Mass Balances in Porous Foods Impregnation

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ABSTRACT: A simple model based on mass balance equations is proposed for prediction of the final mass and composition of products subjected to vacuum impregnation. It was applied to some tropical fruits in a fruit-sucrose solution system. The phenomenon can be described in terms of volumetric fraction of impregnating solution as the basic modeling parameter, instead of effective porosity. To use the equations of the model, only routine laboratory equipment and simple experiments are required. Prediction of the final weight of impregnated fruit was accomplished with an average absolute error of 2 to 3%, while in final composition of the fruit (total solids), it was 5.7 %.

Keywords: vacuum impregnation, fruit processing, hydrodynamic mechanism, mass balances

Introduction

POROUS MICROSTRUCTURE OF FOOD MATRIXES IS AN IMPORtant property often neglected by food processors who usually are not aware that it can be successfully taken advantage of in food preservation operations and product development. Foods exhibiting this characteristic can be impregnated; that is, their pores can be filled with a suitable solution, introducing solvents and solutes of choice into their porous spaces that are originally occupied by a certain amount of occluded gas, by means of capillary action or by the combined effect of capillary action and pressure gradients imposed on the system. A way to accomplish this is to apply vacuum to a product while immersed in the liquid and then reestablish the atmospheric pressure. This causes the gas to be expelled from the pores, which is replaced by the entering liquid in a vacuum impregnation process. A proper formulation of the impregnation liquid allows convenient compositional changes in the solid matrix that may result in quality and stability enhancement of final products, without submitting the food structure to the stress due to long exposure to gradients in solute concentration.

The use of vacuum impregnation techniques by apple processors for firming the tissue and improving the quality of canned and frozen apples dates back to the fifties. The use of aqueous solutions that may contain sugar, calcium salts, organic acids, colors, flavors, sulphurous salts, and a combination of them, has been reported (Hoover and Miller 1975). Other applications of vacuum impregnation in food processing are the removal of trapped gases and whey in the cheese curd (Reinbold and others 1993), jam processing (Shi and others 1996), salting of fish (del Saz and others 1994) and cheese (Chiralt and Fito 1996), addition of cryoprotectant agents to apple (Martínez-Monzó and others 1997), and incorporation of antibrowning compounds to cut fruits and vegetables (Sapers and others 1990).

A major drawback of earlier works was the lack of a mathematical approach to describe the phenomenon of vacuum impregnation. Fito and co-workers have proposed a general model that describes the so called hydrodynamic mechanism (HDM), later extended to include deformation-relaxation phenomena (DRP) in the solid matrix, to explain the mass transfer kinetics produced by driving forces due to pressure gradients (Fito and others 1993; Fito 1994; Fito and Pastor 1994; Fito and Chiralt 1995; Fito and Chiralt 1996).

This approach requires the proper determination of basic modeling parameters, such as effective porosity (Ee) and relative sample deformation (γ), which are determined by laborious measurement (Fito and others 1996). The parameters depend on the food and its geometry, and are affected by operation variables such as vacuum pressure and temperature. However, based on some of the mathematical definitions proposed by Fito and Pastor (1994), it is possible to address the impregnation issue in a rather practical way, requiring only routine laboratory equipment and simple experimental techniques.

Fruits in general have microporous structure, and can be vacuum impregnated rapidly through HDM without requiring the establishment of the initial concentration gradient characteristic of time-consuming Fickian diffusion. This could be appropriate in the development of new fruit products as minimally processed or in improved pretreatments for traditional preservation methods as canning, freezing and drying.

In this work, the vacuum impregnation of some tropical fruits with a sucrose syrup was studied using the volumetric fraction (X) of the impregnated products instead of effective porosity; and prediction of final mass and composition of products was accomplished using mass balance equations derived from the approach of Fito and Pastor (1994).

