

Comparative Study of Various Pre-Treatments Coupled to Vacuum Drying in Terms of Structural, Functional and Physical Properties of Carrot *Daucus Carota*

Wafaa SAHYOUN¹, Farid ZERROUQ², Karim ALLAF³

^{1,3}University of La Rochelle, Intensification of Transfer Phenomena on Industrial Eco-Processes, Laboratory of Engineering Science for Environment LaSIE - UMR-CNRS 7356, 17042 La Rochelle, France

¹Faculty of Sciences, Department of Biology, Lebanese University, Tripoli, Lebanon

³University of Fez and Ecole Supérieure de Technologie BP 2427 Morocco.

Abstract— Different processes were studied as pretreatment operations in order to adequately intensify the drying of carrot. Blanching, freezing/thawing F/T, Steaming implying high saturated steam pressure and 120-s decompression time, Instant controlled pressure drop process (DIC) with pressure-drop time of about 0.02 s were studied. Their various texturing impacts were defined through the value of porosity ration ρ and the Scanning electron microscopy (SEM). More than the determination of the specific impacts of each considered pretreatment operation; correlations were established between various structural and functional parameters, and between drying performances and final product quality.

Keywords— Carrot, Instant controlled pressures drop (DIC), Freezing/thawing, lipid content, carotene content, vacuum drying.

I. INTRODUCTION

Carrot is a seasonable vegetable, whose preservation has required various techniques conducted to preserve its availability. One of these most known operations has been the drying. Drying processes are carried out on most of the vegetables to extend their shelf-life (Barat and Grau, 2016; Jangam et al., 2016; Moses et al., 2014). They include convection drying using hot air (Gamboa-Santos et al., 2013), freeze-drying (Garcia-Amezquita et al., 2016), vacuum drying (Richter Reis, 2014), micro-wave drying (Cui et al., 2004), infrared technology (Kocabiyik and Tezer, 2009), etc. Compared to the other techniques, vacuum drying has the advantage to use reduced pressures and low temperatures to perform water evaporation (Richter Reis, 2014). This should decrease the thermal degradation and favor the quality of the final dried vegetables. Several pretreatment procedures are proposed and used. They differ in terms of nature and effect on the drying kinetics and the nutritional and functional quality of the final dried product. Since the natural structure of plant is inadequate for internal diffusion of water mainly because of the presence of cells whose walls act as great barrier, several processes are usually proposed as pre-treatments of drying (Arévalo-Pinedo and XidiehMurr, 2007; Niamnuy et al., 2014). They mainly aim at reducing drying time, preserving the final product quality. They include blanching (Negi and Kumar Roy, 2001), thermo-mechanical treatments, freezing/thawing (Kidmose and Martens, 1999). Blanching and thermal treatment are well-known as leading to the destruction and/or reduction of surface microorganisms, enzyme activity, and removal of air from surface and intercellular spaces which helps in prevention against oxidation (Rahman & Perera, 1999). Freezing/thawing is another pre-treatment procedure used to accelerate drying and enhance the quality of the dried product. By using freezing/thawing as a pretreatment applied before drying, it was possible to imply some rupture of cell walls and higher stabilization of carotenoids and lipid content of the dried carrot (Albertos et al., 2015). Other industrial drying technologies have been defined through introducing a direct thermal and mechanical effect on microstructure of plant organs. This allows reducing cost, increasing both process performance and final product quality. At this respect, the very specific process of swell-drying combining conventional drying and a step of texturing by Instant Controlled Pressure Drop (DIC) technology was defined and has been developed (Mounir et al., 2014). DIC is an innovative process that is based on the thermo-mechanical effects issued from an abrupt dropping of pressure towards a vacuum (Allaf and Allaf, 2014). Thus, DIC is a high-temperature short time (HTST) treatment followed by an abrupt drop of pressure towards a vacuum of about 5000 Pa. This causes an autovaporization, implies a possible texturing of the material, and allows higher drying kinetics. The advantage of DIC treatment is that low pressure induces an instant cooling of treated products towards a temperature level of about 33 °C, thus preventing their thermal degradation. DIC process has been used to swell-dry, decontaminate, and texture various fruits and vegetables (Maritza et al., 2012; Téllez-Pérez et al., 2012); it ensures a high quality by improving the kinetics and the capacity of both

dehydration and rehydration processes as well as the possibility of preserving and even increasing the organoleptic content and the availability of bioactive compounds such as antioxidant activities (Pedrosa et al., 2012).

In previous studies, the effect of DIC pre-treatment on various vegetable drying was investigated (Ben Amor and Allaf, 2009; DEBS-LOUKA et al., 1996; Maritza et al., 2012; Téllez-Pérez et al., 2012). DIC implied reducing drying time and improving the availability of numerous active molecules and lipids. The present study aimed at establishing comparative effect of various pre-treatments in terms of structure and functional behavior and physical properties of carrot as vegetable.

II. MATERIALS AND METHODS

2.1 Sample preparation and pretreatments

Fresh carrots (*Daucus carota* L. var. Nantesa) were purchased in Picardie-France from a popular local market and stored at 4-6 degrees for a maximum of 5 days. Carrots were washed and peeled before treatment. Spikes and collars are removed at the same proportions. The roots were cut into slices of 0.5 cm thick.

2.1.1 Blanching

Blanching was performed using a steam flow with absolute pressure of 0.15 MPa and temperature of 110 °C for duration of 10, 12, or 30 min.

2.1.2 Freezing/ thawing

Each freezing/thawing cycle begins after a 12-min steam blanching stage. Such cycles were applied using a programmable freezer with either fast, intermediate, slow and very slow cycles as mentioned below:

- **Fast freezing:** freezing was performed at -35 °C using low temperature airflow in direct contact with the product. Some hours later, rapid thawing was achieved in a plastic bag at a temperature of 25 °C with an internal product temperature rate of 6 °C/min (for a period of 10 min).
- **Intermediate freezing:** freezing was performed at -25 °C followed by thawing grids at room temperature of 20 °C (1 °C/min in the product and a thawing period of 45 min)
- **Slow freezing:** freezing was carried to reach -25 °C in 100 min. Some hours later, thawing was carried out in 100 min following a temperature change cycle given in Figure 1.a.
- **Very slow freezing:** freezing was operated in 1000 min. Product was placed in aluminum cups covered with kitchen roll paper in order to limit the heat transfer. The cycle followed a temperature-time program as given in Figure 1.b.

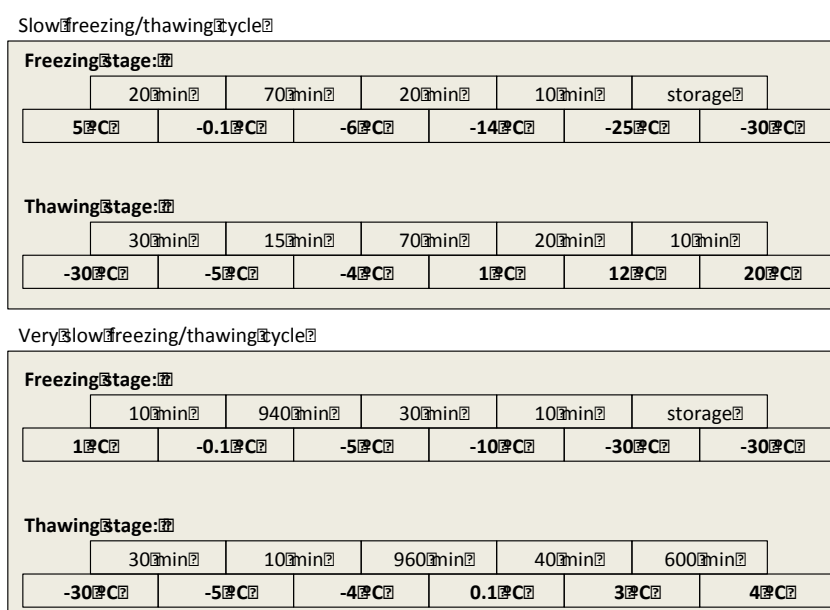


FIGURE 1. PROCESSES OF: A) SLOW FREEZING/THAWING CYCLE AND B) VERY SLOW FREEZING/THAWING CYCLE.

