

Study of Drying Kinetics and Functional Properties of Dietary Fiber Concentrates from Papaya by-Products

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Abstract

Objectives: To analyze the drying kinetics and functional properties of Dietary Fiber Concentrates (DFCs), produced from papaya by-products (pulp or peel) dehydrated by microwave or hot air convection at 40°C. **Methods/Statistical analysis:** The DFCs were produced by modulating three variables through a response surface design. Drying curve of each design system was fitted to the Page model. Subsequently, DFCs dehydrated by microwave or hot air convection at 40°C were produced. The results of these two drying methods were compared in terms of the drying kinetics (Page or Logarithmic model) and the water and oil absorption properties of the DFCs. **Findings:** It was found that the temperature applied for drying had a significantly negative effect on the drying time and a positive effect on the K constant of the Page model. Drying times for dehydration assisted by microwave were lower than for convection drying. In general, DFCs dehydrated by microwave presented better water and oil absorption properties. These results may be related to structural differences in the matrix, which was found to be more porous, especially in the pulp DFC dehydrated by microwaves. These studies contribute to the understanding of the effect of the drying mechanism on the drying time and functional properties of the DFCs. **Application/Improvements:** This study shows that papaya by-products dehydrated by microwaves have huge potential to be reprocessed and used as food ingredients as sorbent of water and/or oil.

Keywords: Drying, Drying Kinetics, Hot Air Convection, Hydration Properties, Microwave

1. Introduction

Papaya (*Carica papaya* L), is a widely cultivated fruit, mainly in tropical and subtropical regions. Due to its physicochemical and organoleptic characteristics, it is a fruit with great potential for agro-industrial applications¹ in the production of jams, preserves of fruits, juices and other biotechnological applications such as the extraction of the enzyme papain² and/or for the elaboration of products for the cosmetics industry³.

The processes of industrialization of fruits and vegetables can generate various types of byproducts such as pieces of pulp, peels, seeds, leaves, stems, and others, depending on the industrial process implemented⁴. These byproducts, which are commonly discarded as waste, constitute an important source of nutrients for the human body and/or for the food industry⁵.

Most of the plant byproducts have high percentages of moisture, which cause difficulties for their manipulation, transport and final disposal, due to the weight and volume

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they may occupy. The drying allows the stabilization of these biomaterials due to the limitation of the microbial growth and the enzymatic action. In addition, it minimizes physical and chemical changes during storage⁶. This process leads to the preservation of food by reducing its water content through the simultaneous occurrence of heat and mass transfer⁷. Among the most common methods used for the drying of plant tissues are, freeze-drying, vacuum osmotic dehydration, dehydration by microwaves and/or the combination between them and other methods. The choice of method depends on factors such as: the characteristics of the drying equipment, the type of product, the cost of dehydration and the desired quality for the dried product, with fuel consumption and the quality of the final dry products being the most critical considerations for choosing the drying method.

In different research where the processes of food dehydration are studied, mathematical models are used for simulating the decay of moisture and the mass transfer. The most studied models are those of Newton, Page, Henderson and Pabis, Logarithmic and Diffusion among others⁸.

In previous studies, an optimization methodology had been developed to produce DFCs from papaya pulp with maximized physical-chemical and functional properties,

using a response surface design. This optimization process was carried out by modulating three process variables, including the drying temperature during the dehydration stage assisted with microwaves⁹. Nevertheless, the study of the drying processes could not be discussed in that opportunity. The aim of the present study was to deepen the analysis of the kinetics of the dehydration processes for obtaining DFCs from papaya pulp through microwave assisted drying. In addition, a comparison between microwave and hot air convection drying in terms of the DFCs functional properties was also carried out.

2. Materials and Methods

2.1 Production of Dietary Fiber Concentrates from Papaya Pulp

The optimization of the production process of the DFC from pulp had been carried out by treating this material with ethanol, with a different Ethanol/Sample (E/S) ratio and varying the treatment time (t) as shown in Table 1. Subsequently, the obtained residue was dehydrated in a microwave, at different Drying temperatures (Dt) according to a response surface design (Table 1).

