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RECONDITIONING BY FILTRATION WITH EARTH DIATOMACEOUS AND REUSING THE SUCROSE SYRUP FOR OSMOTIC DEHYDRATION OF PEACHES

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ABSTRACT A method for reconditioning the sucrose syrup used in the osmotic pre-drying (OPD) of peaches was evaluated consisting of sieving (1mm mesh), filtration with earth diatomaceous, vacuum concentration and syrup replacement. The osmotic dehydration trial was carried out in 15 cycles (50ºC, 65ºBrix, syrup/fruit ratio of 4:1 and process time of 4h), using reconditioned syrup in each cycle. Complementary drying was carried out in a forced air dryer at 65ºC for approximately 5h. Physical (color and turbidity), chemical (soluble solids content, pH, titratable acidity, reducing sugar content, electrical conductivity) and microbiological (yeasts and molds) analyses were carried out on the syrup. The osmotic pre-drying parameters water loss and solids incorporation were also determined. A sensory evaluation was carried out with dried peach using the Difference from Control Method, evaluating the following attributes: flavor, texture, color and appearance. The reconditioning process showed no influence on the osmotic pre-drying parameters, and the microbiological loads of the syrup remained low. The following alterations occurred in the syrup during the OPD cycles with reconditioning and reuse: increase in titratable acidity, lowering of the pH, increase in electrical conductivity and an increase in the reducing sugar content. The turbidity remained constant. Reuse did not influence the flavor or texture and favored maintenance of the yellow color of the dried peach.

Keywords: *Prunus persica* (L.) Batsch, drying, osmotic solution.

INTRODUCTION The osmotic pre-drying (OPD) technique consists of immersing the fruit in a hypertonic solution of one or more solutes such that the partial removal of water occurs as a function of the established difference in chemical potential (Chiralt & Talens, 2005). During this process, other fluxes apart from the removal of water are established, such as a gain of soluble solids and loss of natural soluble solids from the food.

Management of the syrup represents the greatest technological problem in OPD (Warczok, Gierszewska, Kujawski & Guell, 2007), since it is diluted during the process, its volume increasing with this gain in water. Discarding the diluted syrup can make the process economically non-viable and also cause severe environmental impact since it is an effluent with a high organic load (Marouzé, Giroux, Collignan & Rivier, 2001).

Some studies have reported different ways of reusing the OPD osmotic solution, with or without reconditioning (Bolin et al., 1983; Valdez-Fragoso et al., 1998; Valdez-Fragoso et al., 2002). Germer et al. (2009) evaluated a method for reconditioning the sucrose syrup used in the osmotic pre-drying (OPD) of peaches by sieving and vacuum concentration, without the filtration step. In the study, a significant increase in turbidity of the syrup was observed in 15 OPD cycles. According to Germer et al. (2009b), the use of this syrup for the production of peach preserves was successful. However, the clarification of the syrup may be needed in other applications, such that in formulation of some drinks.

The aim of the present study therefore was to evaluate the reconditioning and reuse of sucrose syrup in the osmotic dehydration of peach including the filtration step with earth diatomaceous in order to obtain clear syrup.

MATERIAL & METHODS Peaches (Aurora-1 variety) were donated by the Holambra Agro-industrial Cooperative, Paranapanema, São Paulo State, Brazil. The fruits were harvested in the stage of maturity ideal for commercialization, characterized by the point where the ground color of the skin changes from green to yellow.