2.1.3 Steaming and DIC treatments

DIC treatment was carried out with a laboratory scale DIC prototype at the University of La Rochelle as previously described (ALLAF et al., 1993). After a first short stage of vacuum, saturated steam flux was injected at were used different pressure values (0.25; 0.35; 0.45; and 0.55 MPa) for 20 s as heating time. Two types of decompression were used:

- Progressive decompression requiring 2-3 min to reach the atmospheric pressure (conventional steaming),
- Instantaneous pressure drop within 200 ms to reach 5000 Pa (DIC treatment).

Frozen/thawed samples were compared to a control-A (untreated) and a control-B (only subjected to 12-min blanching).

2.2 Vacuum drying

After their respective pretreatment step, samples were dried by vacuum drying using a specific prototype fabricated at the University of La Rochelle; with pressure stabilized at 1300 Pa and heating plate temperature was 80 °C. Constant weight loss was measured during time intervals. Drying time was monitored until dried product reached the desired weight.

2.3 Assessments of samples

2.3.1 Water content Determination (dehydration time)

Water content was determined according to Karathanos' method (Karathanos1999, Koridaet al., 2003). Water content of fresh and totally dried carrots was gravimetrically measured in triplicate by drying 1 g of sample in a laboratory drying oven UFE 400 at 70 °C during 24 h. The water content dry basis db (W) of samples was calculated using the following equation:

$$W(t) = \frac{\Delta M_{\text{water}}(t)}{M_{\text{Dry product}}}$$

2.3.2 Capacity of Rehydration

The capacity and rate of rehydration of dried samples were evaluated as following: carrot samples (about 0.51± 0.02 g) was placed in the clip handle tea strainers, and submerged in distilled water (at 20±0.05°C). At specific time intervals (0, 1, 20 min) samples were taken off from the water, blotted with tissue paper to remove superficial water, and re-weighted. Weight was recorded using an electronic balance AR2140 (OHAUS, China).

2.3.3 Water Activity (aw)

AUTOSORP (Quantachrome instruments, Florida, USA) apparatus (Biosystems) was used to determine the moisture sorption isotherms at 25 degrees with a relative humidity ranging from 50% to 0%.

2.3.4 Density and absolute porosity ratio ϑ

Sample density was determined through buoyancy effect. The measurements were performed using a Sartorius balance (Göttingen, Germany) provided with a density measurement pattern connected to a PC via a data acquisition card whose sampling time is less than 3 s. The hydrophilic nature of the product normally results in errors, which can be reduced by using short time recording of the evolution of the mass during the immersion of sample in water. The measurements are performed on a number of individuals ranging between 3 and 5 with an estimated 2% error.

Since pycnometer method (Tipler P.A.Sargent Welch N.Y. USA.) allowed determining the intrinsic density of the carrot samples to be 1.258±0.008, the absolute porosity ratio ϑ could be determined as:

$$\vartheta = \frac{V_{app} - V_{int}}{V_{app}} = \frac{1/\rho_{app} - 1/\rho_{int}}{1/\rho_{app}} = 1 - \frac{\rho_{app}}{\rho_{int}}$$

2.3.5 Determination of lipid and carotene content

a) Lipid extraction

Fresh carrots: 10 g of sample were collected from several fresh slices crushed in a food blender and poured into a Buchner funnel (DURAN, fried glass 2) mounted on an Erlenmeyer flask. This puree was extracted twice with 30 ml of chloroform / methanol (2:1) and twice more with 20 ml of chloroform alone. Before each of these extractions, the carrot puree was broken in a mortar. The extracts were combined and washed twice with 100 ml of distilled water by stirring, and then decanted into a funnel. The remaining organic phase was dried over anhydrous sodium then filtered. The filter was rinsed with 10 ml of chloroform. Extracts, constantly protected from light, were freed from solvent in a rotary evaporator at 30°C under vacuum. The residual lipids were taken up in 10 ml of hexane and preserved -20°C.

Dried carrots (which underwent pretreatment and drying): Two grams of dry sample were collected from a few dried slices crushed in a blender, rehydrated in 50 ml of distilled water for 60 min darkness. The rehydrated sample was subjected to the same extraction procedure as defined for the pre-treated carrots.

The separation of the lipid compound and identification were made by combining several conventional techniques:

- The thin layer chromatography of silica 60 (Merck F254, 0.25 mm thickness of the layer) with visualization by a variety of reagents, including the mixture and aldehyde, acetic acid, and sulfuric acid (Lichfield, 1972). Although destructive, it allows us to compare results to standard commercial substances (press cocoa butters) and identify different classes of lipids. Tocopherols appeared with reducing reagents: dipyridile and ferric chloride. Carotenoids were estimated by specific colorimetry. The thin layer chromatography (ether eluent mixture oil diethyl/ether 87:12 and acetic acid) showed that the staining of the lipid extracts was exclusively due to the carotenoids, in agreement with literature data on dominant pigments carrot;
- The lipid extract was analysis by gas chromatography (Chaveron and ADENIER, 1980). Separations were conducted on a chromatograph (GIRDEL, series 3000) equipped with a flame ionization detector and a capillary column of fused silica packed with apolar phase DB1 JW (polydimethylsiloxane, internal diameter: 0.32 mm, 7m, temperature maximum: 350°C). The chromatograph is connected to an integrator (Waters 746, Millipore). The operating conditions are as follows: carrier gas: Helium, 1 bar; temperature injection (a needle): 375°C, is right 10C.min-1. All lipids were thus separated into different classes, including: free fatty acids, free sterols, diglycerides, esterified sterols and triglycerides. The identification of free sterols, tocopherols, diglycerides and triglycerides is provided by co-injection of the respective commercial control compounds. The quantification of lipid compounds is effected with the aid of an internal standard, adding to the total lipid extract a known amount of cholesterol.
- In going to these lipid extracts, α -tocopherol was well separated from campesterol. Its identification was confirmed by mass spectrometry (SSQ FINNIGAN device 710, electron ionization 70 eV). It should be noted that the diglycerides (DG) mainly correspond to those initially present in the extract; but they can also result from a decomposition of phospholipids (PL) into the chromatograph.

b) Carotene Measurement

- Spectrophotometry: The absorbance at 450 nm of the lipid extract in a solvent methanol / chloroform (Wolf, 1968) was measured by spectrometry (SPECTRONIC GENEYS 5) and compared to a reference range achieved between 0 and 5g.l-1 b Commercial carotene (SIGMA)

2.3.6 Scanning electron microscopy (SEM)

Microstructure of dried, pre-treated or rehydrated product was observed by scanning electron microscopy SEM. Prior to SEM observations, fresh samples were maintained in a desiccator of silica gel for 48 h. Dried samples were directly processed. Samples were stained with gold nanoparticles and observed with a JEOL JSM 840 electron microscope.

III. RESULTS

The main results were combined together in order to identify the impacts of processing parameters of various pretreatment operations on the drying and rehydration kinetics, and the product quality attributes. Structural impacts were also studied because of their direct impact on different physical characteristics such as density, porosity ratio, water activity...

By studying these different pretreatment methods, it should have been possible to identify the possible correlations between modifications of structure and the attributes of drying process, rehydration capacity and physical and chemical product attributes.

3.1 Impact of pretreatments on dehydration time

To investigate the effect of blanching, freezing and DIC pretreatments on vacuum drying, time required for vacuum drying of differently pretreated carrots was registered. Comparison of obtained results showed that systematically the pre-treated samples required lower drying time than the control. The lowest drying time was obtained with intermediate 20-min freezing/thawing pretreatment. Thermo-mechanical pretreatment also decreased dehydration times to a similar level of freezing/thawing pretreatments. Table 1 shows that the best conditions for reaching the fastest drying were obtained with 0.25 MPa saturated steam pressure and instantaneous drop of pressure (DIC) rather than a slow progression toward the atmospheric pressure (Steaming).

