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Article

Byproducts of orange extraction: influence of different treatments in fiber composition and physical and chemical parameters

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In this work we evaluated the variability in fiber content and physical and chemical parameters of byproducts from orange juice extraction. Five different treatments and two drying methods were evaluated. The results indicate that drying by lyophilization was better than that drying in an oven. The pH ranged from approximately 3.47 to 3.96. The variation in moisture values was 9.22% ± 0.02 to 18.48 ± 0.52%. The total dietary fiber content in the resulting flours ranged from 42.44% to 62.74%. The soluble and insoluble dietary fiber contents differed among the samples, ranging from 5.04% to 19.95% for the first fiber type, and 23.96% to 57.70% for the second. In conclusion, three treatments, associated with freeze-drying, showed promising results in the development of fiber-rich product. However, some modifications are needed, as well as further analysis, to guarantee the benefits of these products for human health. This study contributes to the possible application of industrial byproducts.

Uniterms: Orange/byproducts/evaluation. Orange juice/extraction. Orange/byproducts/fiber content. Fibers. Orange/byproducts/physical-chemical parameters.

Neste trabalho avaliou-se a variabilidade no conteúdo de fibras e nos parâmetros físico-químicos de subprodutos da extração do suco de laranja. Investigaram-se cinco diferentes pré-tratamentos e dois tipos de secagens dos produtos. Os resultados indicam que a secagem por liofilização é melhor que aquela feita em estufa. O pH dos produtos variou de, aproximadamente, 3,47 a 3,96. A variação entre os valores de perda por dessecação foi de 9,22% ± 0,02 a 18,48 ± 0,52%. O conteúdo de fibras totais encontrado nas farinhas obtidas variou de 42,44% a 62,74%. O conteúdo de fibras dietéticas solúveis e insolúveis diferiu entre as amostras de 5,04% a 19,95%, para a primeira, e 23,96% a 57,70%, para a segunda. Como conclusões, três tratamentos, associados à secagem por liofilização demonstraram resultados promissores no desenvolvimento de produto rico em fibras, entretanto, algumas modificações e novas análises devem ser realizadas a fim de garantir os benefícios desses produtos para a saúde. Esse estudo contribui para uma possível utilização de subprodutos da indústria alimentícia.

Unitermos: Laranja/subprodutos. Suco de laranja/extração. Laranja/subprodutos/conteúdo de fibra. Fibras. Laranja/subprodutos/parâmetros físico-químicos

INTRODUCTION

Among all the types of fruit trees, orange is one of the most well-known and widely studied worldwide. Since the 1980s, Brazil has been the world's largest producer of oranges and orange juice, and the country has more than a million acres of citrus plants. Although the juice is the main product derived from orange, various byproducts are produced during the manufacturing process. These include essential oils, terpenes, and citrus pulp flour (Baptistella *et al.,* 2009; CitrusBR, 2012).

Byproducts are sold on the domestic and foreign markets, where they are used in different applications, including the manufacture of chemicals and solvents, in flavorings and fragrances, as substances for use in paints, in cosmetics, and as an animal feed supplement (Areas, 1994). Pelleted citrus pulp flour or orange peel bran is an example of a processing byproducts obtained by the

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treatment of solid and liquid waste resulting from the process of orange juice extraction (OJE) (Cutrale, 2012; Lousada *et al.,* 2005). Despite all these possibilities, industrial waste from orange juice remains mostly unused.

According to Nogueira *et al*. (2000), orange agroindustry is among the areas that generate large amounts of waste with possibilities for use. This recovery is directly linked to sustainable development and the marketability of these byproducts. Fernández-Lopez *et al.* (2004) state that the byproducts from citrus processing represent a serious problem for industry, given their limited applications and low added value. Orange peel is an example of a primary byproduct from extraction that if not reused, becomes waste and a possible source of environmental pollution. In this work, the authors present alternatives for converting byproducts into promising sources of ingredients for use in the food industry, taking advantage of their interesting technological value and nutritional properties.

