

# THE EFFECT OF PROCESS CONDITIONS ON THE PHYSICAL STRUCTURE OF DEHYDRATED FOODS: An Experimental Analysis

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The modifications of the structure of apple and potato, dehydrated with different techniques, have been related to two physical parameters, the bulk and particle densities. Particle density has been determined experimentally by means of a gas pycnometry technique, modified to take into account the effects of water evaporation during the test. On the basis of the results obtained so far, one can conclude that the independent evaluation of bulk and particle density allows the quantification of the fraction of pores closed to the outside, directly related to the reconstitutability, and to the consistency of dehydrated foodstuffs. As a consequence of the different pore network established under different drying conditions, freeze dried samples show a limited shrinkage, while thermal dried ones show a larger shrinkage throughout the whole moisture range.

*Keywords: dehydration processes; foodstuff; density; porosity*

## INTRODUCTION

Dehydrated foods have been always widely-used, being natural drying one of the few preservation techniques available since the origin of human civilization. The first dried foods were mainly protein based products, while in recent times the processing of vegetables gained importance, due to the interest of the consumer towards ready to use dishes including different ingredients.

The need to producing dehydrated vegetables and fruits means that high quality performance processes are required, because the most valuable nutritional components of these kinds of products are highly sensitive to heat effects. For this reason, vacuum, freeze drying and mild thermal dehydration processes were developed. The evaluation of the objective quality level of such products is yet matter of concern, even if panel tests on the consumer acceptance are frequently run by the major food manufacturers.

Some physical parameters are certainly related to the quality of the products, and among these the apparent density and the open pore volume fraction play an important role. They directly determine the structural properties of the product as well as its rehydration characteristics, while do indirectly influence flavour and taste perception too.

Some literature exists on the determination of apparent density of vegetables and on their shrinkage characteristics during thermal drying<sup>1-5</sup>, while open pore volume fraction has been mainly evaluated indirectly through simple model hypotheses from the true density in the dried state<sup>3-5</sup>, and only rarely through gas pycnometry<sup>1-2</sup>. This also because the application of standard gas pycnometry to moistened samples is

damped by water evaporation during the test and data are not fully reliable. Moreover, no previous work exists on the characterization of the effects of process conditions using different dehydration apparatus on the physical properties above.

This work is concerned with the characterization of the physical structure of dehydrated vegetables produced under different process conditions. The main parameters considered are the apparent sample volume and the open pores volume, determined by liquid displacement and by helium pycnometry, respectively. The latter technique has been modified in order to fit the requirements of materials relatively rich in volatiles<sup>6</sup>.

Experimental determinations are performed on samples at different stages of processing under the following conditions: air drying, vacuum drying, controlled humidity drying and freeze drying. In order to compare present results with existing data, apple and potato were selected as test vegetables. Results confirm on quantitative basis the difference in the structural characteristics of the freeze dried products in respect to thermally dried ones, and also demonstrate that a direct evaluation of open pore volume fraction is necessary to have a precise view of the physical structure of the vegetables tested.

## BACKGROUND AND DEFINITIONS

In this section the definitions of the main parameters used in the following will be introduced.

*Bulk density  $\rho_b$*

Bulk density is the ratio between the current weight of the sample and its overall volume. Considering the

overall volume as the sum of three terms: volume of solid matter,  $V_d$ , volume of liquid matter,  $V_l$ , and volume of air,  $V_a$ , the bulk or apparent density can be defined as:

$$\rho_b = \frac{M}{(V_d + V_l + V_a)} \quad (1)$$

#### Particle density $\rho_p$

Particle density is the ratio between the current weight of the sample and its overall volume diminished by the volume of the pores open to the outside. If the total volume occupied by air is considered as the sum of two contributions  $V_{acp}$  and  $V_{aop}$  ( $V_a = V_{acp} + V_{aop}$ ), related to the pores closed and open to the outside, respectively, then particle density is defined as follows:

$$\rho_p = \frac{M}{(V_d + V_l + V_{acp})} \quad (2)$$

#### Substance density $\rho_s$

The substance density is the ratio between the current weight of the sample and the volume occupied by the solid and the liquid matter only. With the further hypothesis that the mixture of dry solid and liquid is ideal, i.e. the volumes are additive  $V_s = V_d + V_l$ , the substance density is defined as follows:

$$\rho_s = \frac{M}{(V_d + V_l)} \quad (3)$$

It can be noted that particle and substance definitions densities both assume  $V_{acp} \approx 0$ .

