ±0.13

[a]Mean ± std. error

The proximate composition of the diets containing casein, nonsmoked and
smoked mackerel employed to measure the PER in rats is summarized in Table

2. There was no significant difference between the diets in protein and lipid contents. The diet containing smoked fish had a higher level of ash than other diets, mainly because of high level of salt acquired during the brining of fish prior to smoking process (Bhuiyan et al. 1986a).

The performances of rats fed various protein supplements are shown in Table 6. Growth as well as PER of rats fed smoked fish protein was significantly ($P \leq$ 0.05) lower than for rats fed nonsmoked fish protein. Although the weight gain was lower on the smoked fish diet, rats increased intake of this protein, presumably to meet their essential amino acid requirements. The PER of casein and nonsmoked mackerel were 2.77 and 2.83, respectively. Morrison and colleagues (1960, 1961) found that fish flour was more efficiently utilized than casein in terms of PER. Several workers have found the PER of various fish proteins (e.g., cod and red hake) to be higher than that of casein (Munro and Morrison 1965; Finch 1970; Dubrow et al. 1971).

Table 6. Effects[a] of smoking mackerel (spring) on the performance[b] of growing male rats (28 days) and protein efficiency ratio (PER)[a,b]

Diet Group	Average Initial Weight (g)	Average Final Weight (g)	Average weight gain (g)	Average food consumed (g)	Feed/ Gain	Average protein consumed (g)	PER
Casein	55.2±1.14A	162.8A,B	107.6±5.60A,B	379.9A,B	3.53A	39.0±0.86A	2.77 ±0.10A,B
Non-smoked	55.4±1.10A	165.7A	110.3±2.26A	377.4A	3.42B	38.9±0.57A	2.83 ±0.08A
Smoked	55.8±1.06A	155.6B	99.8±3.84B	387.1B	3.87A	38.6±0.88A	2.58 ±0.06B

[a]Means ± standard error
[b]Data within a column with the same letter are not significantly different (P<0.05)

It is generally recognized that heat treatment applied during the processing of foodstuffs causes a reduction in the quality of constituent proteins. The decrease in protein quality is mainly attributed to the reaction of the ε-amino acid group in the lysine with the reducing part of carbohydrates, resulting in the formation of physiologically unavailable product. Platt (1961) reported that heat treatment and drying alone, which are integral parts of the smoking process, can reduce the biological value of meat and fish protein with losses exceeding up to 50%. Hoffman et al. (1977) found that drying at 75 °C and smoking at 100 °C caused a significant reduction in protein quality of smoked fish measured in terms of net protein utilization.

The decrease in the PER value of smoked mackerel muscle is probably attributable to the loss of essential amino acids. However, in the present study losses of only lysine and tryptophan (Tables 3 and 4) were demonstrable. Some workers have reported losses of serine, threonine and sulfur-containing amino

acids during a hot smoking process (Mauron 1970; Erylova 1962). Methionine remains the first limiting amino acid in heated fish (Miller *et al.* 1965) because lysine is present in relative excess in the raw material. The fall in the level of these essential amino acids must have caused a decrease in the nutritive value of smoked mackerel.

Rats placed on experimental diets showed (Table 7) no significant difference in their liver, kidney, spleen and heart weights regardless of the dietary protein. There was no mortality during the experimental period in any of the dietary groups. Physical examinations of individual rats revealed that none of the rats had any abnormality in the eyes. Gross examination of various organs showed pale liver in three rats fed casein and one rat fed smoked mackerel muscle. The pale discoloration resulted from the sporadic presence of depigmented spots which were evenly distributed throughout the liver surface, probably due to fatty infiltration.

Table 7. Organ weights[a,b] of rats fed diets containing smoked and nonsmoked mackerel and casein for 28 days

Diet Group	Body wt	Liver wt	Kidney wt	Spleen wt	Heart wt	Gross changes in liver (%)[c]
Casein	162.8A,B	10.1±0.67A	1.9±0.09A	0.6±0.02A	0.7±0.04A	3/10
Non-smoked	165.7A	10.3±0.35A	1.8±0.06A	0.6±0.02A	0.7±0.05A	0/10
Smoked	155.6B	10.1±0.47A	1.9±0.06A	0.6±0.02A	0.7±0.04A	1/10

[a]Mean ± std. error

[b]Data within a column with the same letter are not significantly different (P≤0.05)

[c]Pale liver incidence

Deficiencies of methionine or choline, two important lipotropic factors, are known to cause fatty infiltration in rat liver (Newberne 1978). All diets were adequately supplemented with choline chloride. Therefore, it is likely that a marginal deficiency of methioine in casein and in smoked fish may have caused fatty infiltration in these rats. Methionine is the first limiting amino acid in casein (Newberne 1978; McLaughan 1972) and processed fish protein (Stillings *et al.* 1969; Chichester 1974; Tannenbaum 1974). The low prevalence of liver abnormality in both the casein and smoked fish groups supports the view of Newberne (1978) that individual animals suffering from nutritional deficiencies have wide variations in tissues reaction and response to the development of deficiency symptom.

Our results indicate that even the mild "hot smoking" process used could slightly modify the protein quality of mackerel. On the other hand, although total protein is increased from 18.3% in raw fish to 24.0% in the finished product (Bhuiyan *et al.* 1986a), the valuable lipid components are not altered (Bhuiyan *et*

al. 1986b). The protein quality of other animal protein supplements is also known to be altered by such processing, particularly with the loss of essential amino acids (Mauron 1970; Erylova 1962; Von der Decken 1983; Dvorak and Vognarova 1965). It is unlikely that smoked mackerel will be the sole source of protein for human consumption, and thus create a serious health problem, except in exceptional circumstances. For example, Singer *et al.* (1983; 1984) fed canned mackerel as an experimental supplement replacing part of a normal diet. From a health safety point of view the few abnormalities observed to be associated with defatted mackerel as the sole source of protein in experimental rats is encouraging. Moreover a sound policy in protein nutrition is to rely on a variety of protein sources in the selection of the diet as they are likely to be more complete and balanced in amino acids than a single protein source.

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DEHYDRATION OF POTATO: 3. INFLUENCE OF PROCESS PARAMETERS ON DRYING BEHAVIOR FOR NATURAL CONVECTION SOLAR DRYING CONDITIONS

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ABSTRACT

Conditions for conducting laboratory simulations of natural convection solar-drying were investigated. In the solar-drying experiments, air speed in the empty drying chamber (called the air flow potential, AFP) varied from 1.3 to 1.8 m/s, while inlet temperature to the loaded drying tower varied from 57 to 71°C. In laboratory simulations of solar-drying, the influence of variation of air flow rate, product loading density and inlet air temperature on drying behavior of potato sticks (12.5 x 6 x 40 mm) was investigated.

It was determined that potato sticks could be solar-dried in 5–6 h to a water activity of 0.7 (15 g water/100 g solids) under natural convection conditions. Drying rate increased with increase of inlet air temperature and/or AFP, with the effect of AFP being most noticeable at the beginning of the drying process. The time required to remove 96% of the original moisture varied by ± 5% for the AFP variation noted in solar-drying (1.54 ± 0.23 m/s) and varied ± 10% for the variation of inlet air temperature noted in solar-drying (65 ± 5°C), indicating a higher sensitivity for natural convection solar-drying to variability of inlet air temperature than fluctuating AFP. The ratio of increase in drying time to increase in bed depth is less than 1, so overall dryer productivity increased with increasing bed depth.

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INTRODUCTION

While sun drying of fruit and vegetables has been practiced for many years, an interest has arisen in increasing the efficiency of utilization of solar energy through development of several specialized solar drying systems (Bolin and Salunke 1982). Advantages of using solar energy for drying are that the energy is free, nonpolluting and readily available in tropical and subtropical countries.

Solar-drying of potato has been reported in the literature for various solar-drying systems. Gupta (1978) solar-dried 5 mm thick potato slices using a 2 stage system (flat plate collector + water heater/storage tank) for heating the inlet air. At a 30 kg/m loading, the moisture content after 10 h of drying had fallen from 386 to 23 g water/100 g solids. While the heat exchanger/storage tank extended the daily drying time available, inlet temperatures were quite variable (26 − 62 °C) due to heating of the water in the morning.

Smith *et al.* (1979) dried diced potato in a solar-drying unit with auxillary electric heaters. When drying 90 kg/day (30 cm bed) of diced potato from 80% to 7% moisture, under good solar conditions, 58% of the energy required was supplied by the solar collector, the remainder from the electric heaters.

Shaw (1981) dried 1 ton/day of cooked, shredded (2 mm) potato at a loading of 10 kg/m² and an air speed of 0.45 m/s in a large scale 2-stage solar-dryer. In stage 1, an overnight drying in ambient temperature wind, the potatoes lost about 45% of their water and became shrunken in appearance. The next day (stage 2), the half-dried potato was placed to receive direct solar radiation for the final drying to 10% moisture.