TABLE 1
EFFECT OF VARIOUS PRE-TREATMENTS ON TIME REQUIRED FOR CARROT DRYING

Type of pretreatment	Conditions	Drying time (min)
No treatment	Control	221
Blanching	Atmospheric vapor 12 min	122
Blanching prior to Freezing/thawing	Fast Freezing	70
Blanching prior to Freezing/thawing	Intermediate Freezing	50
Blanching prior to Freezing/thawing	Slow Freezing	63
Blanching prior to Freezing/thawing	Very slow freezing	58
No treatment	Control	247
Steaming toward atmospheric pressure	0.25 MPa	119
Steaming toward atmospheric pressure	0.35 MPa	107
Steaming toward atmospheric pressure	0.45 MPa	117
Steaming toward atmospheric pressure	0.55 MPa	113.5
DIC	0.25 MPa	102
DIC	0.35 MPa	71
DIC	0.45 MPa	92
DIC	0.55 MPa	86

3.2 Physical changes during carrot drying

3.2.1 Effect on rehydration capacity

TABLE 2.
EFFECT OF VARIOUS PRE-TREATMENTS ON TIME REQUIRED FOR DRIED CARROT REHYDRATION

Type of pretreatment	Conditions	Capacity of rehydration W_t at time t Expressed in (g H ₂ O/100 g dry basis)		
		t=1 min	t=10 min	t=20 min
No treatment	Control	121.4	259.85	390
Blanching	Atm. Pressure for 12 min	248	556.27	670.5
Blanching prior to Freezing/thawing	Fast Freezing	234	497.86	576.75
Blanching prior to Freezing/thawing	Intermediate Freezing	397.4	760.6	949.5
Blanching prior to Freezing/thawing	Slow Freezing	403	625.73	710.5
Blanching prior to Freezing/thawing	Very slow freezing	434	749.7	897.5
No treatment	Control	165	466	592
Steaming toward atmospheric pressure	0.25 MPa	165.8	515.35	623.98
Steaming toward atmospheric pressure	0.35 MPa	184.5	487.55	529.55
Steaming toward atmospheric pressure	0.45 MPa	232.15	521.54	601.82
Steaming toward atmospheric pressure	0.55 MPa	185.8	465	508.224
DIC	0.25 MPa	249.9	628.49	689.76
DIC	0.35 MPa	298.2	571.5	622.6
DIC	0.45 MPa	251.4	532	573.2
DIC	0.55 MPa	248.5	534.8	556.39

Rehydration capacity is an important functional quality parameter of the dried product from consumer point of view for acceptance of dried products. Results showed that all pre-treatments including blanching, freezing/thawing, DIC, and steaming had a positive effect on rehydration capacity measured at different time intervals (Table 2). Moreover, results on rehydration perfectly correlated with those obtained for dehydration. Indeed, similar to the effect observed on the drying process, the intermediate freezing/thawing pretreatment allowed obtaining the best rehydration capacity. Similar impact was noted with DIC thermo-mechanical pre-treatment at 0.25 MPa as saturated steam pressure (Table 2).

3.2.2 Effect on apparent density and porosity ratio

A decrease in density generally accompanies the pretreatment and the drying process. As results shown in Table 1, pre-treatments reduced the product density to more than 50% in intermediate, slow and very slow freezing with a similar effect for DIC pre-treatment. The lowest density values were obtained when samples were subjected to an instantaneous drop of pressure at, initially saturated steam pressure of 0.25 MPa. While porosity value for non-treated carrot was ranged from 8 to 13%, its values were ranged from 37 to 72% for differently treated samples. The higher the porosity ratio, the lower the drying time and the higher the rehydration capacity.

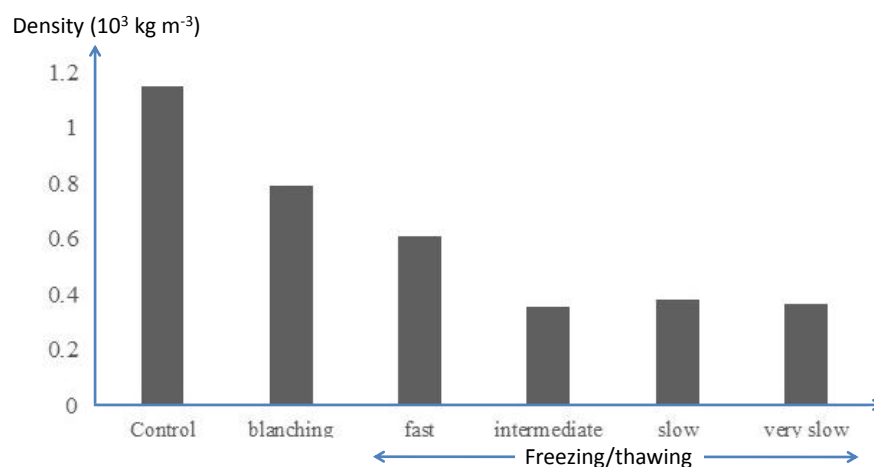


FIGURE 2.EFFECT OF VARIOUS PRE-TREATMENT OPERATIONS ON DENSITY: CONTROL, BLANCHING, AND BLANCHING FOLLOWED BY FREEZING/THAWING.

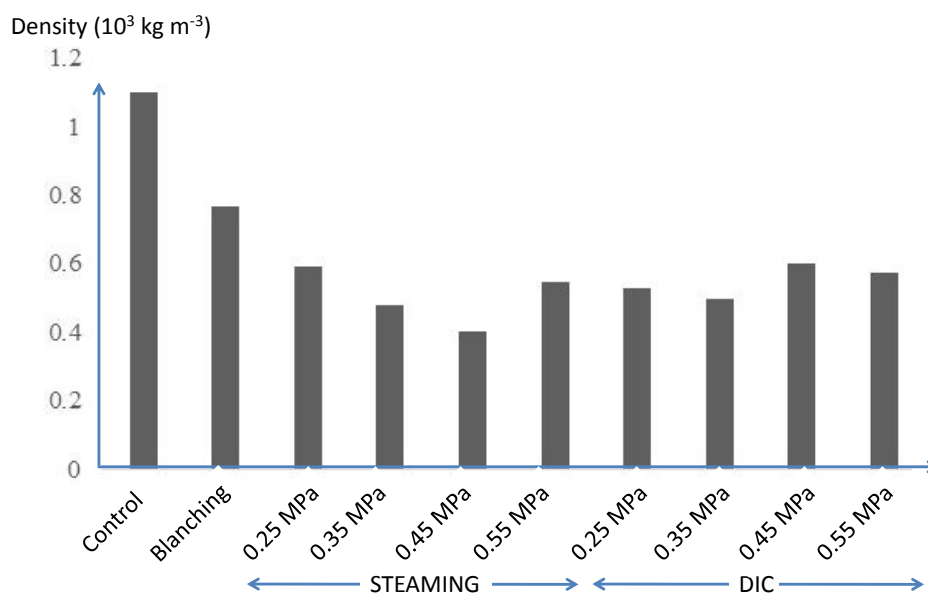


FIGURE 3.EFFECT OF VARIOUS PRE-TREATMENT OPERATIONS ON DENSITY: CONTROL, STEAMING (120 S FOR DECOMPRESSION TIME), AND DIC (0.02 S AS PRESSURE DROP TIME).

3.2.3 Effect on carrot content

Dehydration of vegetables is usually escorted by losses of lipids and carotenes due to thermal treatment and or leaching during blanching. The effect of various pretreatments were assessed for their influence on lipid content notably for phospholipids and diacylglycerols and provitamin A. Results showed that both freezing/thawing pretreatment and DIC pre-treatment coupled to vacuum drying had in general a good effect on preserving lipid content compared to controls (Figure 4). In each type of treatment, an average value has shown its effectiveness in relation to other conditions. For freezing/thawing, a better preservation of phospholipids and diacylglycerols was produced by slow freezing pretreatment. By contrast, provitamin A is preserved during fast freezing explained probably by a weak diffusion of pigments and a reduced exposition of the product to the heat during the drying process. For thermo-mechanical pretreatments, results were similar to our previously published results and the best effect was obtained 0.45 MPa of saturated steam pressure with instantaneous drop of pressure toward vacuum (DIC conditions)(DEBS-LOUKA et al., 1996).