#### Shrinkage coefficient, $s_b$

The shrinkage coefficient is the ratio between the bulk volume of the sample at a certain degree of dryness and that of the fresh sample and is defined as follows:

$$s_b = \frac{V_b}{V_{b1}} = \frac{\rho_{b1}}{\rho_b} (X_d^* + X_w X_{w1}^*) \quad (4)$$

The limit value of the shrinkage coefficient,  $s_b^*$ , can be evaluated from equation (4), by considering the maximum possible bulk volume reduction, i.e. considering the minimum possible bulk volume  $V_{b\min} = V_{d1} + V_w$ ,  $V_w$  being the volume of the water contained into the structure:

$$s_b^* = \frac{V_{b\min}}{V_{b1}} = \frac{V_{d1} + V_w}{V_{b1}} = \rho_{b1} \left( \frac{X_d^*}{\rho_d} + X_w X_{w1}^* \right) \quad (4a)$$

being  $\rho_d = \sum_i \Gamma_i \rho_i$ , with  $\Gamma_i$  and  $\rho_i$  the weight fractions and densities of solid components and with the further assumption that  $V_d = V_{d1}$ .

#### Open pore porosity $\varepsilon_{op}$

The open pore porosity is the ratio between the volume of the pores filled with air connected to the outside and the overall volume and is defined as follows:

$$\varepsilon_{op} = \frac{V_{aop}}{V_b} = 1 - \frac{\rho_b}{\rho_p} \quad (5)$$

#### Total porosity $\varepsilon_{tot}$

The total porosity is the ratio between the total volume of air present in the sample and the overall volume and is

defined as follows:

$$\varepsilon_{tot} = \frac{V_a}{V_b} = 1 - \frac{\rho_b}{\rho_s} \quad (6)$$

## EXPERIMENTAL

Stark Delicious apples and Bintje potatoes were selected as test materials. Table 1 presents data of their chemical composition. Samples, cubes cut from the fresh product, have a suitable overall volume to minimize experimental errors during bulk and particle density measurements. The initial mass of each cube is about 20 g. The reason for keeping the sample size constant is to avoid superimposing other effects on those produced by the process conditions.

Samples, dehydrated with different techniques, were extracted at the required moisture content and stored in plastic pouches, thermally sealed, to standardize moisture and temperature profiles. Four different dehydration techniques were used:

- natural convection drying: 50°C (NCD);
- controlled humidity forced convection drying: 50°C, 15% RH (FCD);
- vacuum drying: 50°C (VD);
- freeze drying (FD).

During thermal drying of potato no gelatinization of starch was observed, due to the low temperature level selected. Bulk density was measured by means of a water displacement technique using a graduated cylinder with a 0.2 ml sensitivity. Before measurements, each sample is coated with a liquid metacrylic glue (Repsol-Polivar) suitably diluted to form a very thin film upon solidification. This is to prevent water absorption, which is likely to become particularly significant at low moisture content.

Particle density is measured by means of a helium electronic pycnometer, with a maximum operative pressure of 20 psi, according to the technique developed by Donsi *et al.*<sup>6</sup> This technique minimizes the error due to water evaporation from moistened samples during the tests. A conventional gas pycnometer performs the determination of the volume free of solid through the comparison, by means of the ideal gases equation, of the pressure values recorded before and after the expansion of an inert gas in the measuring cell. This requires that the total gas mass keeps constant during the test. However, when the sample contains a volatile liquid, some vapour is formed during the test and thus

Table 1. Chemical composition of Stark Delicious apple and Bintje potato.

Component	APPLE		POTATO	
	$\frac{g}{g}$ (dry basis)	$\frac{g}{g}$ (wet basis)	$\frac{g}{g}$ (dry basis)	$\frac{g}{g}$ (wet basis)
water	5.41	0.844	3.35	0.77
dry matter	1	0.156	1	0.23
starch	—	—	0.848	0.195
soluble sugar	0.781	0.1218	0.096	0.022
cell matter	0.219	0.0342	0.0560	0.013

the total number of moles present in the gas phase increases. The measurement errors due to evaporation have been estimated in the experimental conditions tested to be of the order of 30%.

The proposed technique is based on the continuous monitoring of the pressure level in the cell. This allows the determination of the pressure trend before and after expansion and thus the identification of the pressure rise rate due to water evaporation and the elimination of its bias from the inert gas pressure level.

The reliability of the measurements of bulk and particle densities was tested by means four replicates. The average value and the experimental spread are indicated in the figures for each set of experimental data. In order to verify the reconstitutability of the dried structure, rehydratability tests, in water at 50°C, have been performed on apple samples in the case of FD, NCD and FCD, to measure the rehydration kinetics.

