

## OSMOTIC DEHYDRATION OF MANDARINS: INFLUENCE OF REUTILIZED OSMOTIC AGENT ON BEHAVIOUR AND PRODUCT QUALITY

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**Background.** Osmotic dehydration (OD) is a technology that allows the concentration mainly of fruits and vegetables, without change of phase, through partial water removal, when immersed in a hypertonic solution of sugar, salt or others. It can be successfully applied to some products whose production is not fully marketed in fresh form. However, an additional process is necessary to stabilize the product. The process leads to the achievement of high quality alternative products, with an extended shelf-life, economy in storage and transport. The aim of this work was to study, at a pilot scale, the behaviour evaluation of a sucrose dehydration solution, during twelve OD reuses, and the quality of processed mandarins.

**Material and methods.** The process was carried out using mandarins (*Citrus reticulata* Blanco) cv Clementina Nova, from Algarve, Portugal, manually peeled and segments chemically skinned. In assays a 60°Brix sucrose solution was used, conducted in thermo-stabilized baths, at 45°C, 16 h, 40 oscillations per minute and a fruit:solution ratio of 1:2 (m/m). After each OD cycle, the solution was filtered and reconcentrated to 60°Brix by sucrose addition, and adjusted to original volume. The osmodehydrated mandarins were stabilized by pasteurization. The drying solution behaviour and mandarins' quality were assessed through different physical, chemical and microbiological analysis.

**Results.** The factorial discriminate analysis allowed to distinguish a different behaviour between the original and final dehydration sucrose solution during OD processes, but did not affect its desiccant power, only a high pollutant load development explained by BOD<sub>5</sub> values. The results of osmodehydrated mandarins showed that stability was achieved by "combined process" with pasteurization.

**Conclusions.** The results lead to conclude that osmotic dehydration process is a good option to improve mandarin's stabilization, after pasteurization, increasing added value. Its viability is connected to the management of the drying solution and the number of uses

depends on the type of product being dehydrated and their processing sequence. Though the negative microbial load, it must be given attention to dilute the solution before its release as effluent.

**Key words:** dehydrated mandarins, combined process, reuse sugar solution, quality parameters, stability

## INTRODUCTION

The osmotic dehydration process (OD), also named dewatering and impregnation by immersion in concentrates (DII), is a very gentle method for a partial water removal from plant tissues, by immersion of food in concentrated solutions or syrups of soluble solids, without phase change. The complex cell wall structure of the food acts as a semi-permeable membrane, which is not completely selective, resulting in two counter-current mass transfer flows: diffusion of water from food to solution and diffusion of solute from solution to food.

This method is used mainly for the preservation of fruits or vegetables, to obtain several kinds of products such as minimally processed or intermediate moisture content products, or as a pre-treatment combined with an additional classic process, to have a stabilized food at room temperature. It offers interesting perspectives for the preservation, because it allows, not only the achievement of food of high quality, but also economy at storage and transportation, and increase in added value. Moreover, this process is very suitable for fruit showing a short harvest period, with concentration of supply in a particular time of year and sensitive to storage conditions [Themelin et al. 1995, Garcia-Luna-Muñoz and Ryan 1997]. It is easily adaptable to small and medium scale avoids changes induced by heat and preserves most of the quality of fresh product. The selection of varieties and their degree of ripeness are crucial to the effectiveness of the process.

The viability of this process, at industrial level, is connected to the management of the drying solution, considered the main limiting factor, because this becomes gradually diluted, after each processing, lowering the rate of mass transfer, and could compromise its effect on the final product [Dalla Rosa and Giroux 2001, García-Martínez et al. 2002, Valdez-Fragoso et al. 1998, 2002 a, b]. One possible option is recycling dehydration solution, where the solution used in the process is filtered, brought to initial concentration and reused. The number of uses depends on the product being dehydrated and the sequence of processing, required volume (ratio fruit:solution), type of filter used, type of reconcentration and presence or absence of steps to sanitation [García-Martínez et al. 2002, Ferreira et al. 2003]. So, we must take into account not only the cost of producing solutions, but also the elimination of large volumes of sewage with high content of organic matter in sugars and other components.