Kalra and Bhardwaj (1981) compared solar and sun-drying in India. For potato sticks, a solar-drying time of 8 h gave a moisture content of 5%, while sun drying required 10 h to achieve a moisture content of 7%.

In an investigation of solar-drying of taro root slices and shreds, Moy *et al.* (1980) showed that the higher surface to volume ratio of the shreds compared to the slices did not necessarily give more effective solar-drying, as the shred pieces tended to clump together, thereby impeding air passage. Islam and Flink (1982 a,b) studied solar dehydration of potato under natural convection conditions, including an osmotic preconcentration step for increasing product throughout.

Recently, Pinaga *et al.* (1982, 1983) have presented results of solar-drying experiments of various vegetables (not including potato), while Chiang and Petersen (1985) have recently investigated partial air-drying (not solar-drying) of blanched, french-cut potato to 60% moisture, as a prestep to frying and freezing. In these studies, the effect of air temperature and air velocity were presented.

Quantitative evaluations of drying behavior under natural convection solar-drying conditions are important for process design. While Islam and Flink (1982a) indicated that air flow conditions are important, a systematic quantitative evaluation was not conducted. In addition, the influence of inlet air temperature variation under natural convection air flow conditions was not investigated. The current study investigates the influence of air flow rate and inlet air temperature on drying behavior for natural convection solar-drying conditions, and in addition reexamines the influence of loading density on drying time.

MATERIALS AND METHODS

Raw Material

For the major part of this study, potato (Bintjee variety) was obtained from the Danish Potato Research Station (Vandel, Denmark). In a few later experiments, potato was obtained at the local supermarket. Potato was washed and hand-peeled. A French-fries cutter was used to prepare the potato sticks, samples of imperfect cross-section being removed. A grater was used to prepare shreds. After cutting the sample was taken immediately to drying.

The Two-Way Drying System

A detailed description of the "two-way" drying system (having solar and laboratory configurations) is given in Islam and Flink (1982a). Such a "two-way" system was necessary since (1) solar-drying experiments cannot be conducted year-round in Denmark and (2) variable climatic conditions give difficulties in obtaining reproducible process parameters for solar-drying studies, especially for natural convection. In brief, the system comprises a basic dryer body (called the tower) which can be fitted to either a flat-plate solar panel (1.25 m²) or an electric heater/fan combination. The dryer body holds up to 8 trays with wire mesh bottoms (18 x 18 cm); air passes up through the bed of sample on each tray. The solar panel consists of a corrugated black plate heat absorber situated beneath a double-glazed plexiglass cover. The back and sides are insulated with polystyrene foam.

In actual solar drying studies, air temperature to the tower and air flow rates are not controllable, while in laboratory studies, air temperature and flow rates are controlled. In the laboratory, air speed is regulated by means of the fan voltage. Air speed was measured using a mechanical annemometer (Stoppani AG, Bern, Switzerland). The five fan voltage chosen (from 115 to 210v) gave

air velocities in the empty tower of 1.30, 1.50, 1.77, 2.18 and 3.38 m/s. These air velocities are designated the air flow potentials (AFP) for these fan voltages. When the tower is loaded with sample, the actual air speeds for the same fan voltage settings will be lower, their magnitude depending on loading density, piece size and shape, and extent of drying. During a drying experiment, sample weight, air temperature and air speed were measured periodically. From the sample weight data, and the sample's original solids content, drying curves were calculated on the basis of weight (gm), moisture content (MC = g water/g solids), and moisture ratio (MR = MC(t)/MC(i)). Using a curve-fitting computer program which was available, a second-order polynomial of the form

$$x = At^2 + Bt + C \quad \text{where } x = W, MC \text{ or } MR$$

was tested as a means for characterizing the data. In almost all cases, correlation coefficients were higher than 0.999. It should be emphasized that a high correlation for a second-order polynomial does not imply any theoretical basis, but indicates only a good fit of the data within the range of time used in these experiments. In all cases, the calculated results were compared with measured values to insure that the polynomial fit was satisfactory. The instantaneous drying rate was then obtainable from the first-derivative of the polynomial,

$$dx/dt = 2At + B.$$

An examination of the drying curve parameters obtained indicates that at the beginning of the drying process (ie. small t), parameter B is indicative of drying rate. Parameter A first becomes influential at later stages of the drying.

According to Islam and Flink (1982a), shelf-stable potato could be obtained by drying to a water activity of 0.7 or below. From measurements of the water sorption isotherm of freeze-dried potato in a preliminary phase of this study, an Aw of 0.7 corresponded to a moisture content of 15 g water/ 100 g solids, and this value can be used together with the initial moisture content to calculate the desired final moisture ratio (MR). However, initial moisture content variation associated with differing potato maturity resulted in adopting MR = 0.04 as the average final MR for use in determining drying times to Aw = 0.7.

RESULTS AND DISCUSSION

Initial Test on Piece Size

Initial experiments were conducted in the laboratory drying system (at 65 °C; 1.50 m/s; 15 kg/m²) to determine which sample geometry and size to use in the main phase of this study. Sample geometries tested were slices (10 mm thick), sticks (12.5 x 12.5 x 40 or 12.5 x 6 x 40 mm), and shreds (5 x 7 or 2 x 2 mm). Both drying rate and final product quality (general appearance and acceptability) were included as criteria for choice.

Moisture contents after 210 min of drying was used as a measure of drying rate. For the 5 samples, the moisture contents (g water/100 g solids) were: 100 (10 mm slices), 80 (12.5 x 12.5 mm potato sticks), 28 (12.5 x 6 mm potato sticks), 14 (5 x 7 mm shreds) and 8 (2 x 2 mm shreds). It is seen that shreds dry at the fastest rate, with the 12.5 x 6 mm potato sticks being a little slower.

Product acceptability included a subjective organoleptic (visual, mouthfeel, taste) evaluation of the product as potato component in curry dishes (potato, or potato with green vegetables) as prepared on the Indian subcontinent. Shreds, due to their small piece size, failed to give the mouthfeel which is desired in the traditional Indian dishes, and were thus not studied further. All remaining samples were judged suitable quality-wise, but as the drying rate for 12.5 x 6 mm potato stick was significantly higher, this size was selected. (For another usage, however, shreds could be preferable due to their shorter drying time).

Solar-Drying

In conducting engineering studies of solar drying, it is necessary to resort to simulated solar-drying experiments, because it is difficult, if not impossible, to control and reproduce all process parameters during an actual solar drying. A particular problem is duplication of weather conditions (air temperature, wind velocity and direction, insolation levels, presence of clouds, etc.). To overcome this, we have utilized a "two-way" drying system which allows solar-drying and laboratory air-drying to be conducted in the same chamber. To conduct meaningful laboratory simulations of natural convection solar-drying, it is necessary to obtain operating data from actual solar drying experiments. The two most important operating conditions are air speed in the empty tower and air temperature at the inlet to the loaded tower.

The air velocity measured in the empty tower is important as this indicates the Air Flow Potential (AFP) (i.e., maximum air flow possible without product resistance) for the system under natural convection conditions. The actual air speed in the loaded tower will be determined by combining the AFP and product-based air flow resistances. For example, for a given set of weather conditions, which would result in a fixed AFP from the solar-panel, actual air flow in the dryer will vary with sample loading. In the laboratory, AFP can be set to a desired level by adjusting the fan voltage prior to loading the tower. The actual air speed will then change during drying as product resistances change. Both AFP and actual air speed are reported in the following sections in m/s. The above mentioned conceptual difference should be remembered to avoid confusion.

Knowledge of inlet air temperature for the loaded tower is also important, as this is a controllable parameter for laboratory drying studies. In solar-panel heating, outlet air temperature from the panel (ie., inlet to the dryer) will depend

on air flow in the panel (and dryer), and thus while AFP should be measured in the empty tower, inlet air temperature should be measured for the air flow conditions which actually exist during solar-drying.

Eight solar-drying experiments were conducted on sunny days using potato sticks loaded into 1 or 2 trays, each tray having 500 g. Drying was conducted for 300 min, this being adequate to evaluate drying behavior. Results of these studies are given in Table 1. AFP, measured for 6 of these tests, varied from 1.30 to 1.83 m/s, with an average of 1.5 m/s. On these good solar-drying days (ambient temperature between 22 to 27 °C), the inlet air temperature to the drying tower varied from 55 to 71 °C (average = 65 °C), while the actual air speed during drying varied from 0.5 to 1.2 m/s. On days with lower ambient temperature, or with cloudy or partly-cloudy conditions, lower values of AFP (0.7 to 1.2 m/s), air speed (0.3 to 1.0 m/s) and inlet air temperature (40 to 55 °C) were observed.