## EXPERIMENTAL RESULTS AND DISCUSSIONS

Data on the apparent density of apple as a function of moisture content are shown in Figure 1 for the four different dehydration techniques tested. It appears that the density of thermally treated dried samples exhibits a moderate decrease as drying proceeds, while freeze drying promotes a more sensitive and continuous density drop. It means that in thermal processes the weight reduction due to water evaporation is almost balanced by a reduction of the total volume of the same order of magnitude, while the volume reduction of freeze dried samples is much lower.

This interpretation is confirmed by analysing the trend of the shrinkage coefficient of apple samples undergoing different drying processes. Shrinkage coefficient can be calculated using in equation (4), the experimental values of the bulk density  $\rho_b$  versus water mass fraction, and is reported in Figure 1. It is worthwhile to show also the values of this parameter, in order to separate the effect of volume changes from that of mass reduction. Moreover, the shrinkage factor itself is a widely-used quality parameter related to the process history of the product.

Shrinkage data, reported in Figure 2, shows the effect of process conditions. They demonstrate that volume reduction is almost absent in freeze dried samples, while in thermally dried samples shrinkage follows the same trend, irrespective of the process used. For sake of comparison, the line of maximum shrinkage is also drawn in Figure 2, as predicted by equation (4a). Experimental data for thermally dried samples follow the trend of this line, but are always above it by a constant amount.

Bulk density and the shrinkage coefficient of potato dried by FCD and FD processes are reported in Figures 3 and 4. The trend of the data is similar to that shown by apple and a very limited shrinkage is detected in freeze dried samples. It must be noted that the maximum shrinkage value is attained by thermally dried potato samples, which is different from that observed in Figure 2 for apple. This can be related to the different initial volume fraction of air of the two foodstuffs (apple  $\approx 20\%$ , potato  $\approx 2\%$ ). Figures 1 and 3 show, for

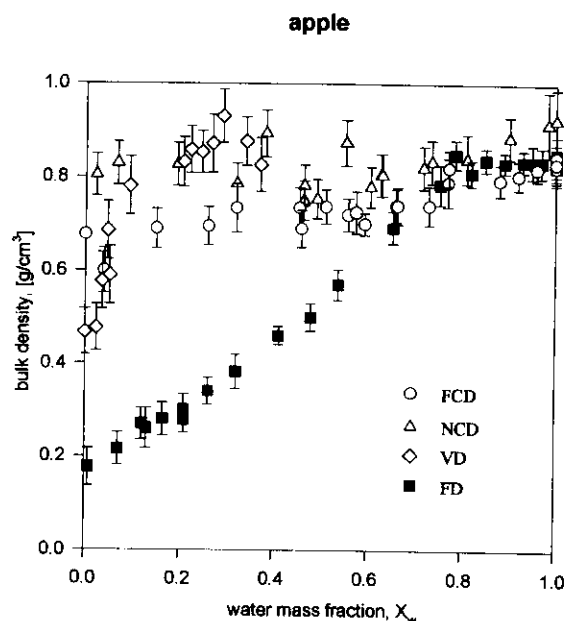


Figure 1. Bulk density of apple versus moisture content for different dehydration processes.

each drying process, the deviations of the experimental data.

The characteristics of the shrinkage phenomena occurring under different process conditions have also been analysed by means of a transmitted light microscope. Figure 5 shows a comparison between the structure of fresh and completely dried samples in FD and FCD conditions. Freeze dried samples (Figure 5(c) and 5(d)) are characterized by a structure with minimum deformation in respect to fresh samples for both products (Figure 5(a) and 5(b)). On the contrary, thermally dried samples