Table 1. Box Behnken experimental design, factors and responses for microwave drying

System	t (min)	E/S (mL/g)	Dt (°C)	Drying time			R ² of the regression	Standard error of estimate
				(min)	K	n		
1.	15 (-1)	2 (-1)	60 (0)	19	0.0190	1.7125	99.27	0.0348
2.	45 (1)	2 (-1)	60 (0)	24	0.1013	1.2260	99.95	0.0083
3.	15 (-1)	5 (1)	60 (0)	24	0.0647	1.3480	99.81	0.0166
4.	45 (1)	5 (1)	60 (0)	21	0.0267	1.8059	99.65	0.0252
5.	15 (-1)	3.5 (0)	40 (-1)	60	0.0194	1.2518	98.81	0.0379
6.	45 (1)	3.5 (0)	40 (-1)	75	0.0180	1.1587	99.47	0.0245
7.	15 (-1)	3.5 (0)	80 (1)	14	0.0914	1.5600	99.86	0.0161
8.	45 (1)	3.5 (0)	80 (1)	21	0.1486	1.2102	99.98	0.0051
9.	30 (0)	2 (-1)	40 (-1)	65	0.0127	1.3202	99.57	0.0230
10.	30 (0)	5 (1)	40 (-1)	65	0.0186	1.2511	99.52	0.0241
11.	30 (0)	2 (-1)	80 (1)	18	0.1433	1.2386	99.97	0.0069
12.	30 (0)	5 (1)	80 (1)	18	0.1841	1.1583	99.80	0.0178
Central 1	30 (0)	3.5 (0)	60 (0)	24	0.0495	1.4303	99.84	0.0156
Central 2	30 (0)	3.5 (0)	60 (0)	27	0.0611	1.3255	99.82	0.0157
Central 3	30 (0)	3.5 (0)	60 (0)	24	0.0452	1.5167	99.61	0.0245

t: time of treatment with ethanol, E/S: ethanol/sample ratio, Dt: Drying temperature, K and n are parameters of the Page adjustment. The numbers in parenthesis represent the values coded for each level of the design

For each system of the experimental design, 300 +/- 0.01g of papaya pulp tissue were processed with ethanol (96 mL/100mL) in a food processor 3MQ 300 (Braun, Barcelona, Spain). Subsequently, the residue was filtered and placed in a polypropylene tray (PP5), obtaining a = 4 cm bed height. The tray was left under fume hood, for one hour to eliminate the remaining ethanol. There after it was placed inside a microwave oven (Ethos plus, Milestone Italia) working at a maximum power of 600W, intermittent, in order to keep the temperature of the sample around 40°C. This temperature was selected from previous works⁹.

For hot Air Convection (FAC) a chamber, Argentina, and Model SRBCO 4040) with a hot air velocity of 2.0 m/s was used. Air temperature was controlled in order to keep similar sample conditions (40°C) than microwave process.

During the dehydration of the DFCs, the weight loss of the samples was recorded through time until to reach a constant weight and aw values less than 0.6 to guarantee the microbiological stability¹⁰. The drying curves were drawn, plotting the variation of the moisture ratio (MR) as a function of the time of dehydration.

2.2 Modeling of Drying Curves

All the data corresponding to the microwave drying curves during the development of the experimental design were modeled by a non-linear regression analysis using the page (Eq.1) equation:

$$MR = \frac{(m - m_e)}{(m_o - m_e)} = e^{(-k \cdot t^n)} \quad (1)$$

Where:

MR represents the variation of the moisture ratio, m the moisture content at a given time t (% on dry basis), m_o the initial moisture content (% on dry basis), m_e the moisture content at equilibrium (% on dry base), (t) is the time in minutes; (K) and (n) are the parameters of Page⁶. The equilibrium moisture (m_e) was determined based on the last three points of the drying curve, for equally spaced intervals (Eq. 2).

$$m_e = \frac{m_1 * m_3 - (m_2)^2}{m_1 + m_3 - 2 * m_2} \quad (2)$$

Where:

m_1 is moisture on dry basis at time t
 m_2 is the moisture in base dry at time $t + dt$
 m_3 is moisture on dry basis at time $t + 2dt$

The parameter R^2 and the standard error were used to evaluate the adjustment of the experimental points to the regression curve. The parameters (K) and (n) of Page, were obtained with the regression models for each system.