Materials and Methods

Mathematical modeling

The major mass inputs in vacuum impregnation of a solid matrix with a liquid can be described as: FCi_F (F: mass of fresh sample), SCi_S (S: mass of impregnating solution penetrating sample by the hydrodynamic mechanism); ACi_A (A: mass of adhered and capillary action driven solution), and the output is MCi_M (M: mass of vacuum impregnated sample), while Ci is the concentration, percentage or mass fraction of a component i. Minor possible transfers that may occur as a result of the vacuum pulse, such as air purging, evaporation or dragging of internal liquid, or water-solute transfers in the case of any osmotic effect considering a short time vacuum pulse (Fito and Chiralt 1996) are neglected. The volumetric fraction of impregnated sample (X) which is the fraction of porous structure occupied by the liquid impregnating solution, can be estimated using Eq (1) (adapted from Fito and others 1996):

$$X = [\,(M - A) - F\,] / \rho_S V \tag{1}$$

In Eq (1) the final mass M must be corrected for the adhered liquid A in order to estimate the actual penetrated liquid by vacuum effect. The theoretical prediction of the mass A is not an easy task, since it is affected by variables like surface tension, density and viscosity of the impregnating liquid, size, geometry, external area and surface texture of sample, solid-liquid contact time, speed of removal of sample from liquid, time and procedure of draining, room relative humidity conditions, etc. (Sakiadis 1995).

The term A represents the amount of impregnating solution that adheres to the sample by combined effect of surface adherence and capillary action at atmospheric conditions, and by definition is calculated using Eq (2):

$$A = M_{atm} - F \qquad (2)$$

Substituting Eq (2) in Eq (1), and considering that the volume of fresh sample is the relationship between F and ρ_F, Eq (3) is obtained:

$$X = [(M - M_{atm})/F]\ (\rho_F/\rho_S) \qquad (3)$$

The weight of liquid due to adherence and capillary action can be expressed in terms of a convenient ratio R:

$$R = M_{atm}/F \qquad (4)$$

The following global and component mass balances can be stated:

$$M = F + S + A \qquad (5)$$

$$Ci_M = (Ci_F\, F + Ci_S\, S + Ci_A\, A)/M \qquad (6)$$

where S can be calculated using Eq (7):

$$S = V X \rho_S \qquad (7)$$

A simple expression (Eq. 8) that allows calculation of the final mass M can be obtained by combining Eqs (2), (4), (5) and (7):

$$M = R F + V X \rho_S \qquad (8)$$

Eq (8) is obtained neglecting any volume change due to DRP on the assumption that the initial volume of sample (V) remains constant as in the early approach of Fito (1994). This author analyzed HDM in fruits applying an equation without considering DRP, which is equivalent to assuming that the samples have a constant volume throughout the process. Refinements of equations based on coupling of HDM and DRP show that volume deformation in most fruits is within the range of ± 10% (Fito and Chiralt 1995, 2000), low enough to support the constant volume assumption.

Knowing that the volume of the fresh product is the relation between F and ρ_F, Eq. (8) becomes:

$$M = F\ [R + X\ (\rho_S/\rho_F)] \qquad (9)$$

Since the adhered solution is none other than the impregnating solution, then

$$Ci_S = Ci_A \qquad (10)$$

Considering Eq. 10 and introducing the global mass balance (Eq. 5) into the component mass balance (Eq. 6), it is possible to obtain Eq. (11) for estimation of the final concentration (Ci) of any component i (water, total solids, major solutes, additives, preservatives, even microorganisms) in the pulsed vacuum impregnated sample:

$$Ci_M = (Ci_F - Ci_S)\ (F/M) + Ci_S \qquad (11)$$

If the term A is included in S, equation 1 becomes:

$$X = (M - F) / \rho_S V \qquad (12)$$

that can be simplified to:

$$X = [\rho_F/\rho_S]\ [(M/F) - 1] \qquad (13)$$

The pulsed vacuum impregnation process can be scaled up using Eqs. 9 and 11, requiring only as input data, the mass of fresh product (F), densities of both fresh sample (ρ_F) and impregnating solution (ρ_S), the concentration of any pertinent component in the fresh sample (Ci_F), the concentration of the same component in the impregnating solution (Ci_S), and the values of the parameters X (Eq. 13), and R (Eq. 4). The values of X and R can be obtained experimentally by the desiccator technique described by Fito and Pastor (1994). Conditions to be fulfilled in a pilot or industrial level must be simulated at lab level with respect to pressure and duration of the vacuum pulse, composition and temperature of the impregnation solution, geometry, size, pretreatments, procedure, and time of draining, and composition and physiological state of samples.