Following previous studies about mandarins [Sapata et al. 2000 a, b, 2003, 2004, 2005, Ferreira et al. 2003, Leitão et al. 2003], this study aimed to evaluate the behaviour of a sucrose dehydration solution, during twelve OD cycles and the quality of processed mandarins, at a pilot scale, through physical, chemical, microbiological and sensorial parameters.

## MATERIAL AND METHODS

In assays, a mandarin (*Citrus reticulata* Blanco) cv Clementina Nova was used, from Algarve – Portugal, selected for its display size and consistency of the buds and lack of seeds. The fruits were placed in a cooling chamber before processing. Uniform ripe fruits were washed for removal of dust or pesticide residues. Then they were manually peeled and separated segments were chemically skinned by immersion in a solution of HCl 0.5% (40 min at 30°C) followed by immersion in 0.15% NaOH solution (20 min at 25°C) and rinsed in tap water for 60 min [Luth and Kean 1975].

Osmotic dehydration was carried out in a 60°Brix solution of sucrose. The OD cycles were conducted in thermo stabilized baths, with stainless steel vats with 20 L capacity and agitation of 40 oscillations per minute. The procedures were performed at a temperature of 45°C for 16 h, a fruit:solution ratio of 1:2 (m/m), according to the criteria established in previous studies [Sapata et al. 1999, 2000 a]. Then the products were washed with water and drained, and pasteurized (70°C, 30 min) [Sapata et al. 2002] (Fig. 1).