For the comparison of the two drying methods (microwaves and hot air convection) at 40°C, the curves representing the moisture loss of the four DFCs during the dehydration, were adjusted to the Page and Logarithmic models⁸, this last model is described by the equation:

$$MR = a \cdot e^{(-K \cdot t)} + c \quad (3)$$

Where:

MR is the relative moisture, for each (t) time, and (K) and (c) are the constants of the Logarithmic model.

2.3 Experimental Design and Statistical Analysis

The response surface methodology is an empirical modeling technique used to establish the relationship between a set of experimental factors and the observed results. For the study of the drying processes of the DFCs, the model of Box Behnken (Table 1) was used, to verify the effect of the 3 variables involved in the production process (treatment time with ethanol (t) , ethanol/sample ratio (E/S) , and drying temperature (Dt) on the responses: drying time and the constant drying rate (K) of the Page equation. Each process variable was analyzed on two levels and central points in triplicate (Table 1).

Using an analysis of variance (ANOVA), verification was made regarding which of the process variables had significant effects ($p < 0.05$) on the analyzed responses. The proportion of the variance detailed by the proposed model was calculated by determining the co-efficient R . In addition, the adjustment of the model was evaluated by using the lack of fit test¹¹⁻¹², which is significant for values less than $p < 0.05$.

For the formulation and analysis of the experimental design, as well as to perform each of the non-linear regressions for the modeling of the drying curves, the statistical program Stratgraphics Centurion XV (02/15/06 v, 2007) StatPoint Inc. Warrenton, Virginia was used. Moreover, to compare the properties of water and oil absorption of the DFCs, described below, an ANOVA and a Tukey Test were also performed using the same program.

2.4 Water and Oil Absorption Properties

These properties were determined on the pulp and peel DFCs, produced after anethanolic treatment for 15 minutes, using an ethanol/sample ratio of 2.9 mL/g and dehydrated by microwave or hot air convection at a temperature of 40°C. The effect of the drying mechanism on the properties such as: Water Holding Percentage (WHP) and Oil Holding Percentage (OHP) was measured. These properties were determined after the hydration and/or contacting with oil a weighed amount of DFC during 18 h at 25°C. The determinations were made at least in triplicate and are briefly described below.

2.4.1 Water Holding Percentage

After the hydration of the concentrates, the supernatant was discarded; the weight of the hydrated residue was recorded and subsequently was freeze dried for 48 hours. WHP was calculated as the percentage ratio between the water absorbed by the DFCs and the water added.

2.4.2 Oil Holding Percentage

After the contact time between the sample and the oil, it was centrifuged for 5 minutes at $1500 \times g$. The supernatant was discarded, and the pellet was weighed. OHP was calculated as the percentage ratio between the oil absorbed by the DFCs and the oil added.

2.5 Microstructure Analysis

The microstructure of the pulp papaya DFCs was analyzed by means of SEM (Zeiss Supra 40, Germany). The DFCs were previously stabilized in a desiccators containing CaCl_2 during three days. After this time, they were fixed on a bronze support and covered with a thin gold foil before microscopic observation (Sputter Modelo 108, Cressington Scientific Instruments Ltd., Watford, GB, and United Kingdom). The samples were tested under vacuum at an accelerating voltage of 3.00 kV.

3. Results and Discussions

The effect of the process variables on the responses were analyzed by means of Pareto charts for microwave drying. These graphs show the effects in order of decreasing significance, denoting with a line, the effects that are statistically significant and showing if the impact is positive or negative from the response variable under

consideration. Any bar that exceeds the vertical line is statistically significant with a confidence level of at least 95%. The Page parameters corresponding to the dried systems of the experimental design are presented in Table 1.