Fruits characterization

Fruits used in this work were commercially important Venezuelan varieties of papaya (*Carica papaya* L. tipo Cubana), pineapple (*Ananas comosus* L. var. Española Roja) from Lara State, melon (*Cucumis melo* var. Edisto) from Falcon State, banana (*Musa acuminata* tipo Pineo Gigante) and apricot (*Prunus persica* var. Pavis Amarillo) from Aragua State, as solids, and a 65 °Brix sucrose solution as the impregnation liquid. The impregnating solution was prepared with food grade sucrose due to its taste compatibility, low cost, and common use in syrups related to fruit processing. Mature-green sound fruits free of bruises and rots were selected, hand washed, peeled and cut, and characterized by measuring the following parameters using ten replicates: a_w with an Aqualab CX-2 (Decagon Devices, Inc., Pullman, Wash., U.S.A.), total solids by vacuum oven technique (AOAC 1990), pH by potentiometry, acidity by titration, soluble solids by refractometry as °Brix (AOAC 1990) and density (ρ_F) by liquid displacement. Fruits were cut into cubes (papaya), bricks (pineapple), finite cylinders (melon and banana), or quarters (apricot), using calibrated stainless steel borers and knives, in order to standardize shapes and sizes. Characteristic dimensions were measured with a digital caliper. Melon cylinders were cut radially, and banana's transversely.

After cutting, pieces of each fruit were maintained in controlled relative humidity atmospheres provided by isotonic sucrose solutions in closed desiccators to minimize weight losses prior to determinations of moisture and density. Isotonic solutions were prepared using sucrose and a Raoult based equation (Eq. 14) was applied in order to facilitate its preparation in terms of soluble solids. Isotonicity of solution was checked with the water activity meter.

Table 1—Characterization[a] of five tropical fruits for impregnation studies

Fruit	Geometry	Dimensions $\times 10^2$ m	Initial total solids (%)	a_{wF} [b]	pH	acidity (% citric acid)	°Brix[b]	ρ_F [b] (Kg/m³)
Melon	Finite cylinders	Length: 3.0 ± 0.3 Diameter: 2.3 ± 0.1	7.2 ± 1.3	0.99 ± 0.00[c]	6.5 ± 0.3	0.14 ± 0.03	6.8 ± 0.8	891 ± 27
Pineapple	Bricks	Length: 2.3 ± 0.0 Width: 1.8 ± 0.0 Thickness: 1.1 ± 0.1	15.2 ± 2.5	0.98 ± 0.00[c]	3.5 ± 0.3	0.76 ± 0.18	13.3 ± 1.9	898 ± 54
Papaya	Cubes	Side: 1.3 ± 0.0	13.9 ± 1.8	0.98 ± 0.00[c]	5.7 ± 0.0	0.68 ± 0.01	8.4 ± 0.1	910 ± 22
Apricot	Quarters	Thickness: 1.3 ± 0.1	17.1 ± 1.6	0.98 ± 0.00[c]	3.4 ± 0.4	1.36 ± 0.18	13.3 ± 1.4	880 ± 40
Banana	Finite cylinders	Length: 1.0 ± 0.0 Diameter: 1.9 ± 0.0	27.9 ± 1.1	0.97 ± 0.00[c]	4.8 ± 0.2	0.12 ± 0.01	24.0 ± 1.0	887 ± 42

[a]Average from ten replicates
[b]At 25 °C
[c]Standard deviations < 0.004

$$°Brix_{sucrose} = 100\,(1 - a_{wF})\,/\,(1 - 0.947\,a_{wF}) \quad (14)$$

Experimental determination of R and X

In order to obtain R, ten replicates of fruit pieces (values of F) were immersed in a 65 °Brix sucrose syrup at 35 °C for 7 min and drained over plastic mesh for 5 min. After this, the pieces were weighed (M_{atm}).

For determination of X, another ten replicates of fruit pieces were subjected to impregnation by the HDM in vacuum desiccators immersed in a thermostatic water bath at 35 °C. The density of the 65 °Brix sucrose solution (ρ_S = 1129.6 Kg/m³) was interpolated from AOAC (1990) data. Five different vacuum pressures (P_p) were applied in two-minute pulses with 5 min of residence time in solution after restoring the atmospheric pressure (these times account for the 7 min used for the above mentioned determination of M_{atm}) and 5 min of drainage over plastic mesh. The short times of vacuum pulses and atmospheric pressure restoration were selected in order to minimize osmotic effect during impregnation. Preliminary work showed that such times were sufficient to reach equilibrium in the vacuum and the impregnation steps. This observation agrees with that of Fito and others (1996) and Chiralt and others (1999) who demonstrated that equilibrium is rapidly reached even for very viscous impregnating solutions. After each pulse, the fruit pieces were individually weighed (M) and their total solids content experimentally determined. Parameters R and X were calculated using Eqs. (4) and (3), respectively, and average values were obtained.