Phytochemicals, such as flavonoids, carotenoids and pectin, are important for health, and are abundant in these food industry byproducts. The highest concentration of flavonoids (the largest secondary metabolite group in Citrus fruits) is present in the peels, while pectins (a class of complex polysaccharides that provide dietary fiber) are found in larger quantities in the albedo, or pith (Bocco *et al.,* 1998; Liu, Shi, Langrish*,* 2006). Thus, byproducts from OJE have potential use as sources of dietary fiber (DF). This material is rich in soluble and insoluble fiber, and is abundant and low in cost (Grigelmo-Miguel, Martín-Belloso, 1998; Santana, 2005; Topuz *et al.,* 2005).

Regular consumption of dietary fiber regular has been one of the most consistent recommendations of nutritionists and official agencies for the prevention of gastrointestinal and cardiovascular diseases, and also for the prevention and/or treatment of diabetes, hypercholesterolemia and obesity. These recommendations are based on the finding that dietary fiber has physiological effects that promote significant changes in human gastrointestinal functions, such as a reduction in nutrient uptake, increased fecal mass, reduction in blood plasma cholesterol levels, and lowered glycemic response (Lajolo *et al.,* 2001; Botelho, Conceição, Carvalho*,* 2002).

Functional constipation can be effectively treated with increased intake of fiber or a diet supplemented with vegetable fibers, fluids, and plenty of exercise (Brunton, 1995), all of which promote rehabilitation of bowel function. According to an expert committee of the US Food and Drug Administration (FDA), the amount of dietary fiber consumed should be at least 20 g/day. Of this, approximately two thirds should consist of insoluble fiber and one-third of soluble fiber (Rakel, 2012).

There is an established relationship between dietary fiber and certain chronic intestinal disorders, such as constipation, hemorrhoids, diverticulitis, colon cancer and rectal cancer (Lajolo *et al.,* 2001). According to these findings, a balanced diet that is rich in favorable nutrients is essential for good performance of the gastrointestinal tract. Information on the of food industry byproducts is limited, but the search for alternatives is of great interest, as it combines both economic and environmental aspects with the production of high quality nutritional food for human consumption (Giuntini, Lajolo, Menezes*,* 2003).

Grigelmo-Miguel and Martín-Belloso (1999) demonstrated that OJE waste is an excellent potential source of dietary fiber, as this material is rich in pectin and is available in large quantities. Citrus fiber is of high quality when compared to other forms of dietary fiber, due to the presence of associated bioactive compounds, such as flavonoids and other polyphenols, and carotenoids (Fernández-Ginés *et al.,* 2003; Wolfe, Liu, 2003). Some studies have demonstrated the importance of the recovery of orange processing byproducts to obtain beneficial substances for the body, especially the gastrointestinal tract. Rincón *et al.* (2005) studied the chemical composition and bioactive compounds found in an orange peel flour (*Citrus sinensis*) from fruits cultivated in Venezuela. The results demonstrated high dietary fiber levels, as well as the presence of polyphenolic compounds with significant antioxidant activity.

In a study by Sáenz *et al.* (2007), the authors developed an energy snack bar, which was obtained by combining a flour from residue of OJE plus other compounds, giving a dietary fiber rich product with high energy intake. In the analysis of the flour obtained, the authors found significant levels of dietary fiber (both soluble and insoluble), and concluded that the product is a good source of fiber, with good acceptability.

This work investigates the physical and chemical parameters and fiber content variability in different treatment products, to determine the feasibility of recovery of byproducts from OJE carried out the processing plant by Petry – Comércio de Alimentos LTDA industries, Ivoti (RS), in the development of a rich dietary fiber product, which can help improve the symptoms of functional constipation.

MATERIAL AND METHODS

Byproducts

Byproducts of orange juice extraction at the processing plant of Petry – Comércio de Alimentos LTDA

industries, Ivoti (RS, Brazil) were used in this work. This material was received in March, 2009 and stored in six bottles.

Sample processing

Treatments

Five different byproduct treatments were performed to investigate their effects on the dietary fiber content and physical and chemical parameters.