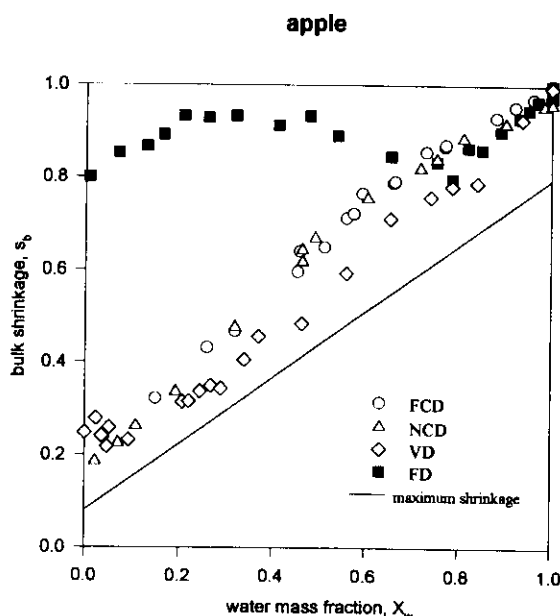


Figure 2. Shrinkage factor of apple versus moisture content. Maximum shrinkage from equation (4a) with  $X_d^* = 0.15$ ;  $X_{wt}^* = 0.85$ ;  $X_c^* = 0.03$ ;  $\rho_{b1} = 0.84 \text{ g cm}^{-3}$ ;  $\rho_{\text{sug}} = 1.56 \text{ g cm}^{-3}$ ;  $\rho_c = 1.55 \text{ g cm}^{-3}$

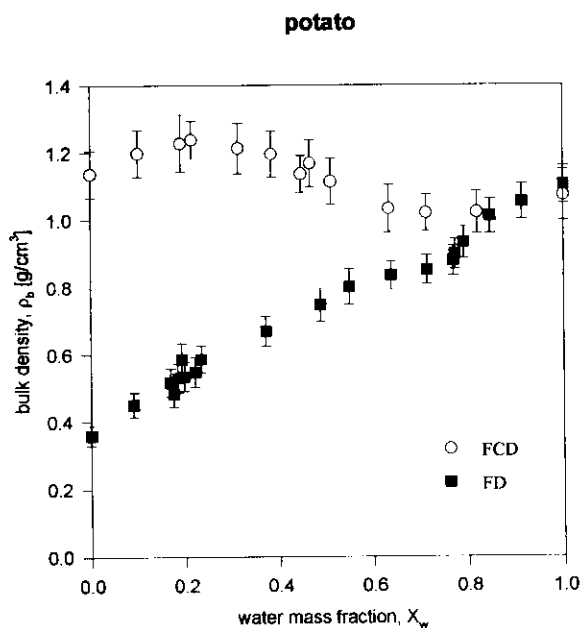


Figure 3. Bulk density of potato versus moisture content for different dehydration processes.

(Figure 5(e) and 5(f)) look more stressed: the cells are deformed and shrinkage is evident. From these pictures, it can be argued that the porosity of FD and FCD samples is likely to be very different. In the latter samples, no voidage at all open to the outside can be detected in the structure.

Particle density data measured with helium pycnometry are shown in Figures 6 and 7 for apple and potato, respectively. The figures also show, as in the case of bulk density, the deviations of the four experimental

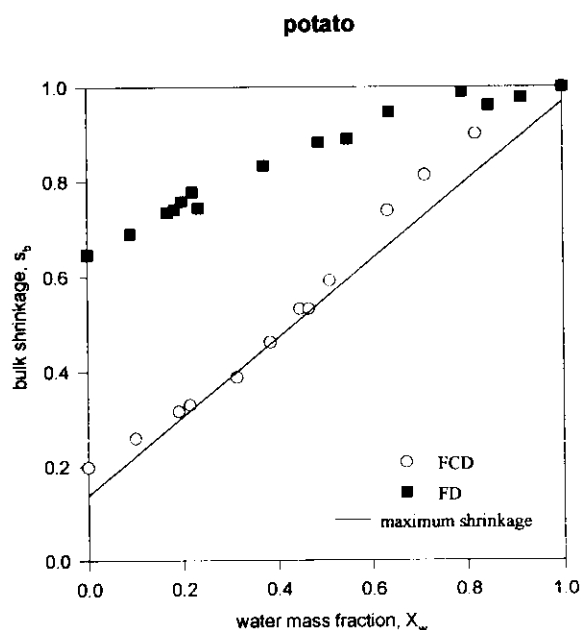


Figure 4. Shrinkage coefficient of potato versus moisture content. Maximum shrinkage from equation (4a) with:  $X_d^* = 0.21$ ;  $X_{cl}^* = 0.79$ ;  $X_c^* = 0.035$ ;  $X_{cl}^* = 0.175$ ;  $\rho_{cl} = 1.05 \text{ g cm}^{-3}$ ;  $\rho_d = 1.60 \text{ g cm}^{-3}$ ;  $\rho_c = 1.55 \text{ g cm}^{-3}$ .

replicates. Analysing the pattern of particle density data for both products, one can conclude that the values of this parameter are substantially unaffected by the type of dehydration process used. For the sake of comparison, values of substance density estimated according to equation (3) are also shown in the same figures as model lines. It can be observed that the experimental data are all in good agreement with the predictions of substance density down to moisture contents of about 0.2 kg water/kg initial water, irrespective of the dehydration process. It can be assessed that the volume amount of closed pores is negligible and that the additivity of the volumes of solid and liquid phases can be adopted. Under this moisture content, density values decrease sharply, signifying an increase in  $V_{acp}$  and that the additivity of volumes is far from being verified in the lowest moisture range. This behaviour could be a consequence of the collapse of the cellular structure due to water evaporation, as already suggested by Lozano<sup>2</sup>.