Model application

An attempt was made to demonstrate the applicability of the model using total solids as the compositional parameter. Thus, for each fruit investigated, a batch of around 0.5 Kg of standardized cut pieces was vacuum impregnated at 35 °C, using a 65 °Brix sucrose solution, and applying a two-minute pulse at one of the five pressures investigated for each fruit. After impregnation, a residence time of 5 min for restoring atmospheric pressure was allowed followed by draining for other 5 min. Each batch was weighed and its total solids content was determined. These values were compared to theoretical values of M and Ci_M obtained from Eqs. (9) and (11) respectively.

Results and Discussion

Table 1 contains the results of characterization of the five tropical fruits used for the impregnation studies. Table 2 contains values of parameters R and X for each application of the two-minute vacuum pulse, using a 65 °Brix sucrose syrup as impregnating liquid. As mentioned before, the parameter R is a measure of the superficially adhered and capillary driven liquid, and even though it depends on several variables, the specific area of samples σ (Eq. 15) seems to exert a proportional effect on R.

$$\sigma = A_{ext}/F \quad (15)$$

For example, papaya pieces which exhibited the highest σ value (0.50 m²/Kg) had the highest R value of all the fruits

Table 2—Values of R and X at different vacuum pressures, for five tropical fruits[a]

Fruit	R[b]	$P_p \times 10^{-4}$ Pa	X[b]
Melon	1.019 ± 0.007	0.41	0.113[c] ± 0.014
		1.74	0.111[c] ± 0.009
		3.07	0.138[c] ± 0.024
		4.40	0.062[d] ± 0.004
		5.81	0.062[d] ± 0.011
Pineapple	1.038 ± 0.005	1.74	0.024[c] ± 0.009
		3.74	0.017[d] ± 0.006
		5.07	0.010[d] ± 0.003
		5.73	0.011[d] ± 0.008
		6.40	0.014[d] ± 0.006
Papaya	1.042 ± 0.005	1.57	0.023[c] ± 0.004
		2.90	0.030[c] ± 0.004
		4.23	0.031[c] ± 0.004
		5.56	0.036[c] ± 0.003
		6.89	0.044[d] ± 0.004
Apricot	1.017 ± 0.005	1.30	0.009[c] ± 0.003
		2.63	0.011[c] ± 0.005
		3.96	0.014[c] ± 0.007
		4.63	0.020[d] ± 0.008
		5.29	0.022[d] ± 0.013
Banana	1.027 ± 0.018	1.70	0.022[c] ± 0.038
		3.03	0.044[d] ± 0.015
		4.36	0.030[e] ± 0.015
		5.69	0.032[e] ± 0.009
		7.02	0.037[e] ± 0.019

[a]Atmospheric pressure: 8.87 10^4 to 8.91 10^4 Pa
[b]Average from 10 replicates
[c,d,e]Similar letters mean non-significant differences ($p > 0.05$)

Table 3—Comparison of experimental and theoretical values of M and Cs_M for five tropical fruits submitted to vacuum impregnation

Fruit	Cs_F	P_p x 10^{-4} Pa	X (Table 2)	F (Kg)	M experimental (Kg)	M Eq (9) (Kg)	Error [a] (%)	Cs_M experimental	Cs_M Eq 11	Error[a] (%)
Melon	0.072	1.74	0.111	0.464	0.574	0.538	6.3	0.161	0.152	5.6
Pineapple	0.152	5.07	0.010	0.441	0.462	0463	-0.2	0.189	0.176	6.9
Papaya	0.139	6.89	0.044	0.427	0.455	0.468	-2.9	0.168	0.184	-9.5
Apricot	0.171	3.96	0.014	0.460	0.484	0.476	1.7	0.184	0.187	-1.6
Banana	0.280	5.69	0.032	0.440	0.471	0.469	0.4	0.318	0.303	4.7

Cs: Total solids fraction
[a]100(experimental value-theoretical value)/experimental value

studied: 4.2 % (Table 2). On the other hand, the simple syrup immersion of fresh pieces of melon increased its weight by only 1.9 %, with a correspondingly lower σ value of 0.27 m^2/Kg. Therefore, for small fruit pieces, the contribution of adhered solution to mass increment of fruit can be more important than HDM.