1. Pressing: A manual apparatus was used to remove the "liquor" from orange peel in order to extract the essential oil. This "liquor" was centrifuged, after removing the supernatant diluted in hexane. The hexane fraction was separated, and at the end of the process, the pulp was stored at -20 °C for subsequent drying;
2. Essential oil extraction by hydrodistillation (Clevenger apparatus): The process was conducted in 120 minutes (Brazilian Pharmacopoeia, 1988a). The oil obtained was stored in amber glass vial and refrigerated for later analysis. It was not used in this study. The extracted orange peel was stored at -20 °C;
3. Peeling by hand: The orange peel was removed manually from the byproduct, as it is the component primarily responsible for the bitter taste. After this process, the product was stored at -20 °C for subsequent analysis;
4. Washing: in this treatment, the orange byproducts were washed with distilled water and sieved, to remove excess water. Finally, the samples were frozen under the same conditions described above;
5. No treatment applied: This sample aliquot was not submitted to any treatment, merely separated and stored at -20 °C.

Drying methods

Three different drying methods were used to compare the efficiency and product characteristics.

1. Freeze-drying: The samples obtained from the different treatments described above were lyophilized using a Savant MicroModulyo Boc Edwards Lyophilizer, at -40 °C, with $3x10^{-2}$ mbar pressure, for 72 hours. The dry products obtained were ground in a knife mill and stored in amber vials, at temperatures below 0 °C;
2. Drying at oven (45 °C) under vacuum: The aliquots obtained from the different treatments were placed in an Heraeus T48 RTV220 oven, maximum temperature 180 °C, under vacuum, with the temperature controlled at approximately 45 °C, for 15 days. After this period, it was observed that the samples were not dried and presented fungal contamination. The samples subjected to this type of drying were therefore discarded;
3. Drying in oven at 40 and 105 °C: The treated samples were placed in an Heraeus, T4P RT360, FNC 30503 oven, at 105°C, for 1 hour and 30 minutes. They were then transferred to another oven (LEO, 694) at 40 °C, for approximately 10 days. The dried samples were ground in a knife mill and stored in amber bottles at -20 °C until use.

Particle size distribution

The milled samples obtained from the freeze-drying and oven-drying (105 °C/40 °C) were sieved sequentially through sieves with mesh apertures of 600, 500, 425, 250, 125, and 63 μm, for 15 minutes (Brazilian Pharmacopoeia, 1988b). The retained fractions were collected , and the particle size range that gave the highest flour yields was selected. The flour was then collected and stored.

Measurement of pH

The pH was determined using a pHmeter (Ultrabasic, Model UB-10, Denver Instrument) according to AOAC (1990a), with minor modifications. The instrument was calibrated with standard pH values of 4.0 and 7.0. Concentrations of 2% sample in distilled water and mineral water (w/v) were prepared in triplicate and placed in a magnetic stirrer for 30 minutes. The samples were then decanted for 10 minutes for pH determination.

Loss on drying

Samples of byproduct were analyzed in triplicate, using the methodology described in the Brazilian Pharmacopeia (1988b). One gram of each sample was placed in an oven (Quimis) for 5 hours, at approximately 105 ºC. After 2, 3, 4 and 5 hours, the byproducts were removed and placed in a desiccator for 30 minutes each time, until successive weighing differed from each other by not more than 5 mg.

Content of total dietary fiber, soluble and insoluble fiber

The methodology used to determine the total dietary (TDF), soluble (SF) and insoluble fiber (IF) is described by the AOAC (1990b). This process was carried out in a collaborative work with the Laboratory of Microbiology and Food Science of the ICTA (Institute of Science and Technology – Federal University of Rio Grande do Sul).

Statistical analysis

The data, obtained in triplicate, were analyzed by ANOVA using Prism 5.0 (GraphPad Software, Inc., CA, USA). Statistically significant differences between the means were compared by Tukey's test, with a confidence level of 95% ($p < 0.05$).