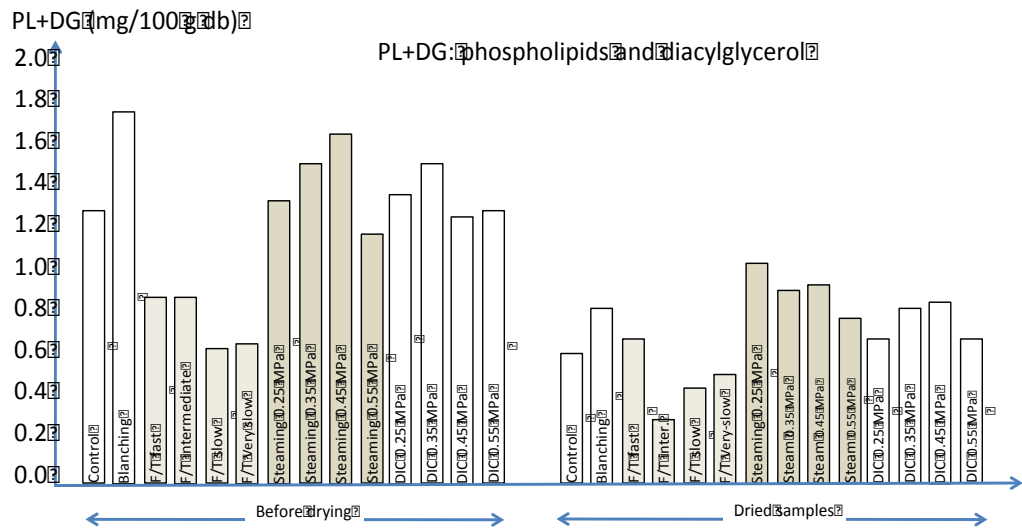


FIGURE 4.EFFECT OF VARIOUS PRE-TREATMENT OPERATIONS ON PL+DG: PHOSPHOLIPIDS AND DIACYLGLYCEROL: CONTROL, BLANCHING, STEAMING (120 S FOR DECOMPRESSION TIME), AND DIC (0.02 S AS PRESSURE DROP TIME).

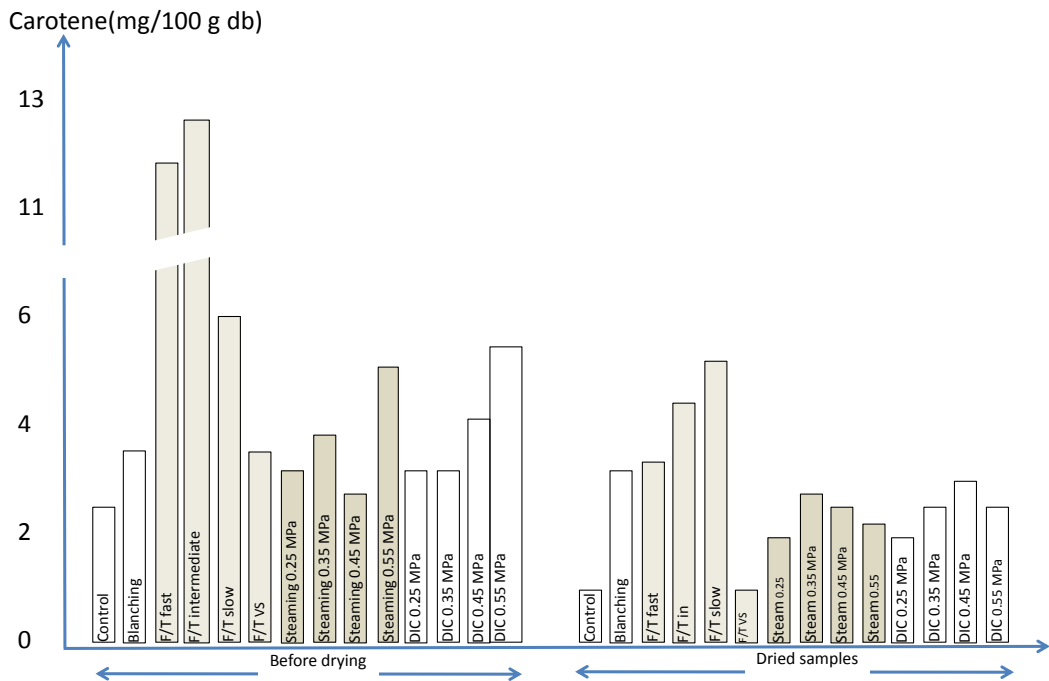


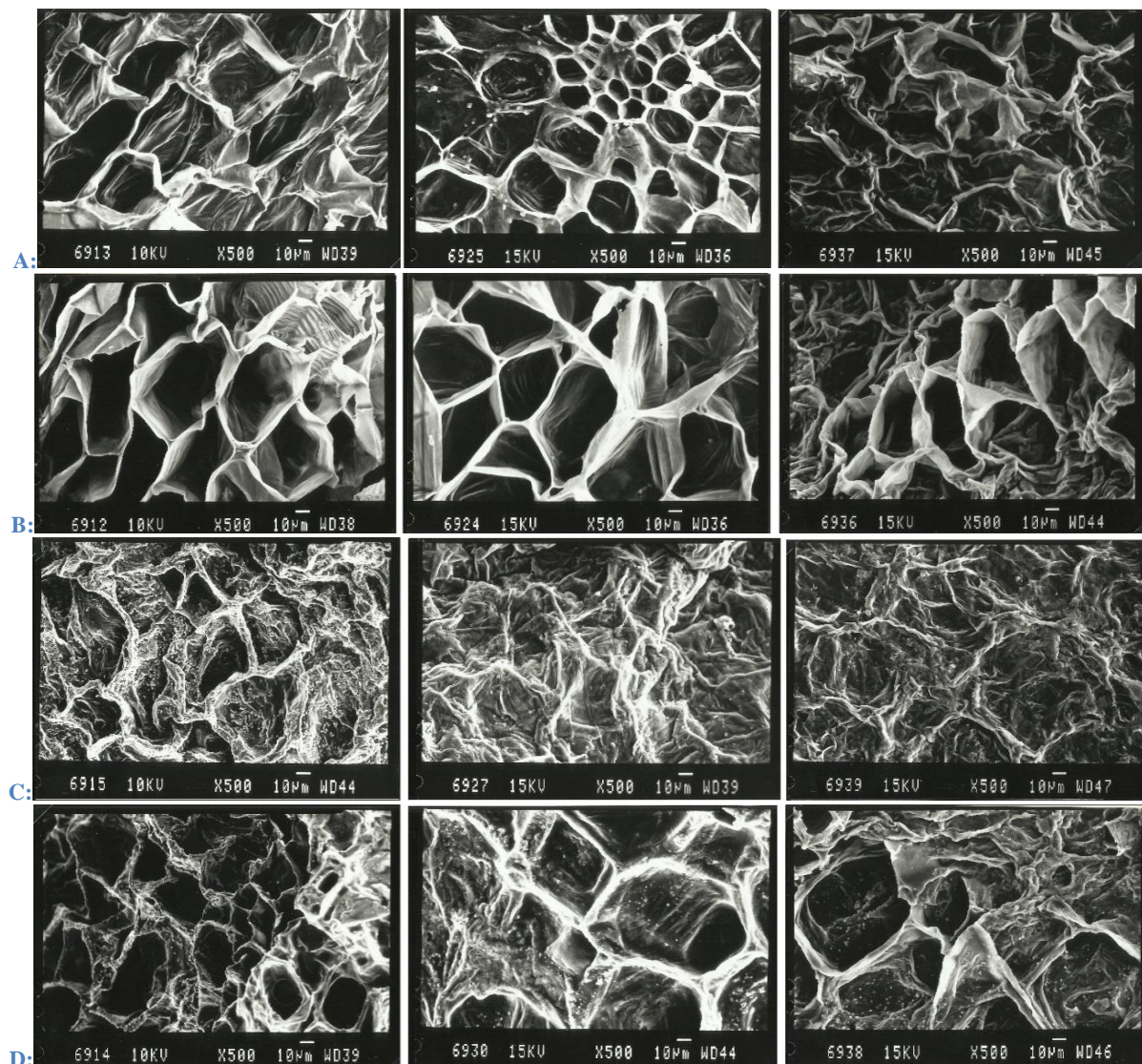
FIGURE 5.EFFECT OF VARIOUS PRE-TREATMENT OPERATIONS ON CAROTENE CONTENT: CONTROL, BLANCHING, STEAMING (120 S FOR DECOMPRESSION TIME), AND DIC (0.02 S AS PRESSURE DROP TIME).