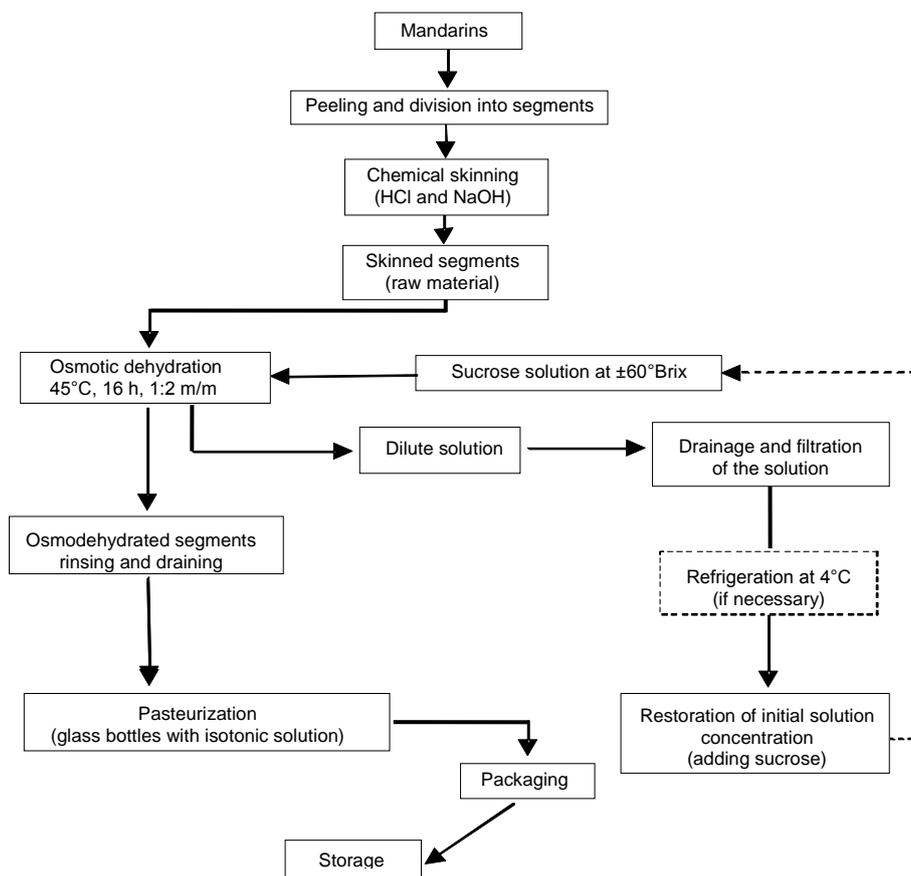


Fig. 1. Mandarins osmotic process flow chart

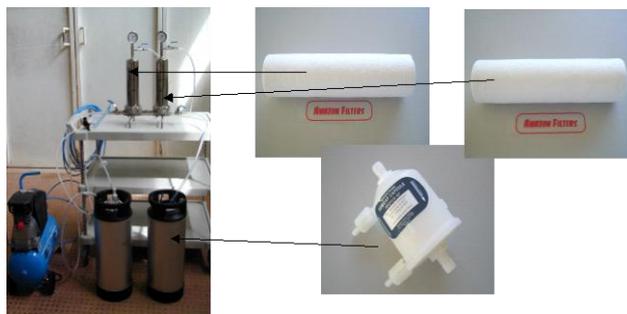


Fig. 2. Filtration system used after each OD cycle

After each cycle the solution was filtered through a pressurized system Amazon Filters Ltd. equipped with 2-p polipropylene Supagard of 5 and 1  $\mu\text{m}$ , with isolation valve and a biological glass filter microfibrer Demicap-Prepor GF of 0.6  $\mu\text{m}$ . After filtration, the solution was reconcentrated to 60°Brix, by addition of sucrose, and adjusted to original volume. The operations were done with the filtered desiccant solution at a temperature of 40°C, and the sanitation system with water at 50-55°C (Fig. 2).

#### Evaluation of the quality of drying solution and osmodehydrated fruits

**Drying solution.** *Physical and chemical analysis:* soluble solids content (°Brix) performed by a hand-held refractometer ATAGO PR 301, pH measured with a potentiometer CRISON Micro pH 2002 with glass electrode, colour evaluated by transmittance, using a Minolta Croma Meter CT 310, electrical conductivity by conductivimeter InoLab Cond Level 2 (results expressed in  $\mu\text{S}/\text{cm}$ ), turbidity using a turbidimeter Turbiquant 1500 IR (results expressed in TNU), viscosity by viscometer Brookfield, model DV – II, with spindle LV n.1 (results expressed in cP). *Microbiological analysis:* from each sample solution desiccant (1 mL), decimal dilutions were made with Maximum Recovery Diluent (Oxoid CM 733). Serial dilutions were assessed in tubes before planting. The media and incubation conditions were: total aerobic mesophilic flora – PCA (Oxoid CM 325), 30°C 48 h, xerophilic moulds – DG 18 Agar (Oxoid CM 729), 25°C, 5 days, osmophilic yeasts – Tilbury medium [Tilbury 1976] 37°C, 48 h and lactic flora – MRS Agar (Oxoid CM 361), 37°C, 48 h. The counts were expressed as mean value CFU/mL.

*Assessment of pollutant load development – total coliforms – VRBL (Oxoid CM 101), 30°C, 48 h, faecal coliforms – VRBL (Oxoid CM 101), 44°C, 48 h, in double layer, Streptococcus faecalis – Slantez and Bartley Medium (Oxoid CM 377), 37°C, 48 h, The counts were expressed as mean value CFU/mL; BOD<sub>5</sub> by Oxitop IS 6 System and total insoluble solids content of the final solutions (4, 8 and 12 cycles) performed with a filtration System Millipore with Millipore Nylon Net type NY11 (results expressed in g/100 mL). *Statistical analysis – Results were submitted to principal component analysis (PCA) and factorial discriminate analysis (FDA) with the Statistica™ v. 5.0.**

**Osmodehydrated fruits.** *Physical-chemical parameters:* soluble solids content (°Brix) obtained by a hand-held refractometer ATAGO PR 201, pH measured with a potentiometer CRISON Micro pH 2002, using a penetrating electrode, colour by