RESULTS AND DISCUSSION

Drying processes

The byproduct aliquots subjected to lyophilization showed a yellow-orange color at the end of the process, with a characteristic orange odor, except for the sample subjected to the Clevenger extraction of volatile oil. This sample presented a dark color, probably due to the burning of sugars during the process. The oven dried aliquots showed a dark color, with low characteristic orange flavoring and a high burnt sugar odor.

Oven drying of the samples is a low cost process compared to lyophilization. However, this advantage did not outweigh the advantages of drying in a lyophilizer, for which the samples presented a more pleasant flavor, better appearance, and maintenance of phytochemical composition. This latter characteristic is extremely important, as it provides bioactive compounds that are responsible for many beneficial properties for the organism.

In the oven drying, samples were subjected to temperatures of approximately 105 ºC, for 1 hour and 30 minutes. It was previously demonstrated that changes in functional properties, as well as in the content of polyphenols, tannins, anthocyanins and proteins occur above 65 ºC (Larrauri, 1999). Samples subjected to this type of drying were discarded and an alternative source of dietary fiber developed, as no significant advantages were observed in relation to the lyophilized dried samples. In addition, similar results were found for pH and fiber content.

Particle size distribution

Byproduct aliquots with particle sizes of between 600 and 125 µm were selected due to the high yield of material obtained in this range, thereby avoiding excessive losses of material. This is also considered a good particle size interval for handling by producers and consumers, and for ingestion. The yields were particularly higher, on average, in the 450 and 250 µm mesh size. Table I shows the yields of total material retained in the selected mesh size, for the samples subjected to different treatments and drying processes.

TABLE I - Yield, as percentage values, of byproduct aliquots subjected to different treatments and drying processes, retained sieves with mesh size of between 500 and 125 µm

Treatment/Drying	Yield (%)
Pressing/freeze-drying(1)	87.96
Pressing/oven drying(2)	85.74
Clevenger/freeze-drying(3)	97.65
Clevenger/oven drying(4)	99.21
Peeling by hand/freeze-drying(5)	79.84
Peeling by hand/oven drying(6)	93.65
Washing/freeze-drying(7)	90.33
Washing/oven drying(8)	87.49
Not treated/freeze-drying(9)	90.27
Not treated/oven drying(10)	90.84

(1): pressed sample, dried by lyophilization; (2): pressed sample, dried in oven; (3): Clevenger extracted sample, dried by lyophilization; (4): Clevenger extracted sample, dried in oven; (5): hand-peeled sample, dried by lyophilization; (6) hand-peeled sample, oven dried; (7): washed sample, dried by lyophilization; (8): washed sample, oven dried; (9): untreated sample, dried by lyophilization; (10): untreated sample, oven dried.

We selected a particle size range of between 500 and 125 µm, considering this a reliable and homogeneous interval. This range also presented the highest yield of products. Barroto *et al.* (1995) studied the water retention capacity of orange fibers of different diameters (1000, 500, 280, 200 and 110 µm) and observed that as the particle size decreases, there is a proportional reduction in water retention capacity. This is due to changes in the fiber matrix structure, breaking the pores, which leads to compression of the fiber and causing it to retain therefore less water. According to Larrauri (1999), the particle size influences the texture, appearance and quality of foods containing fibers, therefore a particle size of between 430 and 150 µm is recommended.

In the study carried out by Figuerola *et al.* (2005), the authors determined the fiber content in orange peel, using a particle size of between 600 and 460 µM, as this range did not alter the texture of the concentrate fibers due to their moisture characteristics. According to the method described in the AOAC (1990b) for determination of total dietary, soluble and insoluble fiber, the suggested particle size is between 500 and 300 µm. Sáenz *et al.* (2007) evaluated the fiber content in dried byproducts of orange

juice extraction (Valence cultivar), with grain sizes of between 600 and 500 µm.