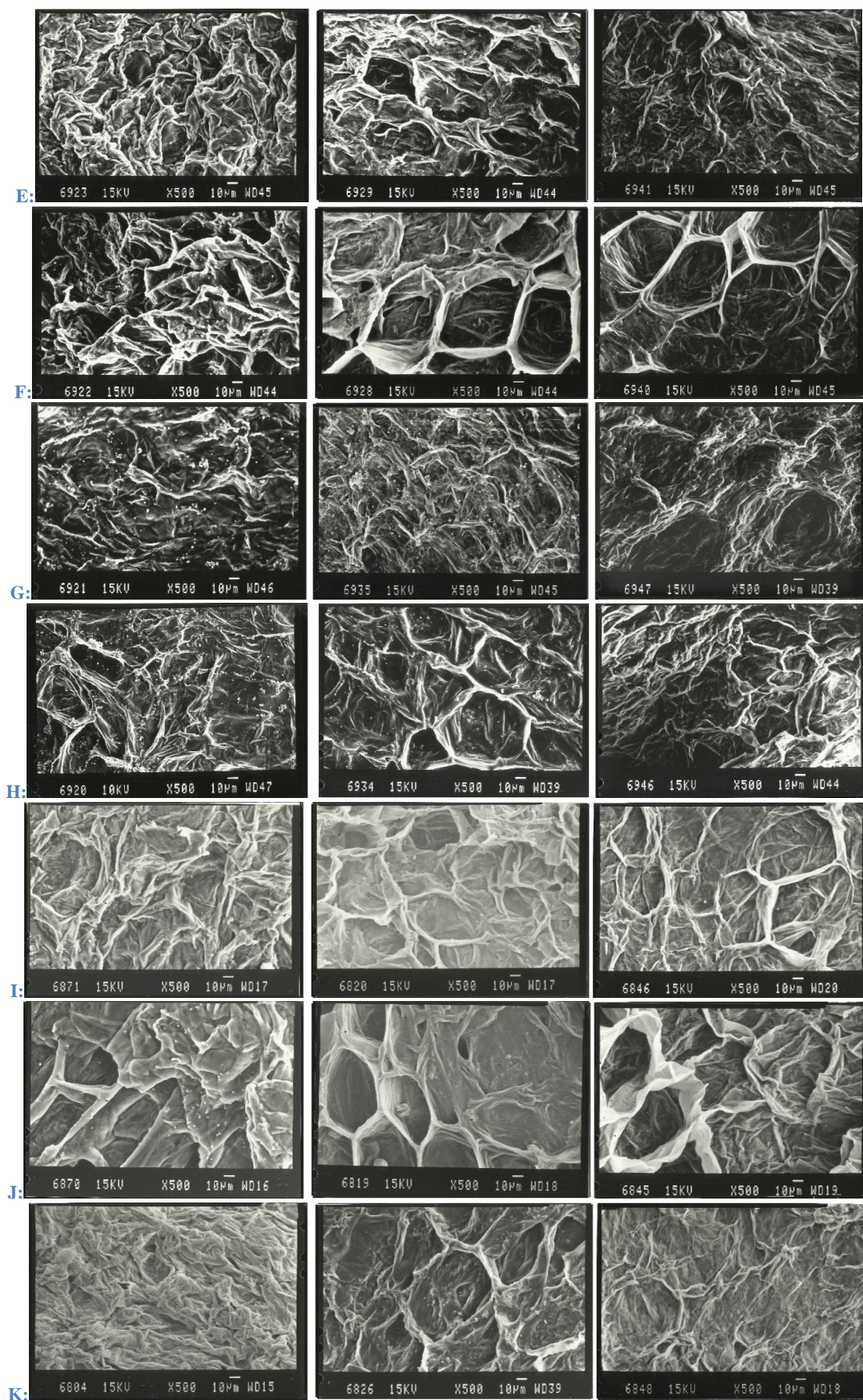
3.2.4 Microstructure changes affected by various pre-treatments

SEM analyses were used to identify the effect of various pretreatments on microstructure (Figure 6). Results show that control carrots have polyhedral cells of similar size filled with starch and lipid droplets indicating a normal organization of the vegetal tissue (Fig. 6.A, Fig 6.B)

Immediate SEM analyses after blanching and drying or after rehydration, show that cell structures undergo large changes compared to the control (Fig 6.C). The rigidity of the xylem was enhanced by the presence of tracheid (Fig 6.D).

Freezing/thawing had more impact on the phloem rather than on the xylem (Fig 6.E, Fig 6.G). The latter still maintains the integrity of its cells with a larger number of intercellular spaces (Fig 6.F, fig 6.H). Rapid freezing provides very slight modifications compared to blanching with a greater preservation in xylem cells than those of the phloem; the latter seems slightly narrowed with thinner cell walls. This deformation increased with the duration of freezing, the cells became more and more mechanically damaged (Fig 6.E, Fig 6.F) and increasingly frequent intercellular spaces are observed in xylem (Fig 6.G, Fig 6.H). Freezing/thawing prior to the drying induced more dilation and cellular breakdown than a blanching pretreatment especially after a long freeze-thawing cycle, and thus, it facilitates the diffusion of water within the cells during rehydration. Mechanical damage as well as cellular changes weaken the bounds between the matrix and lipids, had little or no effect for fast or intermediate freezing/thawing. The lipid availability decreased due to a larger cell change resulting from slow freezing causing diffusion during sample processing. After drying, lipid had relatively lower levels than only blanched samples. This should be due to the reduced dehydration time. The cell change was less important for fast freezing, and since pigments diffuse less outside for slow freezing, it results in a preservation of the pigments.