reflectance evaluated by a Minolta Croma Meter CR 200b, texture determined with a texturometer TA-Hdi with 10 blad Kramer Shear Cell (results expressed in N/g), water activity ( $a_w$ ) by electronic hygrometer Rotronic Hygroskop DT, organic acids by HPLC-DAD with a chromatograph Beckman at 214 nm, eluent  $H_2SO_4$  5 mM, Aminex HPX-87H column from Bio-Rad (results expressed in g/100 g d.m.), carbohydrates and ethanol by HPLC chromatograph Waters, RI detector, Sugar-pak column (Waters), eluent EDTA-Ca 50 ppm (results expressed in g/100 g d.m.). *Microbiological parameters:* samples (10 g) were blended with 90 mL of Maximum Recovery Diluent (Oxoid CM 733) and homogenized in a stomacher pouch, Type 400 LabBlender 100. Serial dilutions were made in tubes before planting. The media and incubation conditions were: total aerobic mesophilic flora – PCA (Oxoid CM 325), 30°C 48 h, xerophilic moulds-DG 18 Agar (Oxoid CM 729), 25°C, 5 days, osmophilic yeasts – Tilbury medium [Tilbury 1976] 37°C, 48 h and lactic flora – MRS Agar (Oxoid CM 361), 37°C, 48 h. The counts were expressed as mean value CFU/g. *Sensorial evaluation:* The general overall quality samples were assessed by a panel of 10 tasters using the quantitative descriptive analysis on a scale from 1 to 5 (1 = bad and 5 = excellent).

## RESULTS AND DISCUSSION

### Osmotic dehydration cycles effect on quality of drying solution

Figure 3 presents results of Principal Component Analysis (PCA) and Factorial Discriminate Analysis (FDA), obtained during mandarins OD process. The distribution of experimental points distinguishes a different behaviour between the original (1 to 12) and final (1F to 12F) solutions, which can list them as follows: an initial group of solutions consisting of three subgroups and another group of final solutions, also composed of three subgroups. To mention the approximation of solutions 1 and 1F, that is justified by the fact that the first processing didn't practically influence the characteristics of the solution.

Regarding the distribution of other experimental points, which characterize the other cycles, it appears that the initial solutions, explained by varying viscosity and °Brix, are opposed to the final ones, justified by the variables conductivity, turbidity and colour, with a direct link between the initial and final solutions of cycles 2 and 3, and other solutions, between the beginning and ending of remaining cycles. This distribution is due to the fact that in cycles 2 and 3, the final solution presented a relatively minor modification to the cycles 4F to 12F [Sapata et al. 2003].

The sucrose solution at 60°Brix, reused in the mandarin osmotic processing, did not affect its desiccant power. Regarding °Brix, for all cycles, the final solution values were lower than the initial ones, because the diluting effect during each cycle. pH decreased dramatically at the end of the first cycle and remained constant until the end of the 12 cycles. Conductivity and turbidity increased gradually due to the increase of salt content in fruits and presence of substances in suspension.

In colour parameters, there was a slight increase in yellow, more pronounced in the final solutions, due to fruits pigments leaching. Viscosity and soluble solids content (°Brix) ranged proportionally within each cycle. From microbiological point of view, the solution remained stable. High concentration of sugar and low pH values, hostile for