The drying times varied according to the modulation of the process variables, ranging between 14 and 75 minutes. The drying temperature was the process variable that significantly affected the drying time ($p < 0.01$) in a negative manner, as observed in the Pareto analysis (Figure 1), indicating that higher drying temperatures will result, as expected, in lower drying times.

Likewise, the quadratic term (CC) for this same variable has a significant effect on this response, which can explain the curvature of the response surface (Figure 2). It was determined that the Box Behnken model represent the 99.01% of the variability of the drying time results ($R^2 = 99.01$). In addition, it was found that the lack fit test was not significant for this response ($p = 0.144$), indicating that the model is adequate to describe the observed data. During dehydration of vegetables, the drying time is inversely proportional to the drying temperature

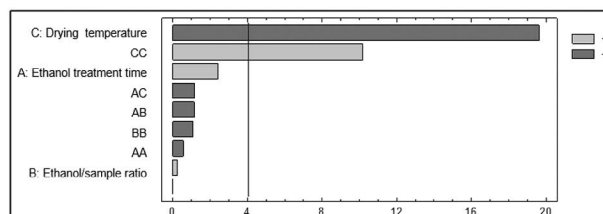


Figure 1. Pareto analysis for the drying time using a microwave.

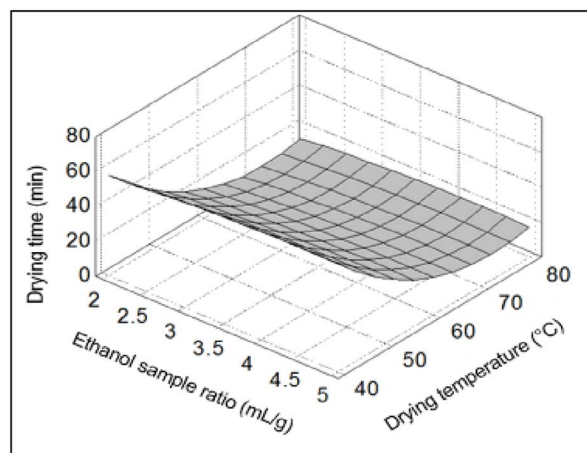


Figure 2. Surface response for the analysis of the drying time by microwaves.

and/or to the power applied in the case of microwave dehydration⁶.

As shown in Figure 2, for any E/S ratio, the drying time decreases with the increase of the drying temperature, for a treatment time with ethanol set at 15 minutes. On the other hand, in Figure 3, it is observed that the drying temperature is the factor that significantly influences ($p < 0.01$) and in a positive manner on the constant speed K of Page, which indicates that at higher drying temperatures, there will be higher values of K and therefore a higher drying speed. In the studies carried out by¹³, it was demonstrated that the increase of the drying temperature, during the drying of apricots (*Prunus armeniaca*), produced a proportional increase of the Page K parameter. Figure 4 shows the variation of the K parameter of the Page equation, due to the modulation of the E/S ratio and the microwave drying temperature, with a treatment time with ethanol of 15 minutes. It is observed that (K) increases mainly when the drying temperature is increased for any E/S ratio. For this response, an R^2 of 95.42 was obtained, and it was also found that the lack of fit test was not significant ($p=0.101$), denoting

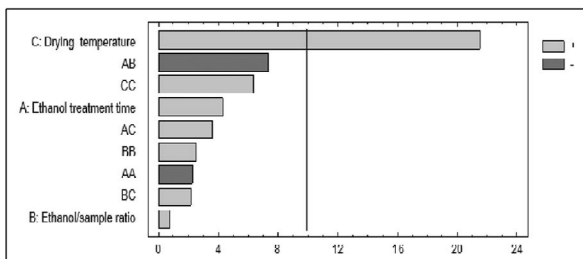


Figure 3. Pareto analysis for the constant K of the Page equation. Drying by microwave.