Fifteen OPD cycles were carried out, starting the first cycle with fresh syrup and employing the reconditioned syrup in the subsequent cycles. The fruits were selected, washed, peeled, de-stoned and cut manually into slices. They were chemically blanched by immersion in a citric acid (4g/100g) plus ascorbic acid (1g/100g) solution for 40s to avoid enzymatic browning. An 8-liter bath with a 10 liter/min agitation (Immersion Circulation, Model 1266-02, USA) was used. The first cycle started with fresh sucrose syrup (6 kg) with a concentration of 65ºBrix. The following parameters were used in the OPD: syrup mass:fruit ratio of 4:1, process temperature of 50ºC and process time of 4 hours. At the end of the operation, the pieces of fruit were removed from the bath, drained, rinsed in water and carefully dried with absorbent paper. The pieces of fruit and the syrup were weighed at the beginning and end of the OPD operation. The syrups were then submitted to the reconditioning operation for use in the next cycle. The following operations were carried out at the reconditioning step: sieving through a steel sieve (1 mm mesh); filtration through diatomaceous earth; concentration in a vacuum vat; addition of new syrup to return to the original mass. The filtration operation was carried out using a Buchner funnel (diameter 12.3 cm) and kitassato (4 liters) attached to a vacuum pump (Tito Manzini & Figli, model AM1, Italy). Qualitative filter paper pre-coated with the diatomaceous earth Celite (Diactive 11F, Chile) enriched with cellulose fiber, was placed in the funnel. The pre-coating was formed preliminarily using a mixture of earth and water so as to obtain a 1 kg/m^2 layer, according to the manufacturer's instructions. This procedure aimed to simulate the pre-coating filter operation used in the clarification of syrups and beverages. The concentration of the syrup was carried out in an evaporator (vat with vacuum) with a steam jacket and constant agitation (Mecamau, model C055, Brazil), and the vacuum applied was approximately 0.5 to 0.7 kgf/cm^2. After concentration, the initial syrup mass was recovered by adding new syrup in amount so as to maintain the mass ratio of 4:1. The reconditioned syrup was maintained at room temperature until the next cycle. The pre-dried fruit pieces were dried in a conventional tray dryer with air circulation (velocity of 1.5 m/s) at 65ºC until they reached a final moisture content between 16 and 19%. The parameters of water loss (WL) and solids incorporation (SI) of the fruits during the osmotic drying process were calculated for all the cycles, using the following equations:

$$WL = \frac{(UiMi - UtMt)}{Mi} \times 100 \qquad \text{(g of water/100g of initial mass)} \qquad (1)$$

$$SI = \frac{STtMt - STiMi}{Mi} \times 100 \qquad \text{(g of solute/100g of initial mass)} \qquad (2)$$

where: Ui = Initial moisture content; M_i = initial mass; Sti = initial solids content; Ut = Moisture content at time t=4h; M_t = mass at time t=4h; STt = total solid content at time t=4h;

The soluble solids contents of the initial and final syrups were determined for all the cycles, as also the moisture and total solids contents of the raw materials and pre-dried fruits. At every third cycle the following analyses were also carried out on the syrups: pH, total acidity, nephalometric turbidity, electrical conductivity, instrumental color. The microbiological analyses were carried out on the initial syrups from the 1st, 5th, 10th and 15th cycles, and samples of the dried fruits resulting from the same cycles were reserved for the sensory analysis.

Analytical methods The soluble solids were determined using an optical bench refractometer (AO Abbe Refractometer model 10450, USA) and total acidity (TA) by an acidimetric method.

The moisture content was determined in a vacuum oven at 70ºC for 24 hours adapted by Carvalho, Mantovani, Carvalho & Moraes (1990). Turbidity was determined using a turbidimeter (Hach, model 2100P, USA) and electrical conductivity in a conductivimeter (Digimed, model DM 31, Brazil). The above cited analyses were all carried out in triplicate. The initial masses of the fruit pieces, as also those of the pre-dried fruit, were weighed on a mechanical balance (Mettler, P10N, Germany). A chromameter colorimeter (Minolta, CR300, Japan) was used for the color analyses using the Cielab system, making a direct reading from the sample with the d/0 configuration and D65 illuminant, using a special support for liquids (Minolta, CRA-70, Japan). The analyses were carried out with three repetitions with three shots each. The yeast and mold counts were based on the methodology described by Downes & Ito (2001). The sensory analyses were carried out evaluating samples of the final products (dried fruits) from the following cycles: 1, 5, 10 and 15. The Difference from Control Method was used (Faria & Yotsuyanagi, 2002) with a panel of 20 trained judges, and products from the first cycle were used as the standard (S). The attributes evaluated were appearance, color, texture and flavor of the product. The scale for the attribute appearance was divided into 5 levels, varying from “better than S” to “worse than S”. The scale for the attribute color (characteristic peach yellow) was divided into 7 levels, varying from “much less intense than S” to “much more intense than S”. The scale for the attribute texture also had 7 levels, varying from “much softer than S” to “much harder than S”. Finally the scale for the attribute of flavor was divided into 6 levels, varying from “better flavor than that of S” to “worse flavor than that of S with strong off-flavor”. The results were analyzed by the Variance Analysis (F test) and Dunnett Test (difference between means) employing the SAS program (Statistical Analysis System, SAS Institute, Inc, USA).