The fraction of porosity due to open pores has been evaluated from equation (5) for both products and for all processes tested, and is reported in Figures 8 and 9 for apple and potato respectively. Open pore porosity is calculated on the basis of two separate sets of experimental data, bulk and particle densities determined independently. On the contrary, open pore porosities reported in the literature are mainly model estimates<sup>4,5</sup>, based on the hypothesis that volume additivity holds throughout the moisture range. Accordingly, the possible error due to the failure of the latter hypothesis is not taken into consideration.

Data on Figure 8 and 9 demonstrate that the volume fraction of open pores is usually higher for freeze dried samples in respect to all thermal processes. For apple, this is true for all moisture contents lower than about 0.6 kg/kg initial water, while for larger moisture contents there is no effect of the process conditions on the open pore porosity. For potato, the open pore porosity is almost zero under thermal drying conditions throughout all the moisture range and the difference from freeze drying conditions is always large.

An additional evaluation has been performed, in order to stress the deviation from ideal behaviour, i.e. volume additivity, occurring in the lowest moisture range. For this purpose, the fraction of porosity due to pores closed to the outside and referred to the total porosity at dried condition, defined as  $(\epsilon_{tot} - \epsilon_{op})/\epsilon_{tot0}$ , was calculated for both products and reported in Figures 10 and 11.

Results demonstrate that the fraction of closed pores is almost zero for all processes down to a moisture content of about 0.2 kg/kg initial water. It means that in such range, the ideal hypothesis of volume additivity is true and a single determination of bulk volume is sufficient to evaluate both the total and the open pore porosity of the sample. At lower moisture contents, the closed porosity fraction increases dramatically. This is particularly so for thermally dried samples, while for freeze dried ones the effect is less sharp.

The results above demonstrate also that the drying rate is a key parameter in determining the properties of