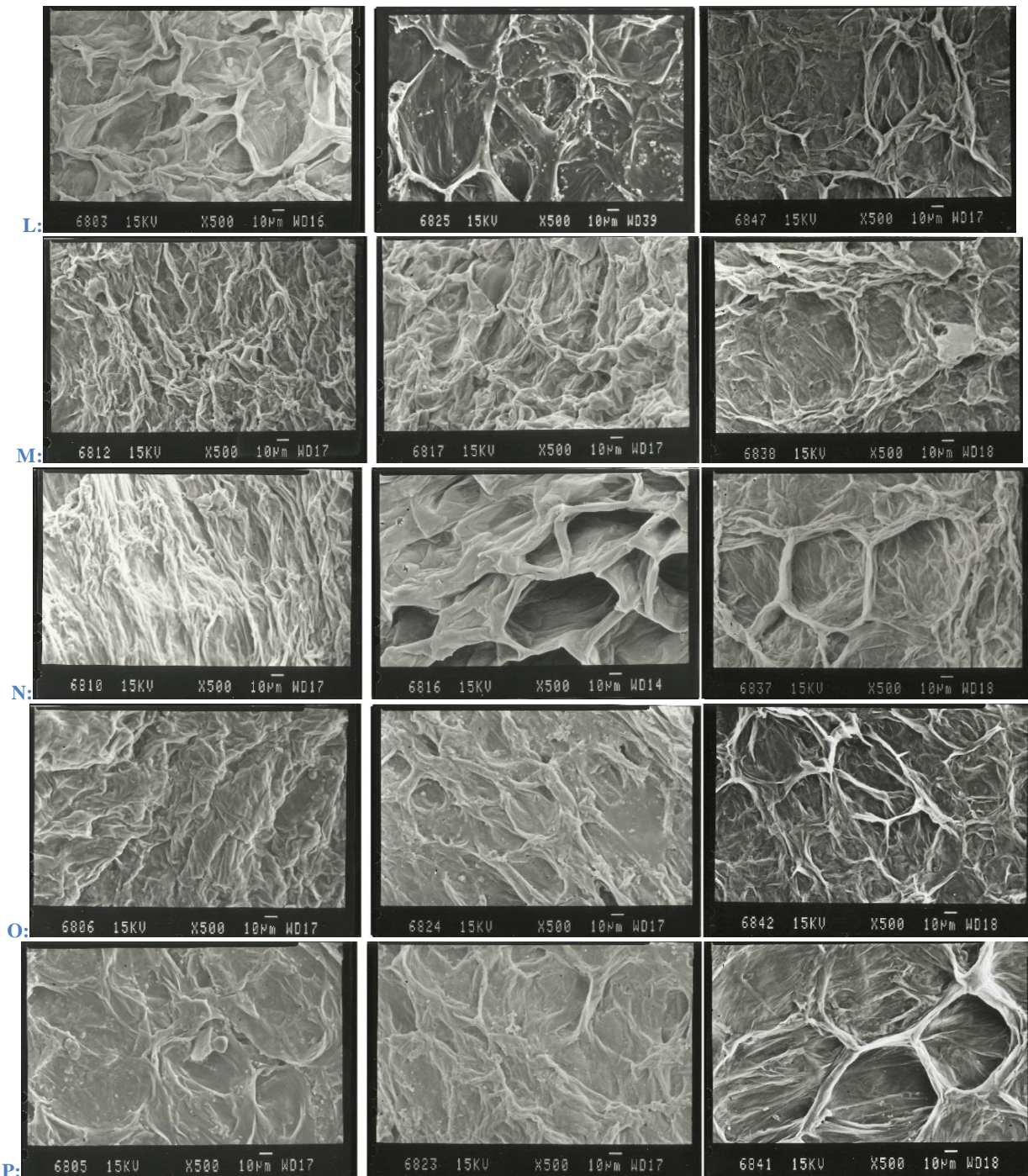


FIGURE 6.EFFECT OF VARIOUS PRE-TREATMENT OPERATIONS ON MICROSTRUCTURE:CONTROL CARROT: PHLOEM A.1) FRESH, A.2) DEHYDRATED, AND A.3) REHYDRATED. // XYLEM B.1) FRESH, B.2) DEHYDRATED, AND B.3) REHYDRATED.CARROT SUBJECTED TO BLANCHING: PHLOEM C.1) PRETREATED, C.2) DEHYDRATED, AND C.3) REHYDRATED. // XYLEM D.1) PRETREATED, D.2) DEHYDRATED, AND D.3) REHYDRATED.CARROT SUBJECTED TO F/T INTERMEDIATE: PHLOEM E.1) PRETREATED, E.2) DEHYDRATED, AND E.3) REHYDRATED. // XYLEM F.1) PRETREATED, F.2) DEHYDRATED, AND F.3) REHYDRATED.CARROT SUBJECTED TO F/T V.S: PHLOEM G.1) PRETREATED, G.2) DEHYDRATED, AND G.3) REHYDRATED. // XYLEM G.1) PRETREATED, G.2) DEHYDRATED, AND G.3) REHYDRATED.CARROT SUBJECTED TO DIC 0.35 MPa: PHLOEM I.1) PRETREATED, I.2) DEHYDRATED, AND I.3) REHYDRATED. // XYLEM J.1) PRETREATED, J.2) DEHYDRATED, AND J.3) REHYDRATED.CARROT SUBJECTED TO STEAMING 0.35 MPa: PHLOEM K.1) PRETREATED, K.2) DEHYDRATED, AND K.3) REHYDRATED. // XYLEM L.1) PRETREATED, L.2) DEHYDRATED, AND L.3) REHYDRATED.CARROT SUBJECTED TO DIC 0.55 MPa: PHLOEM M.1) PRETREATED, M.2) DEHYDRATED, AND M.3) REHYDRATED. // XYLEM N.1) PRETREATED, N.2) DEHYDRATED, AND N.3) REHYDRATED.CARROT SUBJECTED TO STEAMING 0.55 MPa: PHLOEM O.1) PRETREATED, O.2) DEHYDRATED, AND O.3) REHYDRATED. // XYLEM P.1) PRETREATED, P.2) DEHYDRATED, AND P.3) REHYDRATED.

Microscopic observation of DIC pretreated products highlights the direct impact of this pretreatment on the cell structure. Results show that the change rate directly depended on the adopted operating conditions. Before drying, samples pretreated by DIC at low pressure show an evolution directly linked to the duration of the pretreatment. This development is located on the two cell levels of xylem and phloem.

After DIC treatment at 0.15 MPa for 15 min, cell phloem and even xylem swelled following dissolution of the middle lamella. In the xylem, adjacent cells to tracheid were better preserved. Macroscopic analyses show that under identical processing conditions, DIC pretreatment caused, from a certain steam pressure, holes and cell bursts between the two regions of xylem and phloem appeared and were significantly larger than steaming process. This modification is well observed more at the phloem than in xylem (Fig 6.I, Fig 6.J, Fig 6.K, Fig 6.L). For steam pressures higher than 0.35 MPa, DIC resulted in more frequent intercellular spaces but significantly more localized in phloem than in xylem (Fig 6.M, Fig 6.N, Fig 6.O, Fig 6.P).

3.3 Main correlations

By assembling the different experimental data obtained from the considered pretreatment operations, it was possible to classify chemical composition (DG: diglycerides; PL: phospholipids; carotene content), matrix structure, porosity ratio, water activity with kinetics of vacuum drying and rehydration.

Table 3 allows specifying these data versus the pretreatment operations (blanching, freezing/thawing, steaming, and instant controlled pressure drop DIC) and their respective processing parameters. Dehydration time ranged from 50 to 122 min for differently pretreated carrot samples against about 234 ± 13 min for controls (absence of pretreatment).

TABLE 3
PARAMETERS OF PRETREATMENT OPERATIONS, DRYING TIME AND CHARACTERISTICS OF FINAL PRODUCTS

	F/T time (min)	Steam pressure (MPa)	Decompression time (s)	$W_{1\text{ min}}$ (g H ₂ O/100 g db)	Drying time (min)	Density	□ : porosity ratio	PL + DG (p)	PL + DG (dh)	Carotene (p)	Carotene (dh)	$W_{aw=0.1}$	$W_{aw=0.2}$
Control 1				121	221	1.15	8%	2.6	0.8	1.3	0.6	5.7	10.4
Blanching	(12 min)			248	122	0.79	37%	2.59	0.77	0.77	2.6	0.8	0.8
F/T	10 min			234	70	0.61	51%	11.5	3.3	0.9	0.7	8.8	11.3
F/T	40 min			397	50	0.36	72%	13.0	4.5	0.9	0.3	8.6	11.9
F/T	10 ² min			403	63	0.38	70%	5.9	5.1	0.6	0.4	10.3	12.6
F/T	10 ³ min			434	58	0.37	71%	3.4	1.1	0.7	0.5	10.6	12.7
Control 2				165	247	1.10	13%	3.2	0.8	1.4	0.6	1.8	3.7
Steaming		0.25	120	166	119	0.59	53%	3.2	1.9	1.3	1.0	9.6	16.3
Steaming		0.35	120	185	107	0.48	62%	3.8	2.8	1.5	0.9	9.2	16.1
Steaming		0.45	120	232	117	0.40	68%	2.7	2.6	1.7	0.9	5.6	10.9
Steaming		0.55	120	186	114	0.55	57%	5.0	2.2	1.2	0.7	10.1	18.6
DIC		0.25	0.2	250	102	0.53	58%	3.1	1.9	1.4	0.7	10.0	16.6
DIC		0.35	0.2	298	71	0.50	60%	3.1	2.5	1.5	0.8	8.2	16.0
DIC		0.45	0.2	251	92	0.60	52%	4.2	2.9	1.3	0.9	10.1	19.2
DIC		0.55	0.2	249	86	0.57	55%	5.4	2.6	1.3	0.7	7.5	14.1

TABLE 4
CORRELATIONS MATRIX BETWEEN PARAMETERS OF PRETREATMENT OPERATIONS, DRYING TIME AND CHARACTERISTICS OF FINAL PRODUCTS.