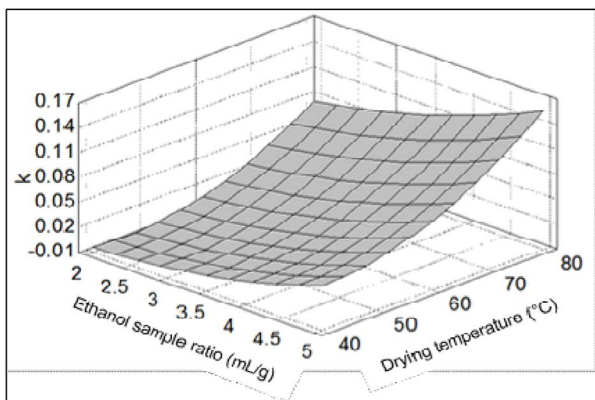


Figure 4. Surface response for the K parameter of Page. Drying by microwave.

the model fitted adequately. In studies carried out by¹⁴, the microwave drying of mushrooms (*Agaricusbisporus*) was analysed using a response surface design, and it was observed that there was a similar tendency of an increase in the Page K parameter, with the increase of the drying temperature.

As for the analysis of adjustment of each of the dried experimental design, values and standard error (Table 1) oscillated between 0.9551 and 0.0379. These values represent the residual variation and allow for inferring that the regression line constructed in the set of data itself is well adjusted; the smaller the variation, the steeper the estimated calculations⁸. The error values obtained in this investigation are on the order of those that have been reported as estimators of a good adjustment made during the drying modeling of other foods such as nuts⁸ with the Page model and other models.

Figure 5 shows the microwave drying the fitted curves obtained for the systems of the experimental design. The longest drying times were presented in systems 5, 6, 9 and 10, which used temperatures of 40°C, while the other drying times systems were similar. Although drying at 40°C took longer drying times for dehydration, this temperature was chosen for comparison with hot air convection drying, since at this temperature, DFCs showed the optimal functional properties⁹.

3.1 Comparison of Two Drying Methods for the Dehydration of Dietary fiber Concentrates from Pulp and Papaya Peel

Using the optimized production method of DFC papaya pulp (t : 15 min, E/S: 2.9 mL/g, Dt: 40 C), dietary fiber

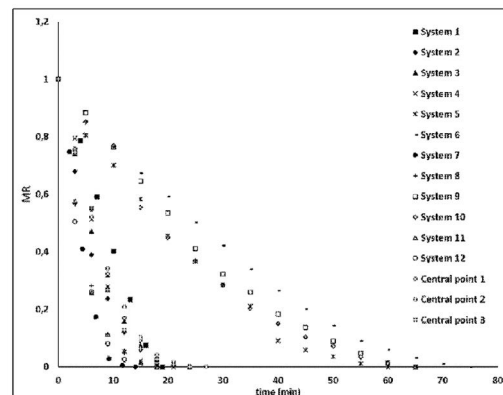


Figure 5. Drying curves by microwave of the box Behnken design.

concentrates from papaya pulp and peel were produced by microwave (MW) or by convection with hot air (CV), to verify the effect of the drying mechanism and the type of tissue (pulp or peel) on the drying kinetics and some functional properties of the DFCs. It was found that drying with microwaves allows the dehydration in almost half the time of hot air convection drying for the DFCs of both pulp and peels (Figure 6).

The data of the curves corresponding to the loss of moisture over time were adjusted to the Page or Logarithmic models and the best fit is reported in each case. The best model to describe the pulp microwave drying (DFC-MW pulp) and the peel convection drying (DFC-CV from peel), was the Page model. Meanwhile, peel microwave drying (DFC-MW from peel) and pulp convection drying (pulp DFC-CV), Logarithmic model improved the adjustment. Drying curves and their respective adjustments are shown in Figure 6.