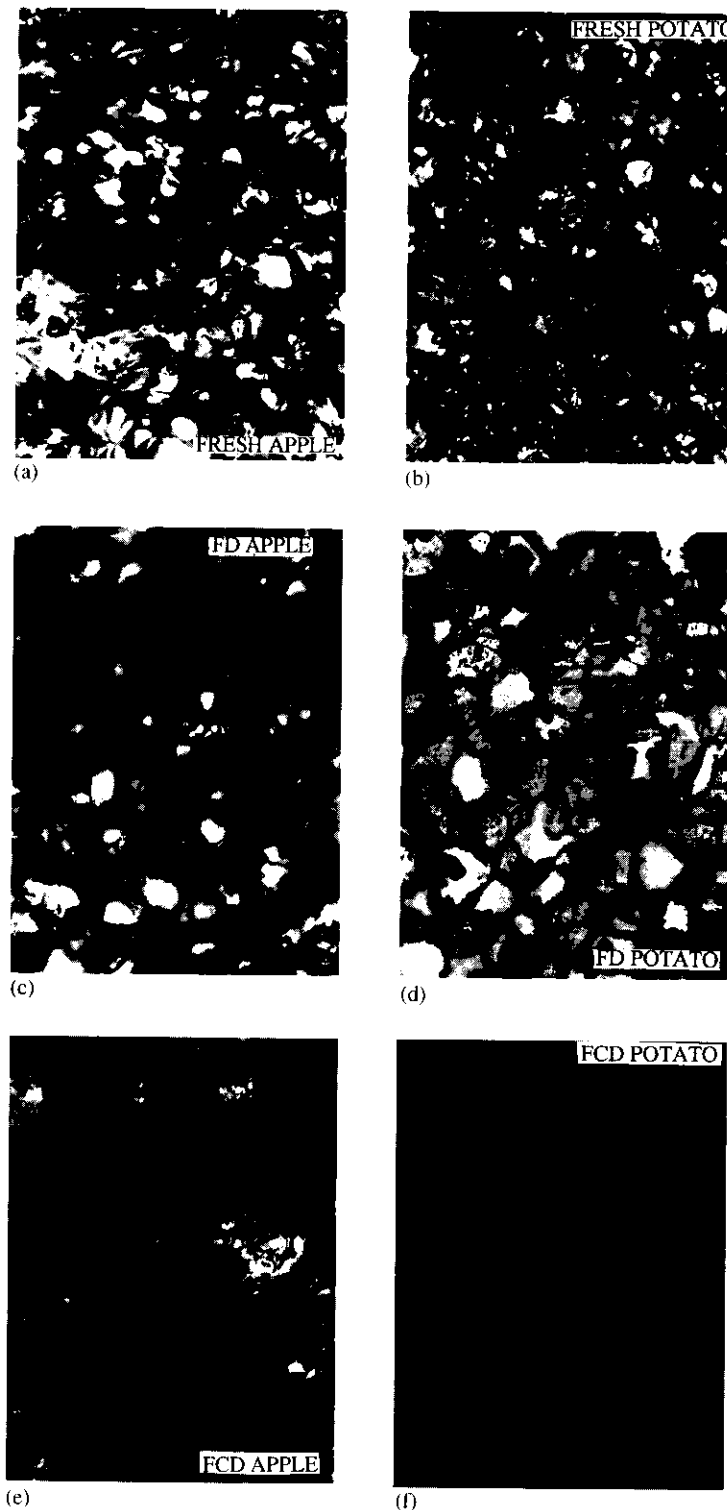


Figure 5. Transmitted light micrographs of apple and potato: fresh samples: 5(a), apple 5(b), potato; freeze dried samples: 5(c), apple 5(d), potato; dried samples in FCD: 5(e), apple 5(f), potato.

the structure of the dried product, but not the only one. The nature of the driving force promoting dehydration is also important, as demonstrated by some differences occurring between the porosity trends of vacuum and thermally dried samples, as shown in Figure 11. However, the effect on the pore structure is significant only in the lowest moisture range. The closed porosity

fraction as defined in the latter figures might be also a useful parameter in order to discriminate between mild and hard dehydration processes.

The reconstitutability of the structure has been analysed only by measuring the rehydration kinetics of apple samples dried with three different processes: FC, FCD and NCD. In Figure 12 the water content in the

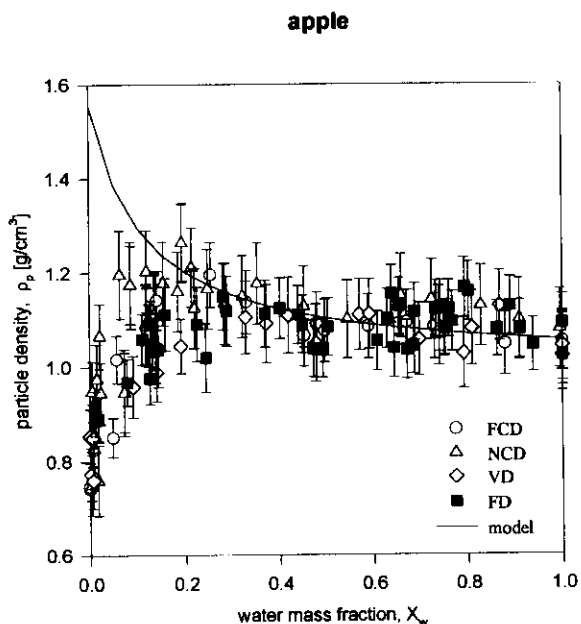


Figure 6. Particle density of potato versus moisture content. Continuous line represents model evaluations made using equation (3).

processed structure versus time is shown. As expected, the FD samples have faster kinetics and a higher maximum value of water absorbed than thermally processed samples.

### CONCLUSIONS

The first conclusions to be drawn on the basis of the experimental program performed so far, are concerned with the methodology followed:

The independent determination of bulk density and particle density for solid foods subject to drying processes can be suggested as a valuable technique in

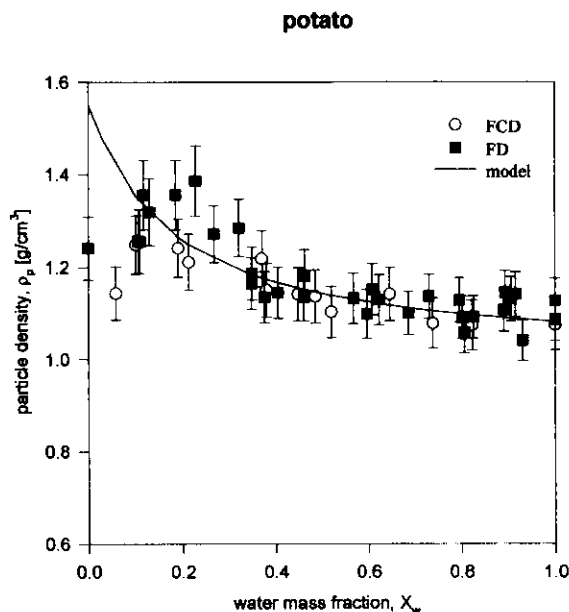


Figure 7. Particle density of potato versus moisture content. Continuous line represents model evaluations made using equation (3).