Table 1. Summarized data for solar drying experiments

Experiment no.	Average ambient temp. (°C)	Average Dryer inlet air temp. (°C)	Loading density (kg/m^2)	Piece size and shape[a]	Range of air speed during exp. (m/s)	Moisture Ratio at 300 min
1	24.5	67	15	A	0.5-1.0	0.076
2	23.5	66	15	A	0.4-1.0	0.103
3	26.5	69	15	B	0.5-1.0	0.038
4	25	67	15	B	0.6-1.1	0.039
5	24	60	15	B	0.6-1.0	0.044
6	23	63	31	B	0.5-1.2	0.053[b]
7	19	61	15	B	0.3-0.9	0.043
8	18	55	31	B	0.3-0.8	0.089[c]

a) A = 12.5 x 12.5 mm sticks. B = 12.5 x 6 mm sticks
b) MR for lowest 15 kg/m²: Average MR for whole bed = 0.077
c) MR for lowest 15 kg/m²: Average MR for whole bed = 0.147

Typical drying curves for solar-drying on sunny days are given in Fig. 1. Three solar-drying experiments (nr 3,4 and 7 in Table 1) conducted under similar conditions gave essentially the same drying curve (the shaded region), while a fourth experiment (nr 5 in Table 1) using potato having a lower solids content gave a different, slower drying curve. Besides demonstrating that it is possible to solar-dry potato sticks to the desired moisture content level within one day (5-6 h), these experiments provide important data for designing the laboratory simulations.
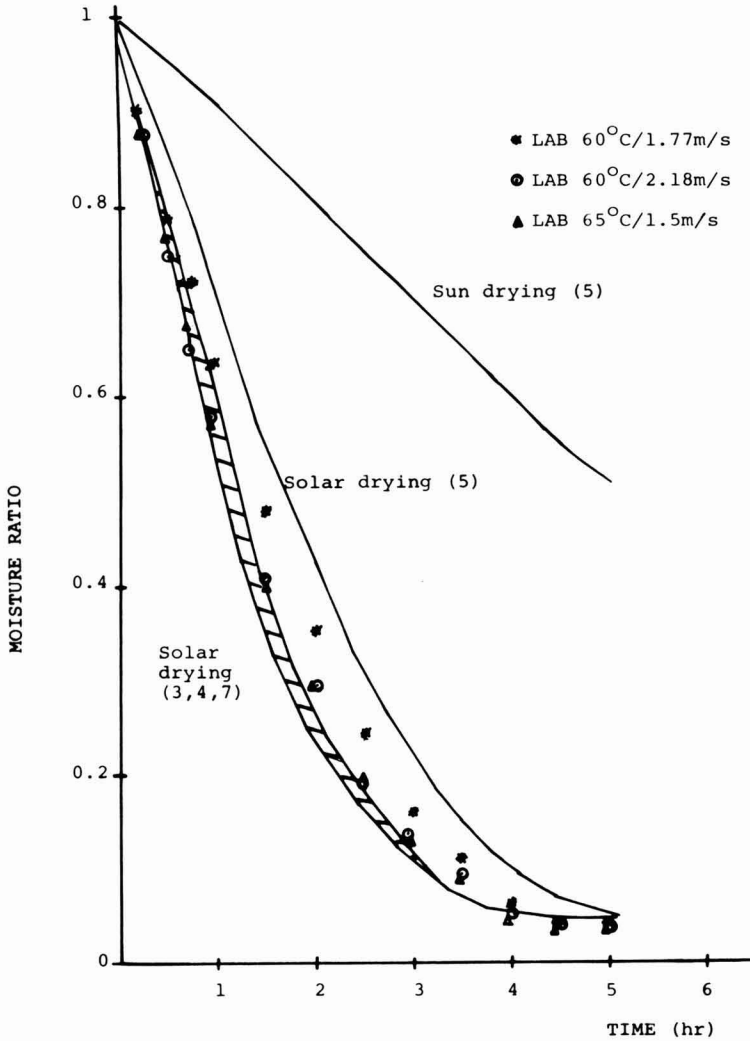
FIG. 1. DRYING CURVES (MOISTURE RATIO BASIS) FOR SELECTED EXAMPLES OF SOLAR-DRYING, SUN-DRYING AND LABORATORY AIR-DRYING OF POTATO STICKS AT 15 kg/m² LOADING
(Numbers for Solar- and Sun-Drying correspond to Table 1).

Also shown in Fig. 1 is a comparison of solar-drying and sun-drying (both marked nr 5) on an average sunny day (ambient temperature 24 °C). The original moisture content of the potato sticks was 483 g water/100 g solids. After 5 h, the moisture content for the sun-dried potato was 247 g water/100 g solids, while the solar-dried was reduced to 21.8 g water/100 g solids. It is obvious that there is a major improvement in drying rate with use of the solar-dryer.

Laboratory Simulation of Solar-Drying

From the solar-drying studies, average values for AFP (1.5 m/s) and inlet air temperature (65 °C) were determined. Since natural convection solar-drying (without external energy input) does not permit control of the drying process, it is necessary to investigate the effect that expected variations of the solar-drying process conditions would have on drying behavior. To accomplish this, a series of drying experiments were conducted using the laboratory configuration of the 2-way drying system where air speed and temperature can be controlled. Parameters investigated included: AFP (1.30 − 3.38 m/s), inlet air temperature (60–70 °C) and potato loading density (15–46 kg/m²).

Before examining the effect of the individual parameters, typical examples of the observed drying behavior are presented as moisture ratio (MR) versus time (Fig. 1) and log MR versus time (Fig. 2). The unconnected data points in Fig. 1 for 3 Lab-drying experiments (2 conducted at 60 °C) show typical shape for MR versus time. When plotted as log MR versus time (Fig. 2) there appears 2 straight line segments (if the final tailing is omitted), with a higher slope for the later segment. We have termed the point where the slope changes as the "turning point". The slope of the log MR versus time drying curve is generally considered to be related to the diffusivity of water inside the sample, provided that external mass transfer resistances are negligible. As external mass transfer resistances are not likely to be negligible for the natural convection situation, what is seen in Fig. 2 is certainly the result of more comples behavior, involving a changing balance between internal and external mass transfer resistances. For example, it can be noted that there is a sizable shrinkage of the sticks during drying, and that actual air speed, measured at regular intervals during drying, was significantly higher after the "turning point" than before. The nature of the "turning point" was not investigated in depth.

Other information obtained from the drying experiments, such as (1) the drying curve parameters (derived from the best fit quadratic equation for the MR versus time data) and (2) measured Moisture Ratio values at 5 h are also presented in Table 2. An analysis of the air conditions (based on a Mollier diagram using typical values observed during drying) indicate that for ambient air conditions of 24 °C and 50% RH, the inlet air to the dryer will have a RH of
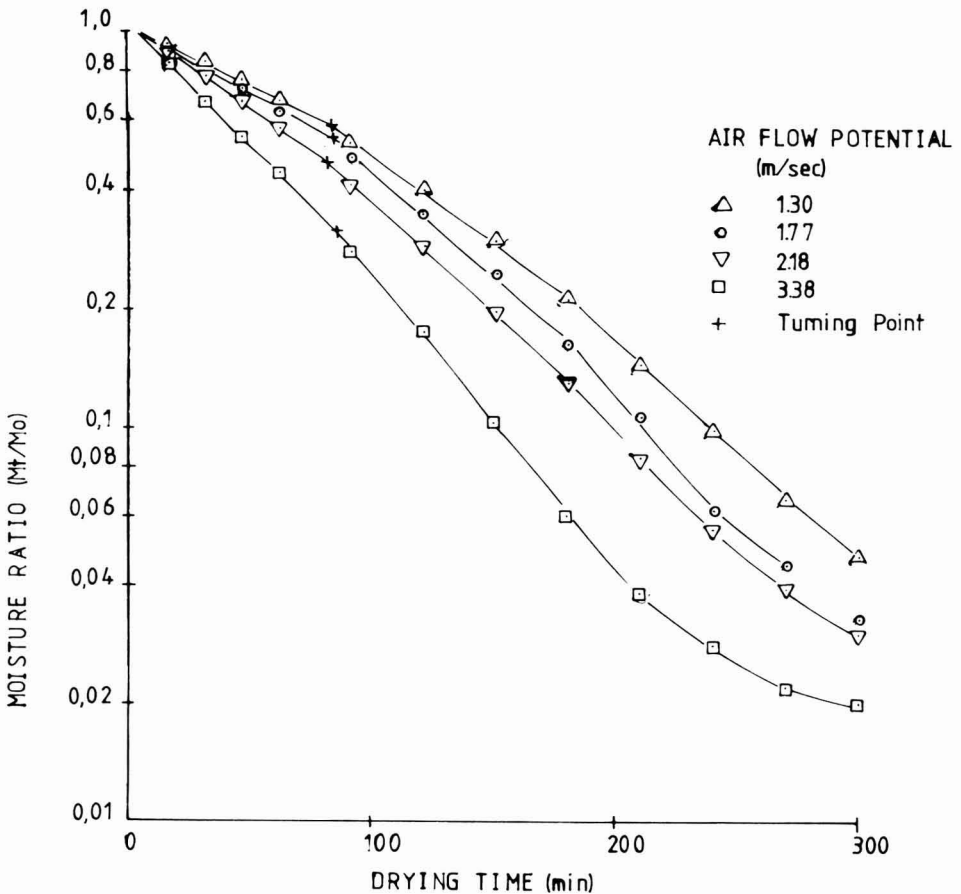
FIG. 2. DRYING CURVES (LOG MOISTURE RATIO BASIS) FOR LABORATORY
AIR DRYING OF POTATO STICKS AT 60 °C INLET AIR TEMPERATURE
AND 15 kg/m² LOADING

6% (which corresponds to a very low equilibrium moisture content for the potato). The average outlet air condition from the tower during drying was 40 °C and 40% RH. From the calculated water uptake of the air and the measured loss of water from the product, an average air speed in the loaded tower of at least 0.1–0.2 m/s is required. In essentially all experiments, the measured air speeds were somewhat above this value.