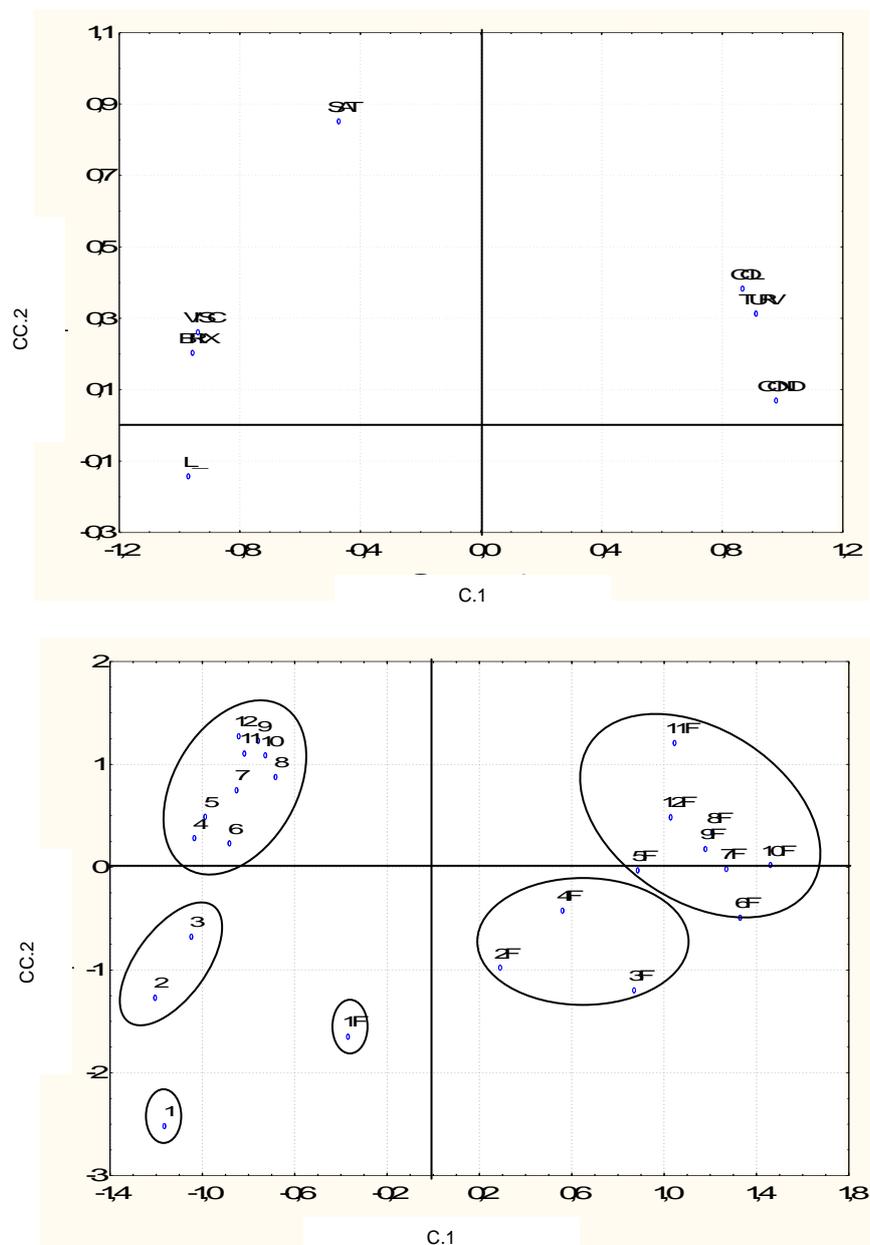


Fig. 3. Principal component analysis and factorial discriminate analysis: 1-12 – initial solutions, 1F-12F – final solutions, BRIX – °Brix, COL – °Hue, COND – electrical conductivity, L – lightness, SAT – saturation, TURV – turbidity, VISC – viscosity

Table 1. Values of solutions as final effluent

Cycles	BOD <sub>5</sub> mg/L O <sub>2</sub>	Total coliforms CFU/mL	Faecal coliforms CFU/mL	<i>S. faecalis</i> CFU/mL
4	40	neg.	neg.	neg.
8	380	neg.	neg.	neg.
12	520	neg.	neg.	neg.

microbial development, were quite favourable, as well as the biological filter of glass membrane used in the filtration system.

For microbiological analysis, it is known that osmotolerant or osmophilic microorganisms are particularly tolerant to high sucrose concentrations and cause few problems in industrial activities of this nature. From the ninth cycle, a slight increase was observed in total aerobic mesophilic flora and xerophilic moulds on the final solutions ( $10^2$  CFU/mL), in each OD cycle. Osmophilic yeasts and lactic flora count was always negligible.

According to these results it seem that the number of microorganisms increased with the number of OD cycles, but never reached a critical value that undermine the stability of microbial processes. This may be due, not only to the filtration system used, but also to the °Brix and pH of the solutions that are inhibitors of microbial development. Furthermore, the temperature used in osmotic processing was not favourable either.

For pollutant load monitoring, the solution was evaluated in final of the 4th, 8th and 12th OD cycle, to determine from which cycle if it would be necessary to dilute the solution, to discharge as effluent. Although from the microbiological point of view there were no changes (limit values: total coliforms –  $2 \cdot 10^2$  CFU/mL, faecal coliforms –  $1.2 \cdot 10^2$  CFU/mL, *S. faecalis* –  $2 \cdot 10$  CFU/ml), BOD<sub>5</sub> values remained above the established limit issue value (ELV) of Portuguese Law, so it would be necessary to dilute the solution before its release as effluent, from the 4th cycle (Table 1).