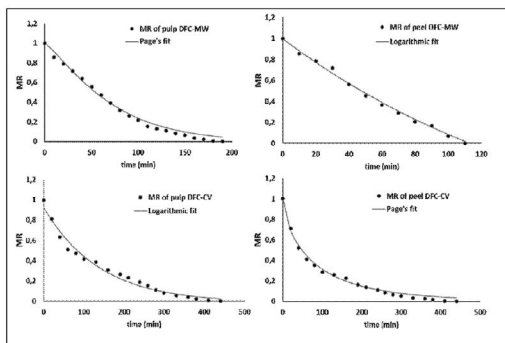
The equations determined, with coefficient $R^2 > 99\%$, that the moisture at certain drying times, for the dehydration of the four DFCs were as follows:

DFC from pulp and dehydrated by microwave, according to the Page model:

$$MR = e^{(-0,0155 \cdot t^{1,2019})} \tag{4}$$

DFC from peel and dehydrated by microwave, according to the Logarithmic model:

$$MR = 2,2373 \cdot e^{(-0,0054 \cdot t)} - 1,2374 \tag{5}$$



DFC-MW: concentrated dietary fiber dehydrated by microwaves. DFC-CV: dietary fiber concentrate dehydrated by hot air convection

Figure 6. Drying curves for obtaining DFC using microwave or convection at a temperature of 40°C.

- DFC from pulp and dehydrated by hot air convection, according to the Logarithmic model:

$$MR = 0,09447 \cdot e^{(-0,0072 \cdot t)} - 0,0183 \tag{6}$$

- DFC from peel and dehydrated by hot air convection, according to the Page model:

$$MR = e^{(-0,0437 \cdot t^{0,7123})} \tag{7}$$

The description of the drying processes through the different mathematical models is a useful way to understand the drying kinetics and for optimization the conditions of the processes¹⁵. Although drying is an important operation during the processing of fruits and vegetables, the drying characteristics of dietary fiber concentrates are rarely available, hence, this project is a contribution made to improve the understanding of the dehydration process of this type of products.

3.2 Water and Oil Absorption Properties of Dietary Fiber Concentrates from Papaya Pulp and Peel

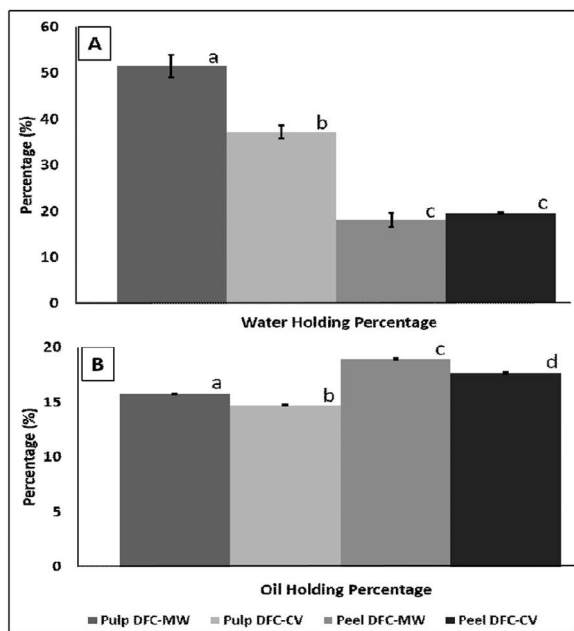
Functional properties related to the absorption of water and oil has been used to study the quality of products obtained from vegetable tissues¹⁶⁻¹⁸. To a large extent, these properties depend on the temperature and the drying mechanism. The results found in terms of the WHP and OHP of the DFCs are represented in the Figure 7.

WHP denotes the percentage of water that the material absorbed into its structure. In some works, such as those carried out by¹⁹, this water is considered to be slightly associated with the dietary fiber, therefore, the measurement of this property gives us a general idea of the function that this material can fulfill in a food and in the organism. DFCs with a high water holding capacity may be used as functional ingredients to help prevent syneresis and modify both the viscosity and texture of some formulated foods¹⁶.