Table 2. Summarized drying data for laboratory simulations of natural convection solar-drying

Loading Density (kg/m²)	Dryer inlet Air (°C)	AFP (m/s)	TP^a (min)	Average actual air speed (m/s)		Drying Curve Parameters^b			R^c	Measured MR at 5 h
				before TP	after TP	A	B	C		
15	60	1.30	81	0.01	0.23	0.036	-0.373	1.004	**	0.05
	60	1.77	84	0.98	1.25	0.043	-0.409	1.000	**	0.03
	60	2.18	77	1.13	1.55	0.052	-0.444	0.981	*	0.03
	60	3.38	80	1.77	2.37	0.066	-0.497	0.925	0.992	0.02
	60^d	1.50	-	-	-	0.040	-0.391	1.002	-	-
	65	1.50	88	0.73	1.11	0.052	-0.445	0.981	*	0.04
	70	1.50	80	0.77	1.15	0.061	-0.482	0.970	0.987	0.03
30	60	1.30	104	< 0.01	< 0.01	0.016	-0.261	1.018	**	0.12
	60	1.77	103	0.10	0.63	0.019	-0.280	1.015	**	0.10
	60	2.18	106	0.83	1.18	0.027	-0.325	1.014	**	0.06
	60	3.38	92	1.38	1.95	0.050	-0.441	0.998	**	0.03
	60^d	1.50	-	-	-	0.017	-0.270	1.016	-	-
	65	1.50	104	< 0.01	0.77	0.028	-0.330	1.016	**	0.07
	70	1.50	92	0.57	0.93	0.039	-0.392	1.026	**	0.05
46	60	1.30	104	< 0.01	< 0.01	0.002	-0.158	1.008	**	0.27
	60	1.77	91	0.17	0.55	0.004	-0.182	1.009	**	0.22
	60	2.18	114	0.50	0.82	0.009	-0.221	1.011	**	0.16
	60	3.38	100	0.87	1.52	0.027	-0.326	1.024	**	0.08
	60^d	1.50	-	-	-	0.003	-0.170	1.008	-	-
	65	1.50	106	< 0.01	0.37	0.001	-0.167	1.017	**	0.23
	70	1.50	98	< 0.01	0.70	0.011	-0.240	1.021	**	0.12
Solar nr.3	69	-	70	0.75	1.00	0.053	-0.457	1.013	**	0.04
Solar nr.5	60	1.50	86	0.75	0.92	0.034	-0.361	1.007	**	0.05

a) TP = Turning point (see text)
b) MR = At²+BT+C   MR=MC(t)/MC(I)   MC=gH₂O/g solids
c) Correlation coefficient of measured and calculated MR values.   **=R≥0.999.   *=R≥0.998
d) Interpolated value

Laboratory Simulation Versus Solar-Drying

Comparisons of drying curves (Fig. 1) or drying curve parameters (Table 2) for solar-drying and the various laboratory drying experiments lead to the conclusion that most of the solar-drying experiments could be simulated with laboratory drying at either 60 °C and 2.18 m/s, or 65 °C and 1.5 m/s (the latter corresponding to the measured average conditions in solar-drying). In Fig. 3, it can be seen that drying rate versus moisture ratio for these 2 drying conditions are almost identical. The slow solar-drying (nr 5 on Fig. 1) was simulated nicely by a laboratory drying at 60 °C and 1.30 m/s (not shown on Fig. 1). Finally, it was noted that a laboratory drying at 60 °C and 1.77 m/s gave a good average value for all solar-drying experiments conducted, but unfortunately these conditions do not correspond to any single observed solar-drying experiment. On the basis of the predominence of experiments corresponding to 60 °C and 2.18 m/s, or 65 °C and 1.50 m/s, these conditions were utilized for the current simulations.

Effect of Air Flow Potential on Drying Behavior

It was observed that drying at higher AFP gives increased drying rates, which can result in significantly reduced drying time (Table 2; Fig. 2,3,4). At 60 °C, to achieve a MR = 0.04 required 312 min at AFP = 1.30 m/s versus only 210 min at AFP = 3.38 m/s. For a given product flow resistance, higher AFP gives a higher actual air speed (Table 2).

In Fig. 4, it is seen that the influence of AFP on drying rate decreases as the moisture ratio decreases (i.e., as drying approaches completion). This would indicate that external mass transport resistances are more important at the beginning of the process, and that the balance between internal and external moisture transport resistances changes as drying proceeds, such that AFP has less effect towards the end of the drying. While AFP is used to characterize air flow in the dryer, it is the actual air speed in the drying tower which actually influences the balance of mass transfer resistances.

It was observed that at a constant AFP, the actual air speed in the drying tower increased as drying proceeded. As an example, at 15 kg/m² and 1.5 m/s AFP, the actual air speed increased from 0.42 m/s at the start of drying to 1.13 m/s after 300 min. This increase in actual air speed is apparently associated with an observed shrinkage of the product during drying, which gives reduced resistance to air flow. Similar behavior was noted at the other loading densities. In Table 2, the difference in average actual air speeds, before and after the "turning point" also characterizes this behavior.
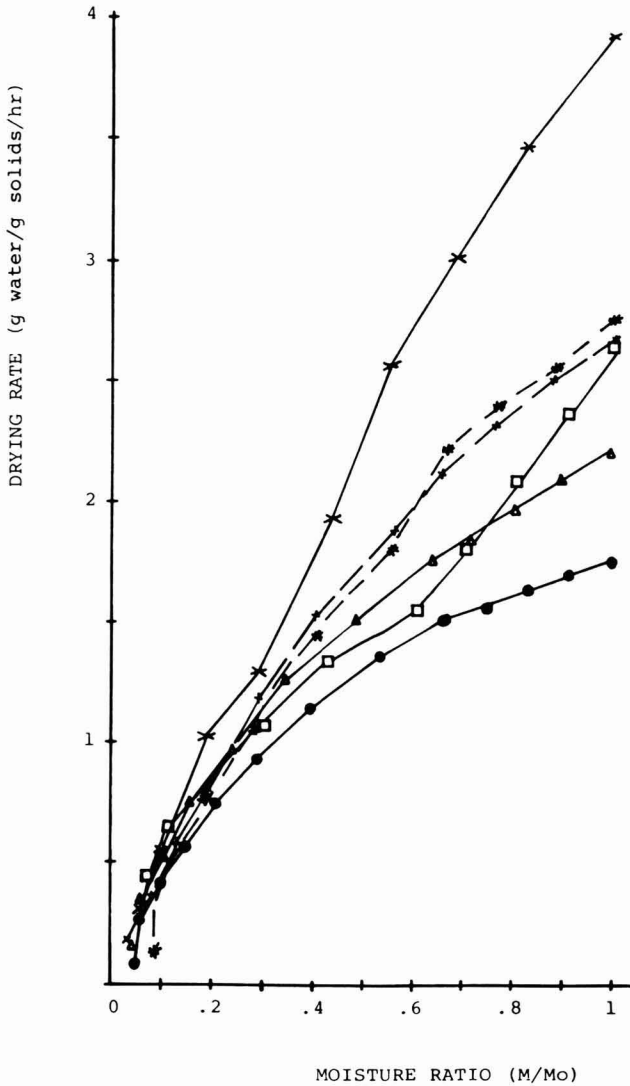
FIG. 3. DEPENDENCE OF DRYING RATE ON SAMPLE MOISTURE FOR A
TYPICAL SOLAR-DRYING AND SELECTED LABORATORY AIR-DRYINGS
X = 60 °C/3.38 m/s; ⋈= 60/2.18; △= 60/1.77; • = 60/1.30; * = 65/1.50; □ = Solar Drying 3.
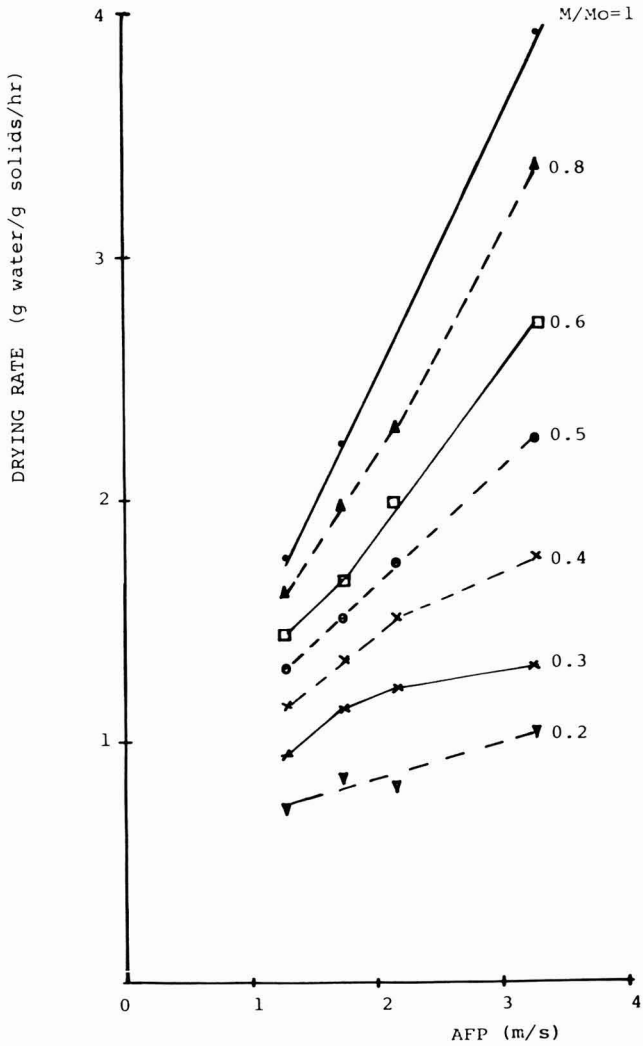
FIG. 4. DEPENDENCE OF DRYING RATE ON AIR FLOW POTENTIAL FOR
LABORATORY AIR DRYING OF POTATO STICKS, 60 °C INLET AIR
TEMPERATURE AND 15 kg/m² LOADING.