### Osmotic dehydration cycles effect on quality of mandarins

An increase in solids soluble content, from 9.4 to 20.84°Brix (average) was observed during 12 mandarin's osmotic cycles. The pH in fresh fruits (RM – raw material) and after processing was identical (3.39 in the final product and 3.41 in raw material), meaning the fruit pH was not affected by the OD process, due to the balance established between the fruit and solution pH in which they were processed, that is an advantage to inhibit microbial growth. Processing caused a slight increase in lightness ( $L^*$ ) in osmodehydrated mandarins (44.94) compared to fresh segments (41.65). Saturation and Hue values were always higher in processed fruits than in raw material: 31.06 and 22.05 for saturation and 85.70 and 82.17 for Hue. The OD cycles caused an increase on texture segments (6.67 N/g relatively to fresh segments 4.42 N/g). One can conclude that, during OD process, the difference generated in osmotic pressure led to a slight increase in the mechanical resistance of the fruits (Fig. 4).

Regarding  $a_w$  determination, there was a decrease in osmodehydrated fruits in comparison to raw material, from an average of 0.998 to 0.975 (data not shown). This decrease, due to the effect of the technological process, became beneficial because,

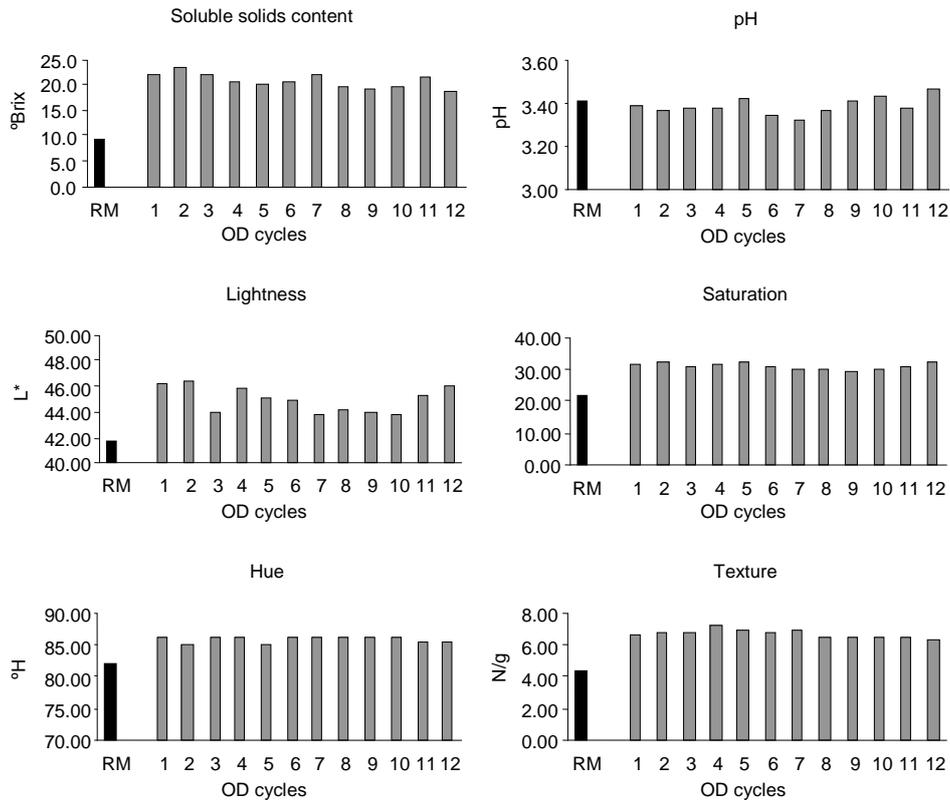


Fig. 4. Soluble solids content, pH, lightness, saturation, hue and texture mandarin's changes

together with low pH values, allows a reduction of bacterial development. However, this value is not low enough to make the final product stable, so a stabilization treatment was necessary in "combined" process (pasteurization).

Figures 5 and 6 illustrate the evolution of sucrose, carbohydrates and ethanol along the 12 OD cycles. There was an increase of sucrose and total carbohydrate in almost all cycles in final product. On the other hand, a decrease was observed in the glucose and fructose levels, by diffusion of these compounds to the solution. The ethanol content, as a result of sugars fermentation, remained constant along the cycles. All osmotic cycles caused an identical reduction in citric, L-malic and succinic acids content in mandarins segments (Fig. 7).