In contrast, Bortoluzzi and Marangoni (2006), in their dietary fiber characterization study on orange juice byproducts with dry bagasse, particle sizes of 30 and 50 µm were used, contradicting the previously defined particle size. Surprisingly, the authors obtained high fiber content and high water retention capacity, compared with those found by other authors. A study by Canteri-Schemin *et al.* (2005) evaluates the effect of particle size on pectin content extracted from apple byproduct flours. The authors observed that the farinaceous fraction with particle size greater than 600 µm showed low pectin extraction yields, while the fractions with particle size of between 250 and 106 µm provided the highest pectin extracted yield, comparing all the tested fractions.

Measurement of pH

According to Gorinstein *et al.* (2001), soluble dietary fibers increase in viscosity when mixed with water. The authors therefore attempted to establish an aqueous byproduct concentration of 2% for pH measurement, since they observed that 1g of sample in 10 mL of water (as determined by the AOAC (1990a) increased the viscosity of the solution too much, no longer characterizing it as a liquid, but as a gel. Table II shows the pH values of the samples subjected to different treatments, with values ranging from 3.47 to 3.96.

Higher pH values, ranging from 3.83 to 3.96, were observed for the samples subjected to pre-washing treatment. This can be explained by the fact that the pH of water is more alkaline than that of the byproducts, and this sample remained in contact with large amounts of water. The samples subjected to hydrodistillation extraction of essential oils showed lower pH values (3.76 to 3.79) than those found for samples subjected to washing, but higher than those observed for other samples. Although it contained more water in the product, this sample showed more acidic pH than that of the washed sample, which may be related to the acidification of water during the extraction process, by the diluted acid byproducts.

There was no significant difference between the three pH measures ($p < 0.05$) for the same treated and dried sample. It was found that the pH value of the hand-peeled samples did not show any significant difference between those subjected to freeze-drying and those obtained after oven drying, including when they were reconstituted in distilled or mineral water (Table II).

On the other hand, samples subjected to the pressing and Clevenger treatments, as well as those without the application of any treatment, showed no significant differences between the pH means of the samples reconstituted in distilled and mineral water, but its pH values differed significantly between lyophilized and oven dried aliquots. The mean pH values of the washed sample showed a statistically significant variation in the comparison of different drying processes, and also in the comparison of reconstitution media i.e. distilled water or mineral water (Table II).

All samples subjected to the same drying type and water reconstitution showed statistically significant

TABLE II - Mean and standard deviation (SD) of pH values from byproducts submitted to different treatments and drying processes. Samples were reconstituted to 2% in distilled or mineral water, in triplicate, separately

Sample	pH mean ± SD (sample at 2% in distilled water – pH distilled water = 5.85)*	pH mean ± SD (sample at 2% in mineral water – pH mineral water = 6.96)*
Pressing/freeze-drying	3.49 ± 0.02Aa	3.47 ± 0.01Aa
Pressing/oven drying	3.58 ± 0.02Ba	3.60 ± 0.01Ba
Clevenger/freeze-drying	3.76 ± 0.01Ca	3.76 ± 0.01Ca
Clevenger/oven drying	3.79 ± 0.00Da	3.78 ± 0.01Da
Peeling by hand/freeze-drying	3.51 ± 0.00Aa	3.51 ± 0.01Ea
Peeling by hand/oven drying	3.53 ± 0.01Aa	3.52 ± 0.01Ea
Washing/freeze-drying	3.89 ± 0.02Ea	3.96 ± 0.02Fb
Washing/oven drying	3.87 ± 0.01Ea	3.83 ± 0.01Gb
Untreated/freeze-drying	3.66 ± 0.03Fa	3.65 ± 0.01Ha
Untreated/oven drying	3.58 ± 0.01Ba	3.59 ± 0.01Ba

*Different lower case letters indicate statistically significant variation between values of the same row ($p < 0.05$); different uppercase letters indicate statistically significant differences between means of the same column ($p < 0.05$).

difference in average pH, except for the samples subjected to pressing, which showed no significant difference in pH when compared to the hand-peeled samples (both lyophilized) and to the untreated samples (both oven dried).