The pulp DFC-MW achieved a higher water holding percentage ($p < 0.05$) than from pulp DFC-CV. Although the pulp DFCs come from the same vegetal tissue, they were dehydrated by using two different mechanisms, which caused the differences observed in this property. The exposure to heat produces changes in the tissues structure and physiochemical properties and consequently their functional properties²⁰. The dehydration

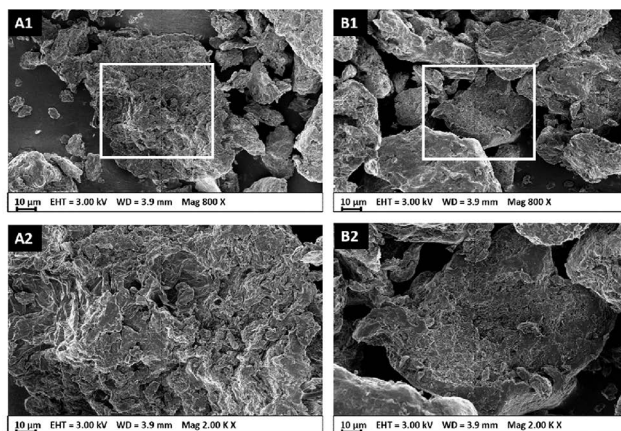
by hot air convection led to longer drying times, which produced a differential alteration of the hydrophilic properties with regards to the DFCs dehydrated by microwaves. The microstructure of the pulp DFCs can be observed in Figure 8. In panels A1 and A2 it was observed that pulp DFC-MW had a more porous structure than pulp DFC-CV (Panels B1 and B2). In turn, these last ones, showed more areas with matrix collapse and more compact and flattened areas. These structural differences could help, in part, to explain the differences observed in water absorption. Microscopic analyzes performed in fractions enriched in dietary fiber obtained from the peach bagasse, had shown that drying by microwave allowed to keep the structure of these tissues after being subjected to dehydration temperatures of 55°C²¹.

On the other hand, no significant differences ($p < 0.05$) were found between the DFCs from the peel in terms of WHP. Figure 7 also shows that samples dried with microwave assistance (pulp DFC-MW and peel DFC-MW) presented slightly higher OHP, than those dried by convection with hot air. It has been reported



DFC-MW: concentrated dietary fiber dehydrated by microwaves. DFC-CV: dietary fiber concentrate dehydrated by hot air convection. Different lowercase letter on the bars means significant differences ($p < 0.05$)

Figure 7. Functional properties found in the dietary fiber concentrates dehydrated by microwaves and hot air convection.



Panels A1 and A2 panels A1 and A2 correspond to pulp DFC-MW and panels B1 and B2 to pulp DFC-CV. The A2 and B2 panels are a magnification of the white box part.

Figure 8. Microstructure of the pulp DFCs.

that this property is largely dependent on the porosity of the matrix²², which is affected by the phenomena of collapse and shrinkage during the drying process due to the effect of the temperature and the presence of free sugars¹². Besides, OHP is a property, which can be important in some food production, helping to reduce lipid loss during processes such as baking²³, but it is also of great importance to the human body due to the capacity for retaining fats in intestinal lumen helping to reduce serum cholesterol levels²⁴.

4. Conclusion

The dehydration process for obtaining dietary fiber concentrates from pulp and papaya peel was studied through the mathematical models of Page and Logarithmic. The analysis of the response surface design demonstrated that the microwave drying temperature had a significantly negative effect on the drying time and positive effect on the Page K parameter. Likewise, the use of dehydration technique and the type of tissue source (peel or pulp) determined that the adjustment of the drying curves was better described with the Page model or with the Logarithmic model.

It was determined that microwave drying took shorter drying time than hot air convection drying. In addition, among the dietary fiber concentrates of the papaya pulp, those that were dehydrated by microwaves, had better water and oil absorption properties than those obtained by hot air convection and the same tendency was showed

with those coming from peel. These results allow us to infer that DFCs dehydrated by microwaves have great potential for use as food ingredients, not only because of their better water and oil absorption properties but also because they can be produced in shorter times, which could also generate some economic benefits. These results represented useful information for the design of drying processes for the production of dietary fiber concentrates from papaya by products.

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