The mandarins' microbiological characteristics evolution, showed values below the limits adopted in the bacteriological standards. Table 2 presents the average values of total aerobic mesophilic flora and xerophilic moulds in fresh and osmodehydrated mandarins segments for each OD cycle. The osmophilic yeasts and total lactic flora count was negative.

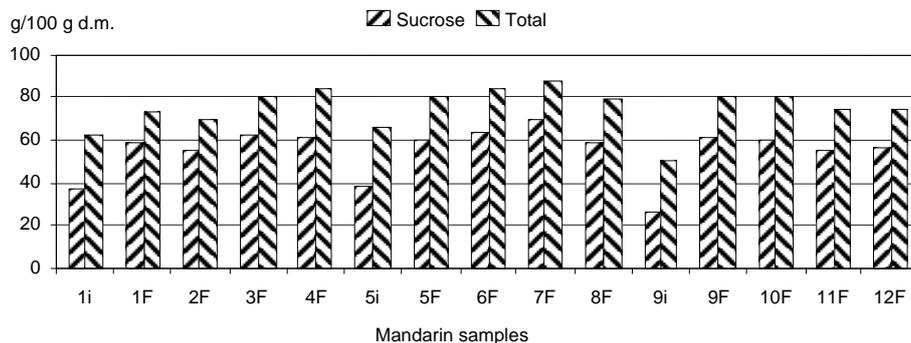


Fig. 5. Mandarins changes in the levels of sucrose and total carbohydrate: i – initial, F – final

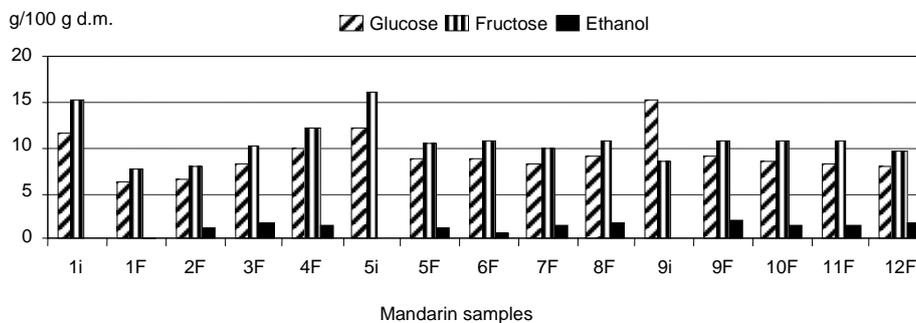


Fig. 6. Mandarins changes in the levels of glucose, fructose and ethanol: i – initial, F – final

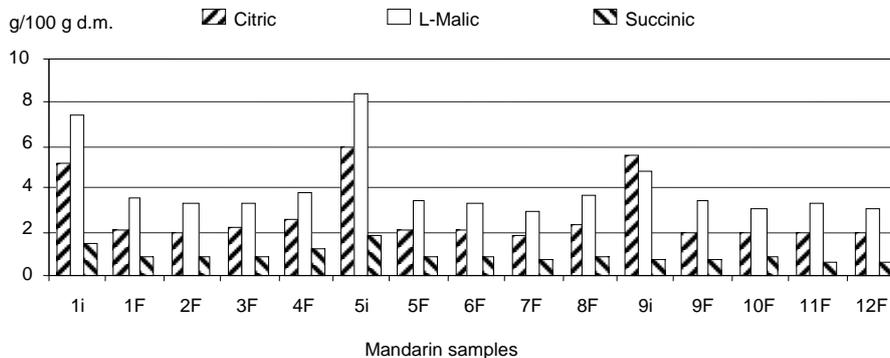


Fig. 7. Mandarins changes in the levels of organic acids: i – initial, F – final

Table 2. Medium values of microbiological parameters of osmodehydrated mandarins

Cycles	Total aerobic mesophilic flora CFU/g	Xerophilic moulds CFU/g
Raw material	1·10	5·10
1	4·10	3·10
2	3.7·10 <sup>2</sup>	2·10 <sup>2</sup>
3	9·10	8·10
4	9·10	2.4·10
5	1·10	9·10
6	2·10	6·10
7	3·10	5·10
8	2·10	neg.
9	3·10	3·10
10	4.7·10 <sup>2</sup>	1.8·10 <sup>2</sup>
11	5·10	3·10
12	4·10	2·10