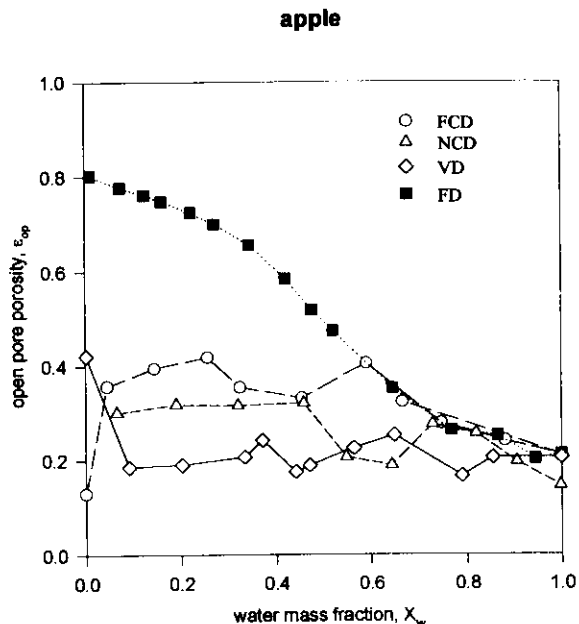


Figure 8. Open pore porosity of apple versus moisture content for different dehydration processes.

order to put into evidence the non-ideal behaviour of some products occurring, particularly in the extreme range of dryness. To this purpose, the modified pycnometry technique set up is a reliable tool for an independent evaluation of particle density.

Present results indicate that one of the features by which the products processed by freeze drying exceed the quality level of thermally dried ones, is the reduction of the porosity fraction closed to the outside. This fraction, which is directly determined as well from the experimental plan set up, seems to be a meaningful parameter in the description of the structure of dehydrated

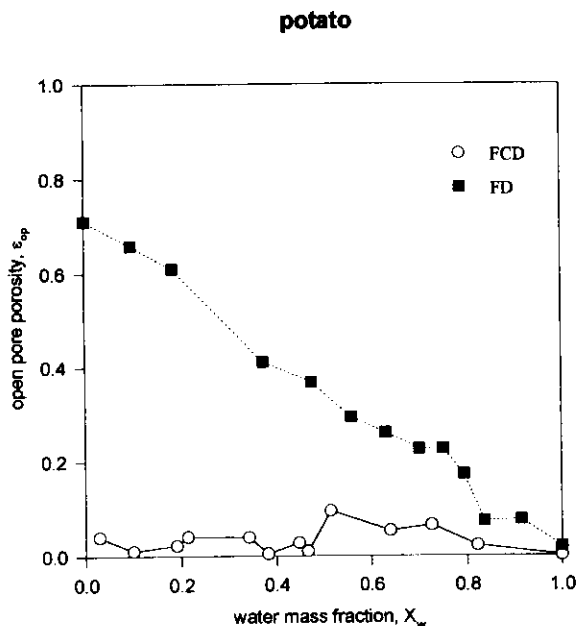


Figure 9. Open pore porosity of potato versus moisture content for different dehydration processes.

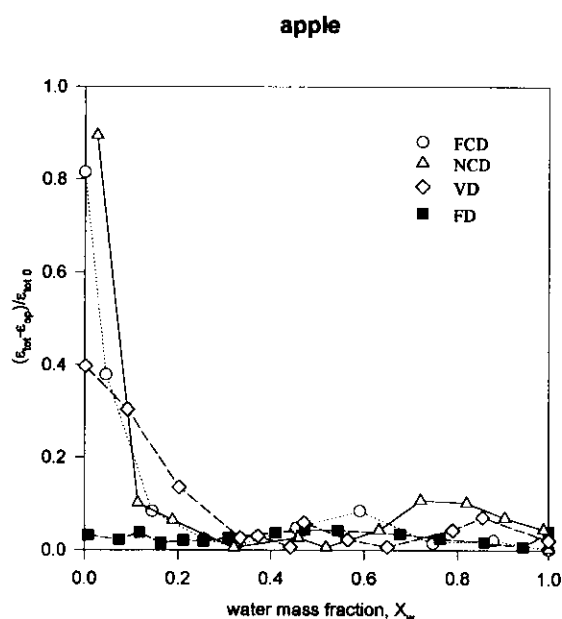


Figure 10. Closed porosity fraction in respect to total porosity at dried condition of apple versus moisture content.