RESULTS Table 1 and Table 2 show the variation in drying parameters and in results of physical and chemical analyses for the initial syrups in some cycles of the trial. Table 3 shows the sensory results obtained for the dried peaches in some cycles.

Table 1. Water loss (WL) and solid incorporation (SI) parameters, soluble solid contents and color parameters of the initial syrups in the OPD cycles with reconditioning and reuse.

cycle	WL g/100g	SI g/100g	soluble solid (°Brix)	L*	a*	b*	Hue angle
1	54.5	6.7	65.2±0.2	37.51±0.23	1.74±0.08	-2.19±0.08	-51.53
4	54.9	7.1	65.3±0.2	39.92±0.32	3.21±0.18	-0.59±0.13	-10,41
7	53.6	5.8	65.5±0.1	39.21±0.32	2.61±0.35	-0.10±0.45	-2.28
10	49.5	1.7	65.0±0.0	39.38±0.44	2.92±0.10	0.17±0.40	3.39
13	51,6	3.8	64.8±0.0	-	1.25±0.10	-	
15	51.2	3.4	64.4±0.1	39.58±0.17	2.65±0.17	0.19±0.14	3.61

Table 2. Titratable acidity, pH, reducing sugar content, electrical conductivity and nephalometric turbidity of the initial syrups in the OPD cycles with reconditioning and reuse.

Cycle	Titratable Acidity 10^{-3}g/100gca	pH	Reducing sugar g/100g dm	Electrical conductivity microS/cm	Turbidity ntu
1	3±0	7.47±0,04	0.0	40.73±0.42	1.31±0.02
4	125±3	4.08±0.02	4.9±0.1	61.07±0.21	1.31±0.02
7	174±1	4.04±0.01	5.0±0.7	80.73±0.67	1.19±0.03
10	173±9	4.19±0.02	5.7±0.1	79.73±0.55	1,19±0.02
13	180±1	4.14±0.01	5.3±0.6		1,38±0.02
15	174±7	4.09±0.02	6.4±0.3	97.43±0.51	1.67±0.01

Table 3. Sensory results for the dried peaches obtained in different OPD cycles with reconditioning and reuse.

Attribute/cycle	1	5	10	15
appearance	3.0^{a}	2.3^{b}	2.3^{b}	1.0^{c}
color	4.0^{a}	5.2^{b}	4.1^{a}	5.8^{c}
texture	3.8^{a}	4.8^{b}	$4.0^{a,b}$	3.2^{a}

flavor	2.2^a	2.6^a	2.4^a	2.4^a

Means followed by the same letter in the same line do not differ significantly (p<0.05)

The syrup reconditioning operations were carried out such that the initial conditions remained very close, with mean of 65.1±0.5ºBrix. The percentage of new syrup mass added after concentration in order to recover the initial mass was 19.4±2.1%. No tendencies for variation were observed in the parameters WL and SI throughout the cycles. There was an expressive increase in titratable acidity (TA) of the syrup in the initial cycles, followed by stabilization around the sixth cycle. The increase in TA was due to leaching of natural acids from the fruit and also of the acids acquired during chemical blanching. Stabilization probably occurred due to the formation of an acid equilibrium between the fruit and syrup, and to the constant replacement of part of the syrup mass. There was a drop in the pH value of the syrup at the first cycle from 7.47±0,04 to 4.27±0.04 at the end of the same cycle. The value became stable in the following cycles. The raw fruits showed mean pH values of 4.31±0.09 and the pre-dried fruits 4.26±0.19. Thus equilibrium was already attained between the syrup and the fruits with respect to pH. According to García-Martinez et al. (2002), this lowering of the syrup pH could be of benefit with respect to its conservation.