Effect of Sample Loading Density on Drying Behavior

The effect of sample loading density on drying behavior is given in Tables 2 and 3, and Fig. 5. Sample loading density was increased by placing 1, 2 or 3 trays, each with the same amount of sample, above each other in the tower. This arrangement allowed measurement of the drying effect at each level in the bed. Results in Table 2 are averages for the total bed, while in Table 3, drying behavior at different levels in the bed is given. It is seen that drying rate (parameter B in Tables 2 and 3) decreases markedly as loading density increases. For all situations, the thicker the bed, the lower the actual air speed through the bed, and the higher the average moisture ratio after 5 h, this behavior being more noticeable at the lower AFP values.

Table 3. Drying curve parameters for various tray loadings for laboratory drying at 60 °C and AFP = 2.18 m/s

Loading density hg/m^2	Tray[a] config.	Drying Curve Parameters[b]			R[c]	Measured MR at 5 h
		A	B	C		
15	1/1	0.052	-0.444	0.981	*	0.03
31	1/2	0.041	-0.393	0.993	**	0.04
	2/2	0.012	-0.257	1.034	*	0.09
	2 AV	0.027	-0.325	1.014	**	0.06
46	1/3	0.041	-0.390	0.997	**	0.05
	2/3	0.008	-0.226	1.035	0.997	0.14
	3/3	-0.020	-0.052	1.014	*	0.29
	3 AV	0.009	-0.221	1.011	**	0.16

a) Tray number (from bottom)/Total number of trays
   bed height and loading density: 1 tray = 3 cm; 15 kg/m²; 2 trays = 6 cm;
   31 kg/m²; 3 trays = 9 cm; 46 kg/m²
b) MR = $At^2 + Bt + C$
c) Correlation coefficient of measured and calculated MR values:
   ** R≥0.999
   * R≥0.998

In Table 3, it is noted that the first layer of the bed dries at about the same rate, independent of the total sample bed thickness. The second layer dries somewhat more slowly, while the third layer in the 46 kg/m² sample has a much slower drying rate. In Fig. 5 it is noted that drying rate of the first layer in a 3-layer sample decreases continuously as water is lost, while for the other layers, it is necessary for the first (lowest) layer to become dryer before hotter and drier air

passes to the upper layers of the bed to give the observed drying behavior. Extrapolations of drying behavior for the highest layer indicated that a moisture ratio of 0.04 would be attained after about 8 h.
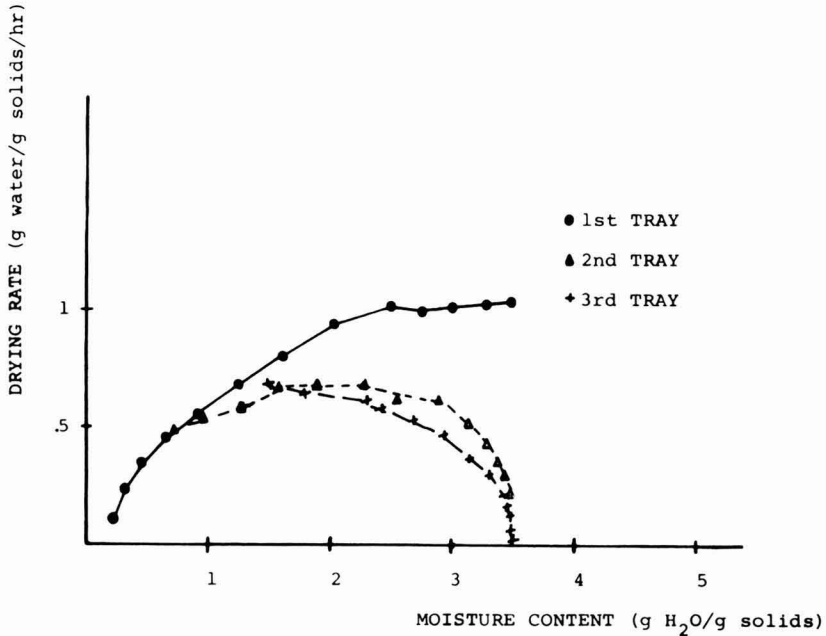

FIG. 5. DRYING RATES AT 3 LEVELS IN THE DRYING TOWER FOR 65 °C INLET AIR TEMPERATURE AND AIR FLOW POTENTIAL = 1.5 m/s
Total sample loading = 46 kg/m².

While drying time (to a MR of 0.04) increased with increasing loading density, the percent increase in drying time was found to be less than the percent increase in loading density. For example, when drying under simulated solar-drying conditions (65 °C, 1.5 m/s), a 100% increase in loading density (from 15 to 31 kg/m²) required only a 23% increase in drying time to MR = 0.04 (from 310 to 380 min). A 200% increase in loading density would require a 55% increase in drying time. Calculated total time to dry 6 trays, 1, 2 or 3 at a time, is 1860, 1140 and 960 min, respectively.

This behavior, which is similar to that found by Islam and Flink (1982a) suggests that dryer productivity can be increased by increasing the loading density, due to an improved utilization of the heat energy of the drying air. When drying rates, product quality and a 1 day drying procedure are all considered, it seems likely that a loading density in the vicinity of 30 kg/m² would be preferred.

Effect of Air Inlet Temperature of Drying Behavior

Results for drying at inlet air temperatures of 60, 65 and 70 °C are given in Table 2. For the comparison, data from 60 °C experiments at 1.30 and 1.77 m/s were interpolated to obtain AFP = 1.5 m/s. As expected, the samples dried more rapidly as the air temperature was increased. Temperature has a larger effect as the bed depth is increased, for reasons noted above.

Sensitivity of Drying Behavior to Expected Variation of Solar Drying Conditions

As solar-drying conditions are sensitive to many environmental factors, and thus will have "natural" variations, it is important to evaluate the sensitivity of the drying processs to expected variations of drying conditions. Based on the solar-drying studies, an inlet air temperature range of 65 ± 5 °C and AFP range of 1.50 ± 0.2 m/s were chosen. As a measure of drying process sensitivity to these ranges of drying parameters, the observed drying times to attain MR = 0.10 and M = 0.04 (Table 4) were used to calculate sensitivity of the drying time. Also given in Table 4 are the observed drying times for actual solar-drying experiments. It is noted that inlet air temperature is more critical for variation of drying time (actual variation ± 30 min) than the AFP (actual variation of ± 19 min).

Table 4. Time to achieve moisture ratios of 0.10 and 0.04

Drying time (min.)

AFP (m/s)	MR=0.10			MR=0.04		
	60[a]	65	70	60[a]	65	70
1.30	237			312		
1.50	227[b]	202	175	306[b]	300	252
1.77	215			285		
2.18	199			270		
3.38	150			210		
Solar Drying 3	189			290		
4	180			320		
5	243			320		
7	189			310		

a) Dryer inlet air temperature
b) Based on interpolated value

As control of inlet air temperature and AFP are difficult for a natural convection solar-dryer, it is necessary to be able to vary the duration of drying to ensure that the product achieves the desired level of dryness. To achieve this, a simple timer, which is coordinated (perhaps by color coding) with a thermometer measuring the inlet air temperature could signal the end of drying. This would require an additional piece of equipment and operator training, but should avoid the under- and over-drying problems associated with a fixed drying time.