Correlationcoef	F/T	Steam pressure	Decompression time	Drying time	$W_{1\text{ min}}$	Density	9: porosity ratio	PL + DG (dh)	Carotene (p)	$W_{aw=0.1}$
F/T	1	-	-	-0.2	0.55	-0.40	0.40	-0.87	-0.56	0.70
Steam pressure	-	1.0	0.0	-0.1	0.1	0.0	0.0	0.5	-0.3	-0.3
Decompression time	-	0.0	1.0	0.8	-0.8	-0.4	0.4	-0.2	0.2	-0.1
Drying time (min)	-0.2	-0.1	0.8	1.0	-0.7	0.9	-0.9	-0.7	0.4	-0.7
$W_{1\text{ min}}$	0.5	0.1	-0.8	-0.7	1.0	-0.7	0.7	0.5	-0.7	0.4
Density	-0.4	0.0	-0.4	0.9	-0.7	1.0	-1.0	-0.7	0.2	-0.6
9: porosity ratio	0.4	0.0	0.4	-0.9	0.7	-1.0	1.0	0.7	-0.2	0.6
PL + DG (dh)	-0.9	0.5	-0.2	-0.7	0.5	-0.7	0.7	1.0	-0.2	0.3
Carotene (p)	-0.6	-0.3	0.2	0.4	-0.7	0.2	-0.2	-0.2	1.0	-0.5
$W_{aw=0.1}$	0.7	-0.3	-0.1	-0.7	0.4	-0.6	0.6	0.3	-0.5	1.0

9: porosity ratio; Freezing/Thawing operation; : porosity ratio; DG: diglycerides mg/100g db; PL: phospholipids mg/100g db; carotene content mg/100g db; $W_{1\text{ min}}$: Rehydration capacity after 1 min soaking; $W_{aw=0.1}$: Water content at $aw=0.1$, both expressed in g H₂O/100 g db; Steaming: saturated steam at high pressure (0.25 to 5.5 MPa) with progressive decompression toward atmospheric pressure in 120 s. DIC: Instant Controlled Pressure Drop with saturated steam treatment at high pressure (0.25 to 5.5 MPa) with abrupt pressure drop toward a vacuum of 5 kPa in 0.2 s.

Linear correlation matrix was used to identify the correlation coefficient (

Table 4). Various positive or negative values of bilateral correlations could be obtained between operating parameters of pretreatment operations (Freezing/thawing time, steam pressure and decompression time of both steaming and DIC operations (120 s and 0.02 s, respectively)) on one hand, and the main functional, and structural/physical properties of the final products, on the other hand.

Other types of correlations was established between the product characteristics (Drying time vs 9: porosity ratio and vs water content at water activity $aw=0.1$; rehydration capacity expressed in “water content dry basis: g H₂O/100 g db” for 1 min ($W_{1\text{ min}}$) vs 9: porosity ratio and vs water content ($W_{aw=0.1}$) at water activity $aw=0.1$).

Drying time is correlated with the type of pretreatment operations as well as their respective processing parameters. The highest effect resulted from the decompression time with thermo-mechanical pretreatment methods (Steaming and DIC). The shorter the decompression time, the shorter the drying time.

Drying time is also correlated with porosity 9 and water content at water activity $aw=0.1$. The higher the water activity, the shorter the drying time (Figure 7). Similar correlation with much higher correlation coefficient ($R^2=0.82$) was obtained between drying time and the porosity ratio J; the higher the porosity, the shorter the drying time (Figure 8).

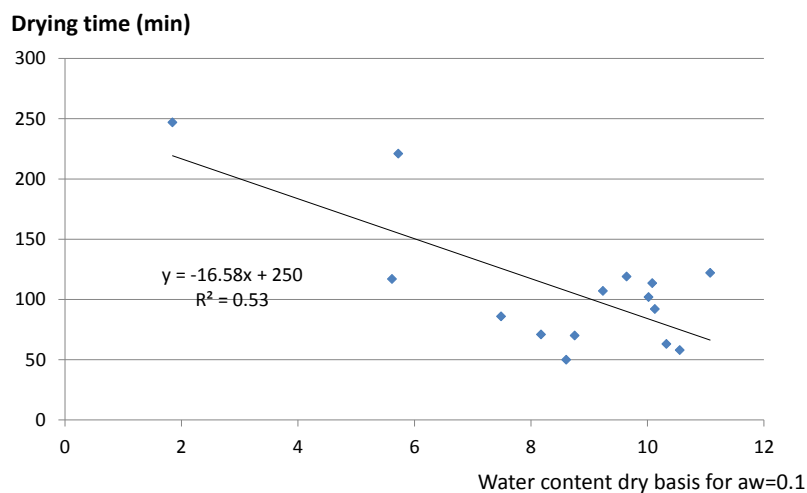


FIGURE 7. LINEAR CORRELATION REVEALING THE GENERAL EVOLUTION OF DRYING TIME VERSUS WATER ACTIVITY (WATER CONTENT AT AW=0.1).

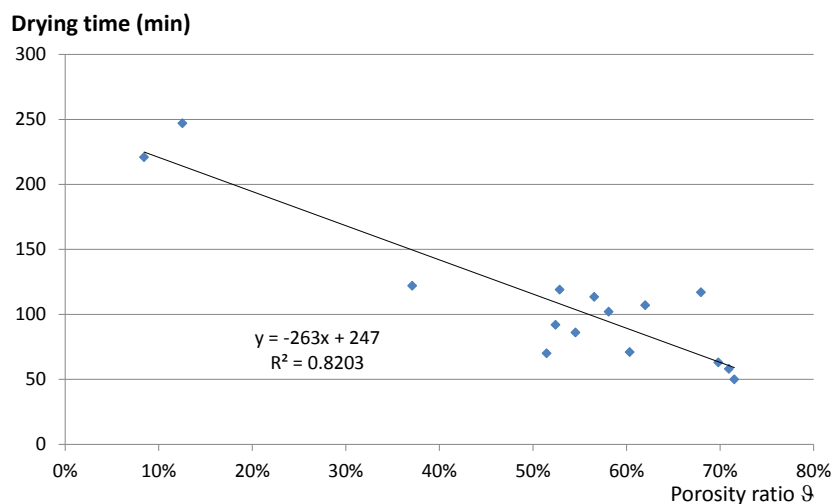


FIGURE 8. LINEAR CORRELATION REVEALING THE GENERAL EVOLUTION OF DRYING TIME VERSUS POROSITY RATIO ϑ .

Similar correlation could normally be established between porosity ratio ϑ and rehydration level (here after 1 min soaking).

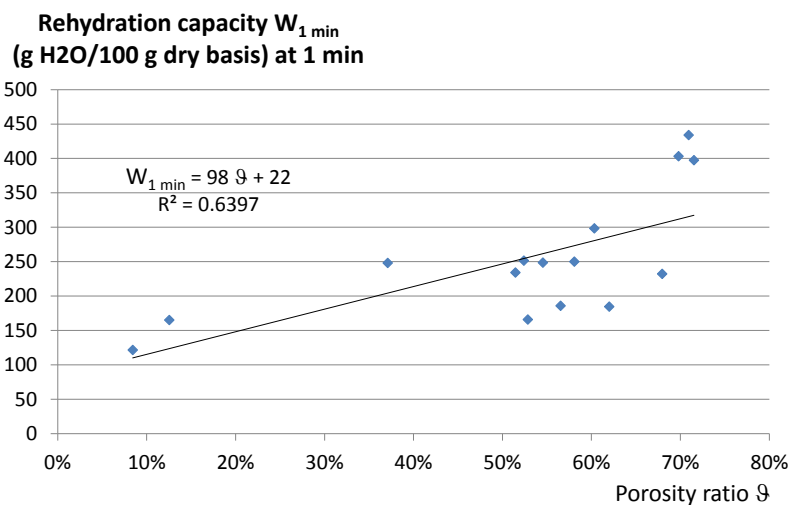


FIGURE 9. LINEAR CORRELATION REVEALING THE GENERAL EVOLUTION OF DRYING TIME VERSUS POROSITY RATIO ϑ .