The screening performed by Grigelmo-Miguel and Martín-Belloso (1999) showed a pH range of 3.63 ± 0.03 to 3.86 ± 0.01 for dietary fibers from dried byproducts of three varieties of *Citrus sinensis*. Disparate results were observed in the study of Bortoluzzi and Marangoni (2006), in which a pH value of 3.47 was obtained for the flour from a mixture of sweet orange (*Citrus sinensis* L.) and Valencia orange (*C. aurantium* L.). It is well established that the pH of fibers may range according to the orange cultivar studied, the region where orange was grown, and the degree of ripeness of the analyzed fruit (Primo Yúfera, 1979; Salunkhe, Bolin, Reddy, 1991).

Loss on drying

The samples remained 5 hours in an oven, at approximately 105 °C. There was no difference of more than 5 mg between the last two successive weights. Table III shows the results of loss on drying after 5 hours of experiment. After drying in an oven, at approximately 105 ºC, the values for lost material ranged from 9.22 0.02% to 18.48 ± 0.52%.

The extraction of essential oil in a Clevenger apparatus and subsequent drying in an oven provided a sample with lower moisture content. This result can be explained by the fact that in the oven, the aliquot was subject to a high heat, evaporating a much higher content of water compared to the same treated sample, dried in a lyophilizer. This finding applied to all the samples, since they showed less loss on drying when previously dried in an oven. It is therefore concluded that this previous drying process generated a greater loss of material, compared to that obtained from the lyophilized samples.

The sample with the highest amount of volatile material was the lyophilized one, submitted to Clevenger extraction. This fact can be explained by standardized lyophilization time, For more effective drying of this sample, longer time would necessary, as this aliquot previously contained more water when compared to the others, due to the hydrodistillation, in which a large amount of water is add to the byproduct. After the extraction process, a small amount of water remained in the sample, an inherent fact of the process.

There were no significant statistically differences ($p<0.05$) between the triplicate analysis of samples subjected to the same treatment and drying process. Analyzing the samples subjected to the same treatment, all presented statistical significant differences in the comparison by the two drying procedures (lyophilization and oven drying) ($p<0.05$). Among the different treatments, when submitted to freeze-drying, there was no significant difference between pressing, peeling by hand and untreated samples. Comparing the oven dried samples, there was no significant variation for those subjected to Clevenger extraction and washing also, separately, for the treatment by the hand peeled and untreated samples.

The investigation carried out by Bortoluzzi and Marangoni (2006) showed a loss on drying of 9.28% for a flour obtained from the mixture of sweet orange (*Citrus sinensis* L.) and Valencia orange (*C. aurantium* L.). Rincón *et al.* (2005), characterized the chemical composition

TABLE III - Percentage average loss of volatile material from samples, performed in triplicate, after 5 hours of drying in oven at approximately, 105 °C. The standard deviation (SD) and relative standard deviation (RSD) were calculated and are shown in the table

Treatment/Drying	Average loss (%) ± SD*
Pressing/freeze-drying	14.94 ± 0.43a (RSD = 2.87)
Pressing/oven drying	12.47 ± 0.17b (RSD = 1.33)
Clevenger/freeze-drying	18.48 ± 0.52c (RSD = 2.79)
Clevenger/oven drying	9.22 ± 0.02d (RSD = 0.22)
Peeling by hand/freeze-drying	14.13 ± 0.30a (RSD = 2.15)
Peeling by hand/oven drying	11.26 ± 0.17e (RSD = 1.54)
Washing/freeze-drying	10.97 ± 0.39f (RSD = 3.59)
Washing/oven drying	9.48 ± 0.09g (RSD = 0.90)
Untreated/freeze-drying	14.28 ± 0.54a (RSD = 3.80)
Untreated/oven drying	11.33 ± 0.29f (RSD = 2.59)

*Different letters indicate statistically significant differences between values ($p < 0.05$).

of fruits from Venezuela, finding a moisture value of 3.31 ± 0.190% for *C. sinensis* L. lyophilized peel. In the study of Sáenz *et al.* (2007), three moisture contents were selected from a flour developed from industrial orange juice waste: 25%, 15% and 10%, in order to compare them with the fiber contents after incorporation of this flour into an energy-enriched product. They found that the loss on drying varies, according to the abovementioned criteria for pH value.