By an osmopasteurized mandarin's sensorial evaluation, regardless of all OD cycles, an improvement was observed, in the appearance, texture, flavour and stability at room temperature, always giving a similar classification (3.89,  $\delta = 0.189$ ). It is therefore, a product of high added value, ready to be consumed or incorporated in other formulations.

## CONCLUSIONS

Taking into account working conditions, particularly the known control variables of OD and the mechanisms involved in the current flows, the biological material and their interactions, as well as the filtration system used, and data analysis, it was possible to reuse sucrose solution in the osmotic process, during several cycles without changes on final product quality. The osmopasteurized mandarin segments revealed to be an interesting product being displayed by sensorial characteristics, so, given its innovative nature; it could become a new convenience product.

It offers good prospects for the future of osmotic dehydration, due to low energy costs associated with moderate processing and the improvement of products quality to various complementary alternatives.

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## OSMOTYCZNE ODWADNIANIE MANDARYNEK: WPŁYW WIELOKROTNEGO UŻYWANIA ROZTWORU OSMOTYCZNEGO NA JAKOŚĆ I TRWAŁOŚĆ PRODUKTU

**Wprowadzenie.** Osmotyczne odwodnienie (OD) jest techniką, która pozwala na częściową dehydratyzację owoców i warzyw, przez zanurzenie w hipertonicznym roztworze cukru, soli lub innym. Może być z powodzeniem zastosowane w odniesieniu do niektórych produktów, które nie będą wprowadzone do obrotu w stanie świeżym. Aby jednak osiągnąć wysoką jakość i trwałość produktu końcowego, niezbędne jest zastosowanie dodatkowego procesu. Celem pracy było zbadanie wpływu procesu osmotycznego odwadniania mandarynek w połączeniu z procesem pasteryzacji na jakość końcową produktu oraz ocena możliwości wielokrotnego wykorzystania roztworu osmotycznego.

**Material i metody.** Materiałem badań były mandarynki (*Citrus reticulata* Blanco) 'Clementina Nova', hodowane w Algarve (Portugalia), ręcznie obierane rozdzielone i pozbawione chemicznie skórki. Owoce przez 16 h były poddane kąpielom w roztworze sacharozowy o stężeniu 60°Brix i temperaturze 45°C oraz poddawane drganiom o oscylacji równej 40/min. Stosunek owoców do roztworu osmotycznego wynosił 1:2 (m/m). Po każdym cyklu OD roztwór osmotyczny był filtrowany oraz uzupełniany przez dodanie sacharozы tak, aby jego stężenie było stałe i wynosiło 60°Brix. Odwodnione mandarynki były stabilizowane przez pasteryzację. Po zakończeniu procesu oceniano różne właściwości fizyczne, chemiczne i biologiczne przetworzonych owoców.

**Wyniki.** Czynnikiowa analiza skupień pozwoliła obserwować, które czynniki wpływają na jakość produktu końcowego. Połączenie procesu odwadniania osmotycznego i pasteryzacji pozwoliło na uzyskanie dobrej stabilności produktu. Wielokrotne stosowanie tego samego roztworu osmotycznego do przeprowadzania dehidratyzacji nie miało wpływu na jakość produktu.

**Wnioski.** Wyniki prowadzą do wniosku, że zastosowanie OD jako wstępnego procesu technologicznego obróbki mandarynek ma pozytywny wpływ na jakość i stabilność owoców poddanych później pasteryzacji. Wielokrotne stosowanie tego samego roztworu osmotycznego nie ma wpływu na jakość produktu końcowego. Podczas procesu utylizacji wielokrotnie używanego roztworu osmotycznego należy uwzględnić jego duże zanieczyszczenie mikrobiologiczne.

**Słowa kluczowe:** odwadnianie osmotyczne, parametry jakościowe, trwałość

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