ACKNOWLEDGMENT

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DEHYDRATION OF POTATO: 4. INFLUENCE OF PROCESS PARAMETERS ON ASCORBIC ACID RETENTION FOR NATURAL CONVECTION SOLAR DRYING CONDITIONS

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ABSTRACT

Ascorbic acid retention of potato sticks processed by natural convection solar drying was investigated. In some experiments, ascorbic acid contents were measured by both Titration and HPLC methods. Comparisons showed that for the dried product, the Titration method gives significantly higher ascorbic acid values, most probably due to formation of reducing substances during drying.

Ascorbic acid retentions for solar dried potato sticks was about 65%, while potato sticks dried under simulated natural convection solar drying conditions retained from 50 to 65%. Ascorbic acid retention was relatively constant for the variations of air temperature, air flow potential and sample loading density which would be expected for natural convection solar-drying.

Ascorbic acid loss during storage was very sensitive to product moisture content. Storage in air at room temperature in a twist-tie bag (m.c. ca 16.7%) gave reasonable ascorbic acid retention (49%), while the tighter-sealed Ziploc bag (m.c. Ca 18%) retained much less (16%). No brown color formation or mold growth was noted in the product. Degree of rehydration was not affected by package type, length of storage, or storage temperature. Rehydration during a normal cooking procedure was satisfactory to give a product organoleptically similar to cooked fresh potato.

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INTRODUCTION

Potato is an important natural dietary source of ascorbic acid (Vitamin C). As such, it is essential to insure availability of good quality potato products over the entire year, especially in developing countries where potato may be the primary Vitamin C source in the diet. Drying of potato is one means available to insure year-round availability of shelf-stable product. This is important in developing countries, as it simplifies distribution. The effects of dehydration and subsequent storage on retention of ascorbic acid are important considerations. Most recent studies on ascorbic acid losses in dehydrated potato have concerned mashed products. Granules obtained by both add-back and freeze-thaw processes were examined by Jadhav et al. (1975). The total loss in the add-back process was close to 75%, a value also found by Steele et al. (1976). Ascorbic acid loss for a freeze-thaw process was 37%. Augustin et al. (1979) reported ascorbic acid retentions for commercially produced dehydrated potato products to be; granules (45% retention), slices (40%) and dices (38%).

Maeda and Salunke (1981) investigated ascorbic acid retention following solar-drying of four vegetables (African spinach, cow pea, sweet potato and cassava leaves), using direct sunshine or enclosed, conventional solar-driers, with and without shade provisions. The maximum ascorbic acid retention was 24.4%, obtained when drying cow pea leaves in an enclosed solar-dryer with shade. Direct exposure to sun resulted in much lower retention (f. ex. 10.6% for cow pea leaves). Islam and Flink (1982b) observed ascorbic acid retentions of 45.5% and 61.3% for solar-dried and osmosed plus solar-dried potato products. (The osmosis was conducted for 3 h in a 45% sucrose/15% salt solution). Pinaga et al. (1983) reported ascorbic acid retentions of 40% for solar-dried green beans. A two-step process, similar to that Shaw (1981) used for drying potato, gave a similar ascorbic acid retention. Studies on storage stability of ascorbic acid in dried products have shown that ascorbic acid reactivity is very sensitive to water activity and storage temperature, as well as oxygen in the package (Karel and Nickerson 1964; Vojnovich and Pfeifer 1970; Labuza 1972; Lee and Labuza 1975; Kirk et al. 1977). It appears that to achieve best ascorbic acid stability, sample temperature, sample moisture content (water activity) and package oxygen level should be low.

While solar-drying has been suggested as a suitable means for drying potato (Smith et al. 1979; Shaw 1981; Islam and Flink 1982a,b; Shakya and Flink 1986), little information is available regarding ascorbic acid retention. The present investigation examines the influence of inlet air temperature, air speed and product loading density on ascorbic acid retention for potato sticks dried under natural convection solar drying conditions. Thereafter, several changes occurring in the dried product (ascorbic acid content, moisture content, rehydration behavior, visual appearance) during storage in simple packaging, deemed suitable for distribution on the Indian subcontinent, are evaluated.

MATERIALS AND METHODS

Raw Material

For the major part of this study, potato (Bintjee variety) was obtained from the Danish Potato Research Station (Vandel, Denmark). In a few later experiments, potato was obtained at the local supermarket.

The Drying Experiment

A description of the drying system and the procedures for conducting laboratory drying experiments simulating natural convection solar drying are given in Shakya and Flink (1986). In brief, potato was washed, hand-peeled, cut into sticks (12.5 x 6 x 40 mm), loaded randomly on wire mesh trays, and dried at 3 inlet air temperatures (60, 65 and 70 °C), 5 Air Flow Potential (AFP) values (1.30 to 3.38 m/s) and 3 product loading densities (15, 31 and 46 kg/m²). Air Flow Potential, (air speed in the empty tower) was set by regulating the voltage supplied to the fan in the heating unit. Air speed was measured using a mechanical annemometer. The actual air speeds in the loaded tower during drying at a fixed AFP setting will be lower, depending on sample airflow resistance (piece size and shape, loading density and degree of piece shrinkage). Moisture content was measured by vacuum oven drying (24 h, 80 °C, 65 mbar).

For the storage stability evaluation, peeled potato sticks (12.5 x 6 x 40 mm) were dried at 65 °C and an AFP of 1.5 m/s. Three drying runs (each of about 6 h) were required to obtain sufficient product. The three dried samples were mixed and equilibrated to get a product of uniform moisture content prior to packing in 3 polyethylene bags. One bag with a Ziploc seal was stored in a freezer (-20°) as a control. Two other bags, one with Ziploc seal and the other closed with a twist-tie, were stored at room temperature (19–24 °C). At 15 day intervals, samples were removed from each bag and analyzed for ascorbic acid content, moisture content, rehydration behavior and visual appearance (color and mold growth).

Determination of Ascorbic Acid

Ascorbic acid contents in fresh, dried, or partially dried potato was determined by a Titration method based on Tillman's reagent (Schanderl 1970; Rauscher et al. 1972). Fifty (50) grams of fresh sample (or the equivalent amount of dried or partially dried potato) was extracted with 2% (w/v) oxalic acid, and the extract then made to a final volume of 200 ml. Two 20 ml aliquoits of this extract were then titrated with 2,6-dichlorophenol indophenol. Standard solutions are prepared in accordance with the AOAC (1980) method.

For a part of this study, it was also possible to measure ascorbic acid using a HPLC method based on the procedure of Moledina and Flink (1982), but where 3% meta-phosphoric acid was used as the ascorbic acid extractant. The extract,

diluted to a concentration of 3–5 mg/100 ml, is filtered through a 0.5 μ Millipore filter, and thereafer passed through a Sep-pak C18 cartridge (Waters Assoc). A 20 μl aliquoit was injected onto a reverse phase ODS-C18 column (Altex Scientific). Ascorbic acid was detected at 254 nm, and its concentration calculated using the method of Moledina and Flink (1982).

The Rehydration Test

Twenty grams of dried potato was immersed in a beaker containing about 300 ml water at room temperature. At 30 min intervals, the potato was removed from the water and the surface lightly blotted with tissue paper. After the weight of potato was noted, it was placed in a measuring cylinder containing a known amount of deionized water. From the rise in total volume in the cylinder, the volume of potato was calculated. The potato was then returned to the rehydration water and the test continued for a total of 4 h.

RESULTS AND DISCUSSION

Ascorbic Acid Retention in Solar-Dried Potato

Solar-drying experiments were conducted on 2 typical sunny days (ambient temperature 18 and 19 °C). Inlet air temperature to the drying tower was 58–60 °C. Air speed in the loaded tower started at 0.33 m/s and increased to 1 m/s in the course of drying. Product loading density on both days was 15 kg/m². Ascorbic acid retentions (Titration method) in the dried product (300 min drying) these 2 days were found to be 72 and 76% of the level in the fresh potato. These samples were measured only by the Titration method. Based on later comparative measurements using Titration and HPLC methods (see next section), it would appear that ascorbic acid contents of dried potato determined by the Titration method are too high, and that a value of about 65% would probably be more correct. This ascorbic acid retention is still much higher than the 38–40% retention levels reported by Agustin *et al.* (1979a) for commercially dehydrated sliced and diced potato, demonstrating that solar-drying can give dried potato with satisfactory ascorbic acid levels.

Changes in Ascorbic Acid Content during Drying

In a series of experiments, ascorbic acid content of potato sticks (12.5 x 6 x 40 mm) was determined for various drying times covering a period of 15–300 min. Drying was conducted at 65 °C inlet air temperature and 1.5 m/s AFP, conditions typical for natural convection solar drying (Shakya and Flink 1986). Product loading density in all cases was 15 kg/m². Overall results are reported in Table 1.