foodstuff. High values of this fraction can be correlated to a poor rehydratability and make for a gummy and/or glassy consistency of the product. These effects are mainly related to the different drying rates obtained under the different process conditions, but the difference in the nature of the driving force causing dehydration is likely to be also important.

Coming to the specific conclusions about the foostuff tested, it can be said that the patterns of both apparent density of apple and potato samples subject to different kinds of thermal drying processes are very different from those of freeze dried product. Shrinkage is much larger in

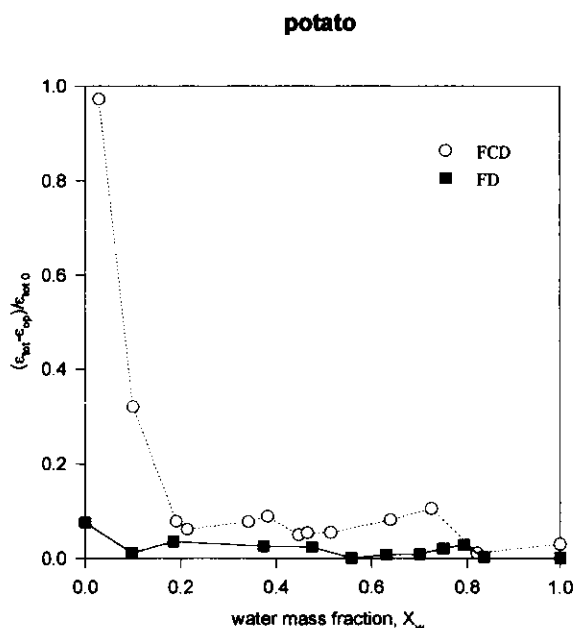


Figure 11. Closed porosity fraction in respect to total porosity at dried condition of potato versus moisture content.

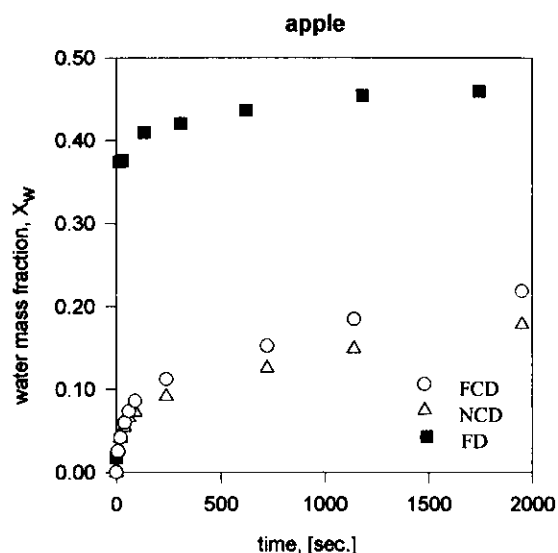


Figure 12. Rehydratability kinetics of dried apple sample ( $T_w = 50^\circ\text{C}$ ).

the first case throughout all the moisture range. In the second case, a limited shrinkage occurs only at the extreme dryness values.

Moreover, the kind of pore network developing under thermal and freeze drying conditions is different for both materials tested. It is demonstrated by the comparison of open pore porosities, which are much higher for freeze dried samples.

It has to be stressed that present results refer to samples of the same size. The effect of sample size is not discussed, but it cannot be simply disregarded without further experimental evidence. This is one of the topics that must be carefully analysed in the future in order to draw general conclusions on the effect of drying conditions on the structure of dehydrated foods.

## NOMENCLATURE

$V$	volume of constituents, $\text{m}^3$
$X_w$	water mass fraction, g/g initial water
$X^*$	mass fraction calculated on wet basis, g/g total (solid + liquid)

### Greek letters

$\varepsilon$	porosity, volume fraction,
$\rho$	density, $\text{g cm}^{-3}$

### Subscripts

a	air
acp	pores closed to outside
aop	pores open to outside
b	bulk, apparent
c	cellular wall
d	solid
l	liquid
op	open pore
p	particle, true
s	substance
st	starch
sug	sugar
tot	total
w	water
l	initial
0	completely dried structure

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