Content of total dietary, soluble and insoluble fiber

Table IV shows the results of fiber content for the samples. According to the table, the sample submitted washing/lyophilization showed a very different fiber content compared to the other samples. Despite having higher total dietary fiber content, this sample contained low amounts of soluble and high of insoluble fibers. The SF/IF ratio of this sample (1:11.45) showed a great discrepancy between the contents of these dietary fibers.

Comparison of the samples showed greater differences between TDF content mainly for pressing/lyophilization and washing/freeze drying of the samples (variation of 20.3%). Among the SF contents, the greatest difference (14.91%) was shown in the comparison of peeling by hand/freeze drying and washing/lyophilization. The same samples also showed the highest variation in IF content (33.74%).

The washed product showed the highest TDF content (62.74%), however, its SF content was very low (5.04%). It is known that generally, the most suitable SF/IF ratio for food ingredients is close to 1:2 (Primo Yúfera, 1979; Jaime *et al.,* 2002) and, as this sample presented a ratio of 1:11.45 ratio, it was therefore discarded as a product for human health. The low SF content of this sample may be due to the fiber loss in the washing process, knowing its water solubility.

Comparing the pressed samples dried by lyophilization and by oven drying, we observed higher levels of TDF, SF and IF for the oven dried aliquot (46.40%, 19.55% and 26.85%, respectively). However, the freeze-dried samples presented a SF/IF ratio (1:1.41) closer to the ideal ratio. The variations in TDF, SF and IF contents between these two samples were 3.96%, 1.96% and 2.00%, respectively. The difference in SF/IF ratio for the samples was 2.84%. Considering the importance of an adequate ratio, the oven-dried samples were discarded, as their use showed no clear advantages.

The Clevenger extracted sample showed mean levels of fiber (TDF: 46.20%; SF: 19.58%; IF: 26.62%) when compared with the other treatments. Although this sample was in contact with water during the extraction, at the end of this process, most of the water was maintained with this product, in contrast to the washed sample. Therefore, this treatment presented a product with interesting TDF content and good ratio between the fiber types. Allied to this, due to the removal of compounds, which are responsible for the bitter taste of the product, this treatment showed promise in the development of products with improved sensory characteristics.

Peeling by hand lead to a product with lower TDF content (43.91%), being higher only than the value for the pressed sample (42.44%), with a difference of only 3.35%. This can be explained by the fact that peel were removed manually and contains fibers, besides the albedo and flavedo. Additionally, soluble fibers, such as pectins, are found in abundance in the albedo, which explains the lower SF/IF (1:1.20) ratio found for this sample.

The untreated samples showed the highest levels of TDF (47.30%) and IF (27.54%) compared with those submitted to any kind of treatment (excepting the washed sample). In terms of SF content, this aliquot showed the second highest content (19.76%), lower only than the hand-peeled sample (19.95%), but without any significant differences between the values in terms of deviations

TABLE IV - Percentage composition of total dietary fiber (TDF), soluble (SF) and insoluble (IF) fibers from different samples, obtained by triplicate analysis

Treatment/drying	TDF* (%)	SF* (%)	IF* (%)	SF:IF Ratio
Pressing/freeze-drying	42.44 ± 1.32a	17.59 ± 0.78a	24.85 ± 1.12a	1:1.41
Pressing/oven drying	46.40 ± 1.45a,b	19.55 ± 1.02a	26.85 ±1.15a	1:1.37
Clevenger/freeze-drying	46.20 ± 1.67a,b	19.58 ± 0.89a	26.62 ±1.23a	1:1.36
peeling by hand/freeze-drying	43.91 ± 1.22a	19.95 ± 1.04a	23.96 ± 1.17a	1:1.20
Washing/freeze-drying	62.74 ± 1.89c	5.04 ± 0.23b	57.70 ± 1.98b	1:11.45
Untreated/freeze-drying	47.30 ± 1.34b	19.76 ± 1.03a	27.54 ± 1.27a	1:1.39

*At least one equal letter in the same column indicate statistically insignificant differences between values ($p < 0.05$)X

($p < 0.05$). Also, this sample presented an appropriate SF/IF ratio. Whereas essential oils influence the sensory characteristics of the product, further studies are needed conducted to ensure the benefits of consuming this flour.