Unlike the previous study, the turbidity remained virtually unchanged during the 15 OPD cycles. An increase of 150% in electrical conductivity of the syrups during the fifteen cycles is observed throughout the reuse cycles. This increase is due to the accumulation of soluble compounds removed from the fruit during OPD, mainly mineral salts and organic acids. There was also an increase in the reducing sugar contents (RS) during the first 4 cycles. According to Dalla Rosa & Giroux (2001) two mechanisms are responsible for the increase in RS content during reuse: the flow of RS from the fruit to the syrup and the acid hydrolysis of non-reducing sugars.

There was a slight increase in L* for the syrup, and this behavior was possibly due to antioxidant action caused by the ascorbic acid used in blanching. There was an increase in the parameter b*, varying from the blue (b*<0) to the yellow (b*>0) region, and the reason was possibly due to the extraction of pigments from the peaches. There was also increases in the value for a*, varying from the neutral (a*≈0) to the red (a*>0) region. In the same way hue angle showed an important variation, from the blue (h*<0) to the red (h*>0) region. The reason for the last two variations were possibly due to a non-enzymatic browning caused by the presence of reducing sugars in the syrup.

The observed variations of physical and chemical properties of the syrup throughout the test followed similar trends with those cited by Germer et al. (2009) in the previous work, except for the turbidity. However, due to higher replacement of syrup's mass in this test, the quantitative properties were slightly lower than those observed in the previous study for the same cycle.

Despite the greater handling of the syrup with the filtration step, the microbial load did not increase throughout the 15 cycles of OPD with reconditioning and reuse, similarly to that obtained in the previous study. The counts for yeast and mold in initial syrup in all sampled cycles were less than 10^2 cfu/ml. The heat treatment carried out during the concentration of the syrup before each cycle apparently had fundamental importance in controlling the microbial load. In this stage of the reconditioning process the syrup was held for approximately 5 minutes at temperatures varying from 68 to 75ºC. According to Dalla Rosa & Giroux (2001), a heat treatment of 72 to 75ºC for 30s would be sufficient to pasteurize the syrup coming from an OPD operation.

It can be seen from Table 3 that the means for the attribute of color increased significantly throughout the cycles (with the exception of the products obtained from cycle 10). The judges attributed an intensity of yellow color equal to the standard for the product from cycle 1 (the very same sample), and a moderately greater intensity than the standard to the product from the 15^{th} cycle. This means that a slight intensification of the yellow color of the products was observed with the reuse cycles, in agreement with the observations from the instrumental determinations of the color of the syrup. With respect to appearance, the products presented significant differences between themselves ($p<0.05$) for the different cycles and also with respect to the standard. It should be remembered that the scale was inverted for the attribute of appearance. Thus for the judges, the appearance of the products improved with the increase in number of cycles, varying from "appearance equal to S" in cycle 1 to "appearance better than S" in the last cycle. It is clear that, in the analysis of appearance, the color of the product showed considerable influence, since both showed the same tendency, that is, to improve with increase in the number of cycles. No adverse factor appears to have had any effect on the appearance of the dried products. With respect to the attributes of texture and flavor, the judges did not note any significant differences between the products of the various cycles (with the exception of the product from cycle 5). The sensory results are similar to those observed by Germer et al. (2009) in the process without filtration.

CONCLUSION The reconditioning and reuse of the syrup had no influence on the water loss and solids gain during OPD up to 15 cycles. The following alterations occurred in the syrup during the OPD cycles with reconditioning and reuse: increase in titratable acidity, in electrical conductivity, and in the reducing sugar content; lowering of the pH. The turbidity remained almost the same during the 15 OPD cycles. The microbial load remained low in the syrups up to the 15^{th} OPD cycle, even with the addition of the filtration operation. This was probably due to the low pH value of the syrup/fruit system and the heat treatment used in the concentration step. The reconditioning and reuse of the syrup apparently contributed to maintenance of the yellow color of the dried peach, positively influencing the sensory evaluation of the final products. Despite all variations observed in the syrup, up to at least 15 cycles, the employment of reconditioned and reused syrup did not affect the sensory evaluation of the flavor and texture of final products. Reconditioned and reused syrup obtained by the evaluated method can still be used as an ingredient in the development of products whose clarity is required such as juices and softdrinks. However the inclusion of a filtration step with a pre-coating filter will mean higher investments and the economic feasibility of the process should be examined.

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