Table 1. Change in ascorbic acid content during drying under solar drying conditions[a]

| Time (min) | Moisture content (g water/100g solids) | Ascorbic acid retention (% of original)[b] | |
		Titration	HPLC
0	345.1	100.0	100.0
15	304.2	100.0	100.0
30	265.8	95.6	92.1
45	229.9	90.4	88.1
60	196.7	90.4	88.1
90	142.7	83.9	80.5
150	68.1	62.3	60.6
300	14.6	75.5	64.8

[a] Air temperature = $65^{\circ}C$; AFP = 1.50 m/s; Loading density = $15kg/m^2$

[b] Original ascorbic acid content = 11.2 mg AA/100g fresh potato = 100% retention

Regarding titration and HPLC -methods, see text.

In this test series, ascorbic acid contents were analyzed by both the Titration and HPLC methods. In Table 1, it is noted that while for fresh potato and potato dried 15 min, the two methods gave the same ascorbic acid levels, drying for 30 to 150 min gave slightly higher (ca 3–5% higher) ascorbic acid contents for the Titration method. For potato dried 300 min, the Titration measured ascorbic acid content was ca. 18% higher than measured by the HPLC method. Steele *et al.* (1976) has noted that most standard methods of ascorbic acid analysis that are considered suitable for raw potato, were unreliable when used on dehydrated potato dried at 60 °C or higher, mainly due to an amino-sugar interactions which yielded products that analytically simulate ascorbic acid. As the drying in the present investigation was carried out at 65 °C, the higher ascorbic acid values found with the Titration method probably result from interference products formed in such amino acid-sugar reactions, which would first form towards the end of drying, when product temperature would be highest and moisture levels relatively low (i.e., reactant concentrations are increased). The HPLC method, being more specific for ascorbic acid, is considered to give the correct values. Figure 1 shows the relationship between drying time and ascorbic acid retention using the HPLC data. The Titration method data point for 300 min is included for comparison. Corresponding moisture contents are given in Table 1.
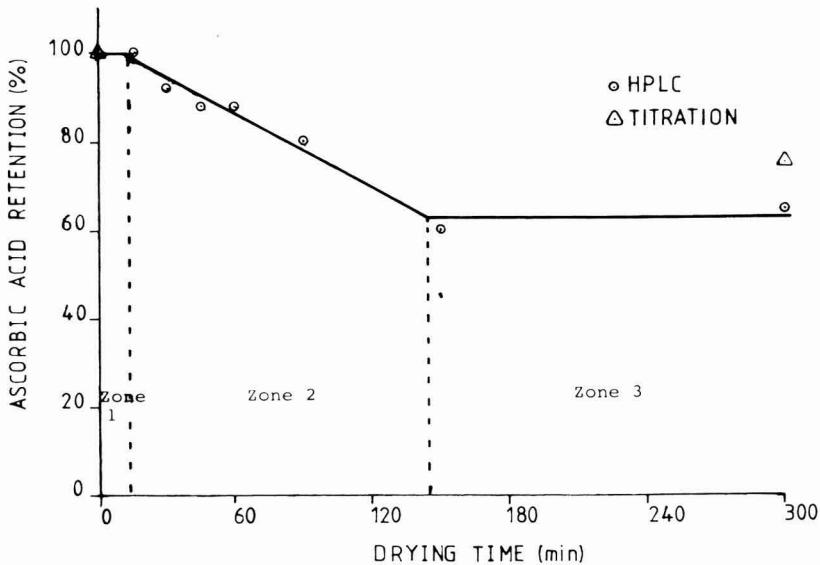
FIG. 1. ASCORBIC ACID RETENTION BEHAVIOR DURING A SIMULATED NATURAL
CONVECTION SOLAR DRYING OF POTATO STICKS

From Fig. 1, it is clear that no loss of ascorbic acid occurred in the first zone
(0–15 min) while moisture content decreases from 345 to 304 g water/100 g
solids. Essentially all of the observed ascorbic acid loss (38%) occurred in the
second zone (up to 150 min) as moisture content decreased from 304 to 68 g
water/100 g solids. In zone 2, the rate of ascorbic acid loss is 0.28%/min. At
drying times greater than 150 min (zone 3), no further changes in ascorbic acid
content were observed even though the moisture content of potato decreased
from 68 to 14.6 g water/100 g solids. As ascorbic acid loss appears to stop when
the product reaches a moisture content of 65–70 g water/100 g solids, process
parameters (piece size and shape, inlet air temperature, air speed, product
loading density, etc.) should be chosen to attain this moisture content level with
ascorbic acid retention maximized. The effect of these process parameters on
drying behavior has already been presented (Shakya and Flink 1986). In the re-
mainder of this paper, the effect of various process parameters on ascorbic acid
retention is given.

Effect of Air Speed on Ascorbic Acid Retention

From Table 2, it can be seen that air speed (AFP 1.30 to 3.38 m/s) has no definite effect on ascorbic acid retention, whereas the effect on the moisture level can be observed. As all moisture levels are below the 65–70 g/100 g level, further ascorbic acid loss in the final drying is not expected. Ascorbic acid retention is about 55% (measured to 65–70% by titration), independent of AFP. From the above, it would appear that while forced convection will give faster drying, ascorbic acid retention will not differ greatly from natural convection conditions.

Table 2. Effect of air flow potential on ascorbic acid retention in dry potato[a]

Air flow potential (m/s)	Ascorbic acid retention[b] (% of fresh)	Moisture content (g Water/100g solids)
1.30	59	23.6
1.50	70	20.1
1.77	62	17.8
2.18	67	15.9
3.38	67	10.9

a) Air temperature = 60°C; Loading density = 15 kg/m^2; Drying time = 300 min

b) Determined by titration method

Effect of Air Temperature on Ascorbic Acid Retention

The effect of dryer inlet temperature on ascorbic acid retention was investigated in 2 experimental series at an AFP equivalent to natural convection solar-drying. In the first series, the Titration analysis indicated that ascorbic acid retention increased with higher inlet air temperature (Table 3-Run 1). In the repeat series, conducted at a later date with stored potato having a lower ascorbic acid content (Table 3-Run 2), both Titration and HPLC analyses were used. This time HPLC gave a constant retention (52%) for all 3 drying temperatures, while the Titration method again indicated an increasing retention for higher inlet air temperature. As noted earlier, this difference is due to formation of browning products, the increasing difference between Titration and HPLC values as drying temperature is raised being indicative of an increased production of interfering substances with increased temperature, probably due to increased formation

Table 3.. Effect of air temperature on ascorbic acid retention in dry potato[a]

Inlet air temperature, °C	Run # 1[b]		Run # 2[c]		
	Ascorbic acid retention (% of fresh) Titration	Moisture content (g water/ 100g solids)	Ascorbic acid retention (% of fresh) Titration	HPLC	Moisture content (g water/ 100g solids
60	70	20	60	52	26
65	74	15	65	52	17
70	84	11	75	52	14

a) AFP = 1.5 m/s; Loading density = 15 kg/m^2; Drying time = 300 min

b) Initial moisture = 340-345g water/100g solids; Initial ascorbic acid = 13.2 mg/100g fresh potato

c) Initial moisture = 390-420g water/100g solids; Initial ascorbic acid = 7.2 mg/100g fresh potato

of browning products at the higher temperature. While the % retentions by the Titration method differed for the 2 test series, the same trend in retention is noted. The differences noted could be due to actual differences in ascorbic acid retention, or to different extents of production of interferring substances.

Moisture contents after 300 min of drying were lower with increasing air temperature, as expected. Allowing the inlet air temperature in a natural convection solar-dryer to rise as high as possible will give a higher drying rate (higher throughput). While HPLC measurements indicate no decrease in ascorbic acid retention as drying temperature is raised from 60 to 70 °C, the organoleptic and nutritional consequences of the increased browning needs to be investigated before temperature limits can be suggested.

Effect of Loading Density on Ascorbic Acid Retention

Loading density was increased by stacking additional trays in the drying tower. Ascorbic acid retention was however only investigated for the lowest tray of each loading. From Table 4, it is seen that ascorbic acid retention in the first tray is independent of the total loading in the tower, whereas the moisture content is higher for the higher loading density. This is undoubtedly due to the fact that inlet air temperature in all 3 cases is the same while actual air speed on average is lower for increased loading density.

Table 4. Effect of loading density on ascorbic acid retention in dry potato[a]

Number of trays in drying tower	Loading density, (kg/m^2)	Ascorbic acid retention[b] (% of fresh)	Moisture content[c] (g water/100g solids)	Range of actual air speeds noted during drying (m/s)
1	15	67	15.9	0.9 – 1.7
2	31	65	22.4	0.7 – 1.3
3	46	66	26.2	0.3 – 1.0

a) Air temperature = 60°C; AFP = 2.18 m/s; Drying time = 300 min

b) Determined by the titration method. Retention for first tray in each case

c) Moisture content of the first tray in each case

Assuming that the upper trays have a similar ascorbic acid retention behavior, the recommended use of increased loading densities for improved throughput rates (Shakya and Flink 1986) should have limited consequences for overall ascorbic acid retention.

PRODUCT STABILITY DURING STORAGE

Room temperature storability in simple packages (Ziploc sealed or twist-tie sealed bags) which could be considered for use on the Indian subcontinent was examined for 90 days. A sample stored at $-20\,°C$ was used as control. Results obtained for ascorbic acid content, moisture content and degree of rehydration are given in Table 5.