Grigelmo-Miguel and Martín-Belloso (1999) studied byproducts from three *Citrus sinensis* cultivars (Navel, Valencia and Salustiana), demonstrating a total dietary fiber content for these varieties ranging from 35.4% ± 1.4 to 36.9% ± 0.3. The SF concentration varied from 11.3 ± 0.3% to 13.0% ± 0.2. For IF content, a range from 22.8 ± 1.1% to 25.5 ± 11.3% was observed. The SF/IF ratio, in the studied orange cultivars, presented a variation of between 1:1.76 and 1:2.26. In contrast, studies by Bortoluzzi and Marangoni (2006) with *Citrus sinensis* and *C. aurantium* byproducts showed TDF content of 68.6%, which is twice as high as those found in the other study. Also, the SF and IF contents (20.7% and 47.9%, respectively) were nearly twice as high, compared with the first cited study. The SF/IF ratio in this last study showed a value of 1:2.31, a little higher than that of the study of Grigelmo-Miguel and Martín-Belloso (1999).

In the study by Sáenz *et al.* (2007), which selected three moisture contents (25%, 15% and 10%) of the flour incorporated into an energy-enriched product, it was observed that higher humidity is correlated with lower content of total dietary fibers in the products. Therefore, the authors selected the product with 10% humidity, with 33% of orange powder, which showed: 22.8 ± 0.8% of TDF content, and 3.7 ± 0.2% of SF and 19.1 ± 0.4% of IF. The product developed in that study presented a SF/IF ratio of 1:5.16, quite different from the ratio considered adequate (1:2).

Figuerola *et al.* (2005) in their studies on byproducts from the juice extraction of *Citrus sinensis* var. Valencia, found high levels of TDF and IF (64.3% ± 0.30 and 54.0 ± 0.23%, respectively), while low SF content was observed for the same sample (10.28% ± 0.30). Thus, the SF/IF ratio in this study (1:5.3) proved to be far from the adequate value.

The study of Rincón *et al.* (2005) evaluated the characterization of chemical composition of fruits cultivated in Venezuela, observing, for *C. sinensis* L. byproduct flour, a SF/IF ratio most outlier from that considered appropriate for foods, comparing with other studies, with 1:27.14 SF/IF ratio value. This unlike ratio amount was due to the high content of IF (48.03% ± 2.04), representing approximately 96.5% of the TDF (49.78% ± 2.04) and low SF value (1.77 ± 0.02%), which is just 3.5% of TDF found in this product. Considering that the SF/IF ratio not only influences the overall food quality, modifying its physiological properties, but also significantly affects the sensory characteristics of the product (Schneeman, 1987), it can be concluded that the flour developed in the study by Rincón *et al.* (2005) would not be suitable for human consumption.

The dietary fibers variation found in different studies might be due to the type of treatment to which the byproducts of OJE were subjected. The present study demonstrated that this step is crucial, and causes considerable percentage differences in TDF content of the studied product.

CONCLUSIONS

The results demonstrated that the applied treatments on byproducts of orange juice extraction led to physical-chemical and fiber content variability, at different levels. Comparing the results, it was possible to conclude that the lyophilization drying process is more beneficial than oven-drying.

Due to the benefits of a daily intake of dietary fiber, it is important to develop alternative sources of these fibers, and to stimulate increased consumption of foods containing these substances. According to the results of this study, we identified three flours, dried by lyophilization, which generated promising results for the development of a rich dietary fiber product, with a good ratio between the types of fiber (soluble and insoluble). Among the flours developed in this study are: the treatment consisted in essential oil extraction by Clevenger; the product generated after pressing the byproducts, and the samples that were not subjected to any treatment. Further evaluations of the cost and feasibility of large-scale production, as well as new TDF evaluations linked to a cost/benefit estimate, will be carried out in the future.

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