The values of X reported in this work indicate that of all the fruits studied, melon exhibited the highest impregnated volumetric fraction, with a maximum value of 13.8% when a vacuum pressure pulse of 3.07 10^4 Pa was used. On the other hand, pineapple and peach are the more difficult to impregnate fruits since their maximum impregnated volumetric fractions were 2.4% (at 1.74 10^4 Pa) and 2.16% (at 5.29 Pa), respectively.

Applied vacuum had a significant effect on X values for each of the fruits considered in this work ($p > 0.05$). In the case of melon and pineapple, impregnation increased when vacuum at reduced absolute pressures was used. These results indicate that these fruits have elastic structural matrixes that withstand the tension generated by the variations in the pressures applied. Banana had a maximum X value of 4.4 % at 3.03 10^4 Pa, stabilizing at values around 3% for higher absolute pressures, while at 1.70 10^4 Pa (the more intense pulse), the value of X fell to 2.2%. This is a reflection of the relaxation phenomena under such extreme conditions. Papaya and peach seem to have structures less resistant to mechanical stress because the highest values of X were found at the highest absolute pressures. For this reason, the impregnation of these fruits should be made using pulses under moderate vacuum in order to take advantage of their intercellular spaces.

These results reflect the complexity of the hydrodynamic penetration mechanism in a fruit, coupled with the relaxation-deformation phenomenon, which depends on multiple variables like size, distribution and form of the pores, effective porosity, viscoelastic properties of the fruits, size and form of the fruit piece, variety and degree of maturity, and intensity of the pulse applied (Fito and others 1996).

In general, the values of X found in this investigation were lower than the volumetric fractions reported for other fruits by other authors (Chiralt and others 1999; Salvatori and others 1998; Fito 1994; Fito and Chiralt 1995, 2000; Fito and others 1993, 1996). This can be explained by the fact that the model proposed in this work considers the amount of liquid incorporated by capillary action and by superficial adherence (A) as a separate term from that which penetrates effectively by hydrodynamic effect (S).

The standard deviations of X (Table 2) were between a minimum 0.3 % and a maximum 2.8%, what indicate a degree of precision comparable to standard deviations estimated from experimental values of X reported by Chiralt and others (1999), that were between 0.2% and 2.6% for several fruits (apple, mango, kiwi, peach, apricot, pineapple, pear, melon and prunes).

Table 3 presents the results of the application of the proposed model to predict the weight and final compositions of batches of cut fruits subjected to impregnation, using two-minute pulses and the pressures selected randomly. The model seems to work appropriately since it predicts the final weight of impregnated fruit (M) with an average absolute error in the order of 2.3%. The average absolute error in final composition of the fruit (total solids) is about 5.7%, which corresponds to a difference around 1 % between experimental and predicted compositional values.

It can be concluded that the use of simple equations based on simply obtained parameters at lab level, like volumetric fraction X (Eq. 14) and a measure of liquid adherence R (Eq. 4), can be used to predict satisfactorily final mass (Eq. 9) and composition (Eq. 11) of fruits that exhibit no major volume changes after vacuum impregnation.

Appendix

- A — Mass of adhered and capillary driven solution (kg)
- A_{ext} — External area of fruit pieces calculated from dimensions (m^2)
- a_{wF} — Experimental water activity of fresh sample
- Ci — Concentration, percentage or mass fraction of a component i
- DRP — Deformation-relaxation phenomenon
- F — Mass of fresh sample (kg)
- HDM — Hydrodynamic mechanism
- M — Mass of vacuum impregnated sample (kg)
- M_{atm} — Mass of sample after immersion under atmospheric condition (kg)
- R — Ratio of mass of sample with adhered solution to mass of fresh sample, measured under atmospheric condition
- S — Mass of impregnating solution penetrating sample by HDM (kg)
- t — Time of vacuum treatment and impregnation (s)
- V — Sample volume (m^3)
- X — Volumetric fraction of sample occupied by impregnating solution as result of HDM (m^3 of solution / m^3 of sample)
- ρ_S — Density of impregnating solution (kg/m^3)
- ρ_F — Density of fresh sample (kg/m^3)
- σ — Specific area of fruit pieces (m^2/kg)

Subscripts

A, F, M, S: Referred to corresponding masses

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