Moisture content. Incomplete drying of the sample gave a moisture content just prior to packing which was significantly higher (23.9 g water/100 g solids) than the desired 15 g/100 g. In the first 15 days of storage, moisture content dropped to 16–18 g/100 g, after which it remained relatively constant (range of 16–19 g water/100 g solids) for the remaining storage time. The Ziploc bags had higher moisture contents throughout the storage period, indicating that the Ziploc seal was more effective in hindering moisture migration.

Ascorbic Acid Retention. Ascorbic acid retention in the dried product was only measured by the Titration method. It is possible to use the differences between HPLC and Titration measurements of ascorbic acid in dried potato (calculated to be ca 1.2 mg AA/100 g fresh potato from the data in Table 1 for 300 min) to correct the Titration ascorbic acid contents given in Table 5. The corrected ascorbic acid contents are also given in Table 5.

Ascorbic acid content decreased rapidly with increasing storage time for both packages. The decrease is particularly high for the first 15 days, where moisture content is higher than desired. This behavior is in agreement with Vojnovich and Pfeifer (1970) and Lee and Labuza (1975), who reported sizable losses for ascorbic acid after short storage periods at moisture levels present in intermediate moisture foods.

As the large ascorbic acid decrease in the first 15 days is due to the abnormally high moisture content in that period, a better measure of storage stability is obtained by examining the period, day 15–90. Over this period, the average moisture contents were 17.9(Ziploc) and 16.7 (twist-tie) g water/100 g solids, while the corresponding ascorbic acid retentions (based on corrected Titration values) were 16% and 49%, respectively. It appears that the small difference noted in moisture content between the two packages gives a significantly higher ascorbic acid retention, indicating that a much better ascorbic acid retention

Table 5. Stability of the dried potato during storage in plastic bags[a]

Storage period (days)	Ascorbic acid content (mg/100g fresh potato)[b]			Moisture content (g water/100g solids)			Rehydration[c] (% of fresh)		
	Z(-20)	Z(RT)	TT(RT)	Z(-20)	Z(RT)	TT(RT)	Z(-20)	Z(RT)	TT(RT)
0	7.4 (6.2)	7.4 (6.2)	7.4 (6.2)	23.9	23.9	23.9	60	60	60
15	5.9 (4.7)	3.1 (1.9)	4.7 (3.5)	17.8	17.3	16.1	55	56	57
30	5.7 (4.1)	2.9 (1.7)	4.1 (2.9)	18.3	17.6	17.2	58	58	58
45	-	-	-	18.2	17.5	16.8	58	55	57
60	5.2 (4.0)	1.05 (0.3)	3.2 (2.0)	17.3	17.1	15.9	58	56	59
75	-	-	-	20.2	19.3	17.5	59	58	57
90	5.4 (4.2)	1.5 (0.3)	2.9 (1.7)	19.6	18.6	16.8	57	56	57

a) Z(-20) = Ziplock seal stored at -20°C; Z(RT) = Ziplock seal stored at room temperature (19-24°C);
   TT(RT) = Twist-tie stored at room temperature (19-24°C)

b) Measured by Titration method. Corrected values are in ( ). See text for correction method

c) Based on weight after 4 h

could have been obtained, had the sample had the moisture level originally chosen for safe storage (15 g water/100 g solids). As expected, ascorbic acid retention (89%) was much higher for the package stored at −20 °C.

Rehydration Behavior. Degree of rehydration was determined on the basis of both weight and volume (as g/g solids or cm³/g solids). In Fig. 2, it is shown that the degree of rehydration increased continuously with increasing rehydration time (0–240 min), though the rate of increase was much higher for the first 60 min, than for the last 60 min. Rehydration data in Table 5 are based on weight change after 4 h. It is apparent that storage time and storage condition (package type or temperature) have no effect on degree of rehydration .
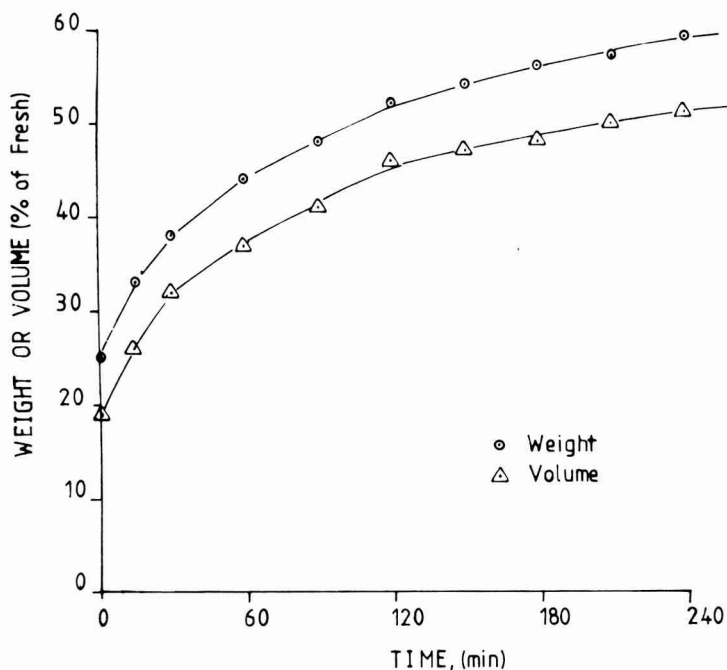
FIG. 2. REHYDRATION BEHAVIOR OF DEHYDRATED POTATO AFTER 2 MONTHS
STORAGE AT ROOM TEMPERATURE IN A TWIST-TIE BAG

In an informal cooking test, dried potato (without prior rehydration) was cooked by placing directly in boiling water for about 10 min. These cooked, dehydrated potatoes were very similar organolepically to cooked fresh potatoes. Smith *et al.* (1979) have also reported that room temperature rehydration of solar-dried potato cubes was equal to that observed for commercially dried product. It has also been shown that it is not necessary to rehydrate the dried potato before cooking.

Visual Changes. Throughout the storage period, the dried potatoes were examined visually for browning or mold growth. Visual appearance remained good for all storage conditions, indicating that a moisture content of 16–19 g water/100 g solids is adequate for hindering browning and mold growth, even though this corresponds to a water activity range of 0.75–0.82, which is somewhat higher than originally desired (Aw = 0.7).

CONCLUSIONS

Within the limits of variations of process conditions expected for natural convection solar-drying it appears that none of the process parameters had a major impact on the ascorbic acid retention levels.

It was noted that the Titration method is not suitable for determining ascorbic acid contents in potato dried at high temperature. To obtain a correct value requires a more specific test, such as by HPLC (or perhaps enzyme) methods.

It was possible to solar-dry potato sticks with a relatively high level of retained ascorbic acid (50–65%). Ascorbic acid loss in potato during drying apparently ceases when the moisture content comes under 65–70 g water/100 g solids.

Significant ascorbic acid loss during storage is noted at moisture levels of 16 g water/100 g solids. The observed sensitivity of ascorbic acid loss to moisture content emphasizes that careful control of final moisture level in drying will be required, even when operating a solar-dryer at the village-use level.

ACKNOWLEDGMENTS

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BOOK REVIEW

Advances in Catering Technology – 3. George Glew, (ed.) 1985. Elsevier Applied Science Publishers Limited. pp. 293. $52.50.

This book contains twenty-five chapters that are based on papers presented at the Third International Symposium on Catering Systems and Design, held September 10–13, 1984 in Harrogate, England. The Symposium was organized by the Editor, George Glew, who is Professor and Head of the Department of Catering Studies and Director of the Hotel and Catering Research Centre, Huddersfield Polytechnic, Huddersfield, UK.

The book is divided into five parts: The Catering Industry and Some Approaches to Planning, Improvement of Cooking and Meal Production Techniques, Waste Control, Cleaning and Sanitation and Management of Technology. A major strength of the book are the chapters on processes used in foodservice such as cooking by conduction, water, oil, air/vapor mixtures and infrared and microwave radiation. Additionally the chapters on understanding what happens in catering equipment during cooking and the need for control over cooking to maximize quality would be of use to foodservice managers.

Food processing engineers and food scientists may find this book of value in increasing their awareness of catering technology. The book is written for the catering or foodservice professional, however foodservice equipment and facility consultants would find it a useful reference. Educators in foodservice programs could use the book to supplement their lectures or as a text in a graduate class.

The book contains a table of contents with the names and addresses of the authors of each chapter, an index, a preface and the recommendations for research by the delegates at the symposium. Chapters are well-organized and contain tables and figures. Many chapters have summaries or references.

The quality of the book is good; however, the level of the material appears to vary among chapters. Nonetheless, for someone with interests in catering technology, this book is a unique source of current information.

M. EILEEN MATTHEWS

F
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ZABORSKY, O. 1973. *Immobilized Enzymes*, pp. 28–46, CRC Press, Cleveland, Ohio.

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