IV. CONCLUSION

The current study compared different types of pretreatment operations; thermal (bleaching, freezing, and steaming) and thermomechanical (instant controlled pressure drop DIC) on vacuum drying process. This drying operation was chosen in order to greatly reduce the impact of properly said drying conditions, on the final quality. Different correlations were established between the drying and physical parameters of the obtained product. A good correlation was obtained between the increase in initial rehydration rate with a decrease in drying time and density. We note that freezing stands on the top of the pretreatments performed directly followed by thermo-mechanical pretreatment achieved with an instant drop of pressure (DIC).

The overall results show the need for pretreatment prior to drying operation in order to relatively preserve lipid and carotene contents. Microscopic study highlights the enormous impact of the structure change on the behavioral properties of the products and the biochemical properties. Thus, experimental results show that increasing the availability of lipids was often correlated to cellular changes obtained after various types of pretreatment. At this stage, the pretreatments mainly acted at the level of the cell structure systematically on the phloem rather than the xylem. Blanching, freezing and DIC as optimized pretreatments act primarily on cell shape and slightly alter the actual cell wall. Drying almost invariably leads to improved cell shape following pretreatments.

REFERENCES

- [1] Albertos, I., Martin-Diana, A.B., Sanz, M.A., Barat, J.M., Diez, A.M., Jaime, I., Rico, D., 2015. Effect of high pressure processing or freezing technologies as pretreatment in vacuum fried carrot snacks. *Innovative Food Science & Emerging Technologies*. doi:10.1016/j.ifset.2015.11.004
- [2] Allaf, T., Allaf, K. (Eds.), 2014. *Instant Controlled Pressure Drop (D.I.C.) in Food Processing*, Food Engineering Series. Springer New York, New York, NY.
- [3] Arévalo-Pinedo, A., XidiehMurr, F.E., 2007. Influence of pre-treatments on the drying kinetics during vacuum drying of carrot and pumpkin. *Journal of Food Engineering* 80, 152–156. doi:10.1016/j.jfoodeng.2006.05.005
- [4] Barat, J.M., Grau, R., 2016. Drying: Principles and Types, in: *Encyclopedia of Food and Health*. Elsevier, pp. 456–461.
- [5] Ben Amor, B., Allaf, K., 2009. Impact of texturing using instant pressure drop treatment prior to solvent extraction of anthocyanins from Malaysian Roselle (*Hibiscus sabdariffa*). *Food Chemistry* 115, 820–825. doi:10.1016/j.foodchem.2008.12.094
- [6] Chen, Z.-G., Guo, X.-Y., Wu, T., 2016. A novel dehydration technique for carrot slices implementing ultrasound and vacuum drying methods. *Ultrasonics Sonochemistry* 30, 28–34. doi:10.1016/j.ultsonch.2015.11.026
- [7] Cui, Z.-W., Xu, S.-Y., Sun, D.-W., 2004. Microwave–vacuum drying kinetics of carrot slices. *Journal of Food Engineering* 65, 157–164. doi:10.1016/j.jfoodeng.2004.01.008
- [8] DEBS-LOUKA, E., Sahyoun, W., Adenier, H., BARBOTIN, J., LOUKA, N., CHABVERON, H., THOMAS, N., Allaf, K., 1996. Déshydratation de la carotte (*Daucus carota* L.) : effet d'un nouveau procédé sur les composés lipidiques et la microstructure = Drying of the carrot (*Daucus carota* L.) : effect of a new process on lipidic compounds and microstructure. *Sca* 16, 491–503.
- [9] Gamboa-Santos, J., Soria, A.C., Villamiel, M., Montilla, A., 2013. Quality parameters in convective dehydrated carrots blanched by ultrasound and conventional treatment. *Food Chemistry* 141, 616–624. doi:10.1016/j.foodchem.2013.03.028
- [10] Garcia-Amezquita, L.E., Welti-Chanes, J., Vergara-Balderas, F.T., Bermúdez-Aguirre, D., 2016. Freeze-drying: The Basic Process, in: *Encyclopedia of Food and Health*. Elsevier, pp. 104–109.
- [11] Jangam, S.V., Mujumdar, A.S., Adhikari, B., 2016. Drying: Physical and Structural Changes, in: *Encyclopedia of Food and Health*. Elsevier, pp. 446–455.
- [12] Kidmose, U., Martens, H.J., 1999. Changes in texture, microstructure and nutritional quality of carrot slices during blanching and freezing. *Journal of the Science of Food and Agriculture* 79, 1747–1753. doi:10.1002/(SICI)1097-0010(199909)79:12<1747::AID-JSFA429>3.0.CO;2-B
- [13] Kocabiyik, H., Tezer, D., 2009. Drying of carrot slices using infrared radiation. *International Journal of Food Science & Technology* 44, 953–959. doi:10.1111/j.1365-2621.2008.01767.x
- [14] Maritza, A.-M., Sabah, M., Anaberta, C.-M., Montejano-Gaitán, J.G., Allaf, K., 2012. Comparative Study of Various Drying Processes at Physical and Chemical Properties of Strawberries (*Fragaria var. camarosa*). *Procedia Engineering* 42, 267–282. doi:10.1016/j.proeng.2012.07.418
- [15] Moses, J.A., Norton, T., Alagusundaram, K., Tiwari, B.K., 2014. Novel Drying Techniques for the Food Industry. *Food Engineering Reviews* 6, 43–55. doi:10.1007/s12393-014-9078-7
- [16] Mounir, S., Allaf, T., Berka, B., Hassani, A., Allaf, K., 2014. Instant Controlled Pressure Drop technology: From a new fundamental approach of instantaneous transitory thermodynamics to large industrial applications on high performance–high controlled quality unit operations. *Comptes Rendus Chimie* 17, 261–267. doi:10.1016/j.crci.2013.10.019
- [17] Negi, P.S., Kumar Roy, S., 2001. The effect of blanching on quality attributes of dehydrated carrots during long-term storage. *European Food Research and Technology* 212, 445–448. doi:10.1007/s002170000279

-
- [18] Niamnuy, C., Devahastin, S., Soponronnarit, S., 2014. Some recent advances in microstructural modification and monitoring of foods during drying: A review. *Journal of Food Engineering* 123, 148–156. doi:10.1016/j.jfoodeng.2013.08.026
- [19] Pedrosa, M.M., Cuadrado, C., Burbano, C., Allaf, K., Haddad, J., Gelencsér, E., Takács, K., Guillamón, E., Muzquiz, M., 2012. Effect of instant controlled pressure drop on the oligosaccharides, inositol phosphates, trypsin inhibitors and lectins contents of different legumes. *Food Chemistry* 131, 862–868. doi:10.1016/j.foodchem.2011.09.061
- [20] Richter Reis, F., 2014. Studies on Conventional Vacuum Drying of Foods, in: Richter Reis, F. (Ed.), *Vacuum Drying for Extending Food Shelf-Life*. Springer International Publishing, Cham, pp. 7–18.
- [21] Téllez-Pérez, C., Sabah, M.M., Montejano-Gaitán, J.G., Sobolik, V., Martínez, C.-A., Allaf, K., 2012. Impact of Instant Controlled Pressure Drop Treatment on Dehydration and Rehydration Kinetics of Green Moroccan Pepper (*Capsicum Annuum*). *Procedia Engineering* 42, 978–1003. doi:10.1016/j.proeng.2012.07.491.