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I am delighted and truly honoured to welcome you to IDS2018. As you know this is a landmark event representing completion of four decades since the establishment of this biennial series in 1978 at McGill University, Montreal, Canada.

I am happy to note that IDS2018, like its predecessor events, has attracted significant academic and industry interest in drying research and development from around the world. Exchange of ideas and dissemination of knowledge about both fundamental aspects and industrial applications have been the prime motivations for holding IDS events around the globe. As an interdisciplinary and multidisciplinary field which combines complex multi-phase transport phenomena with material science, drying is also highly energy-intensive and has controlling influence on the dried product quality in diverse industrial sectors. I believe that IDS and sister conferences devoted to drying have had a very significant impact on enhancements in drying technologies and innovation in dryer design. Much remains to be done of course.

I am sure the participants in IDS2018 will find the proceedings and networking opportunities rewarding and their stay in the wonderful historic city of Valencia memorable.

Finally, on behalf of all the attendees may I take this opportunity to thank and congratulate the Program Chair and his hardworking Organizing Committee supported by authors, reviewers and of course all the volunteers assisting with the smooth running of this complex event.

Arun S. Mujumdar

IDS Honorary Chairman

In this 40th anniversary of IDS it is a real honour to host at the Universitat Politècnica de València the celebration of IDS2018.

This is an event that, like in all other its predecessors, has attracted worldwide attention. Over 300 researchers from over 40 countries will present more than 450 papers at this event. As we can see IDS2018 promises to be a highly successful event. We are confident the founder of the IDS series Arun S. Mujumdar, would be very proud of it.

On this occasion we wanted to bring the event inside the University campus in order to get the students more actively involved in the organization and to make them aware of the importance and the impact of drying on many industries and different aspects of our life. This is a down to earth field that many times has been underestimated by our students and it's value needs to be recognized. Drying involves many aspects that may attract the interest of our students, from sustainability to product quality and the diversity of products. Valencia and the Mediterranean area have a long tradition of addressing the drying process; just remember the importance of dried fish on the Roman times that you still it find in our markets.

The quality of the contributions is very high and the discussion during the event will enhance fruitful exchanges among the participants. We hope that the academic environment will help to attain the goal of a friendly and fruitful interaction in the beautiful city of Valencia. We believe this event will fulfil your expectations.

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KEY NOTES

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Drying of mangoes applying pulsed UV light as pretreatment

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Abstract

High intensity pulsed UV Light is a non-thermal treatment used in sanitization of fruits and vegetables. In this work, we have applied high intensity pulsed UV light as a pretreatment for convective air-drying evaluating the benefits of the pretreatment to the drying process and to the nutritional quality of the dried product. Mangoes were subjected to pulses of UV light. The pretreated samples were further dried in a convective oven-drier until 90% of the initial water content has been removed. Drying kinetics, water apparent diffusivity, vitamin B, vitamin C content and total carotenoids content were analyzed. Pulsed UV light showed to be an interesting pretreatment for mangoes given the higher nutritional content of the dried product.

Keywords: *mango; drying; ultraviolet; vitamins; kinetics.*

1. Introduction

New food product development should focus on retaining as much of the naturally occurring vitamin content as possible, on increasing the availability of vitamins, and on reducing the appearance of undesirable breakdown products. Factors that play a role in the degradation of vitamins during food processing include temperature, air, light, moisture content, water activity, pH and enzymes related to the food spoilage [1]. Air-drying reduces the moisture content of the product decreasing the effect of one of the primary factors, the water activity, that determines the rate of vitamin deterioration by several biochemical reactions [1]. However, air-drying may increase vitamin loss by increasing the food exposition to high temperature for a long period of time.

Pulsed ultraviolet (UV) light processing of foods is a nonthermal technology used to enhance the quality and safety of foods. Its uses range from sanitization of foods (primary use) to extension of shelf life of fruits and vegetables, and enhancement of phytochemical content in fruits [2,3]. UV treatment is currently in use by the industry especially in sanitization of fruit and vegetables. Several studies reported that UV light also contributed towards the enhancement of the nutritional quality of food products, enhancing vitamin content, total carotenoids content, antioxidant capacity and other phytochemical properties [3–5].

Despite the importance of quality parameters in dried foods, most published studies are limited to determining the drying rate and some diffusional aspects and only few studies have focused on evaluating the changes on product quality after drying. In this work, pulsed UV light was applied as a pretreatment prior to air-drying for the drying of mangoes. The influence of the pretreatment and the drying process on vitamins B1, B3, B5, B6 and C, total carotenoids as well as its influence on the effective water diffusivity were evaluated.

2. Materials and Methods

2.1 Preparation of samples

Mangoes (*Mangifera indica* L. var. Tommy Athikins) were bought from the local market (Fortaleza, Brazil). Only fruits with same maturity stage were used. Cubic samples (side 10 mm) were obtained using a household tool from the mango flesh. The moisture content was determined by oven drying at 110ºC until constant weight (24h).

2.2. Pulsed UV light pre-treatment

The pre-treatment was carried out in a pulsed UV light equipment (SteriBeam model Xe Matric A, Germany) equipped with a xenon flash lamp (19 cm). The samples (12 g ± 1 g) were placed inside the treatment chamber of the equipment (20 cm wide × 14 cm deep × 12 cm high) and subjected to UV light pulses. The samples were subjected to 10, 20, 30, 40 and 50 pulses of UV light. Each pulse corresponded to a fluence (energetic density) of 0.36 J/cm^2 and were delivered in 250 μs. All experiments were carried out in triplicate.

2.3 Air drying

Drying kinetics were carried out in a conventional convective oven-drier. Air-drying experiments with mango samples were carried out at 0.5 m/s (air velocity) and 60 °C (temperature). The drying experiments were conducted in triplicates and completed when the samples lost 90% of the initial weight. For each experiment, 25 ± 1 g cubes of mangoes were distributed over a custom sample holder inside the drying chamber.

The air-drying kinetics of mangoes was modeled assuming diffusion-controlled mass transfer. Only the falling-rate period (diffusion-controlled mass transfer period) was considered because the constant-rate period (heat transfer-controlled mass transfer period) was not observed. The model considered the solution of Fick's second law for cubic shaped samples (Equation 1) [6].

$$W(t) = W_{eq} + \left(W_{crit} - W_{eq}\right)\left[\sum_{n=0}^{\infty} \frac{8}{(2n+1)^2\pi^2} exp\left(-\frac{D(2n+1)^2\pi^2 t}{4a^2}\right)\right]^3 \quad (1)$$

2.4 Determination of vitamins

To determine the vitamins of the B complex, 1 g of fruit was homogenized with 6 mL of distilled water for 2 min using a cell homogenizer (Ultraturrax IKA model T25). The vitamins were extracted by adding sulfuric acid 0.25 M (1 mL) to the sample, which was heated for 30 min in a water bath (70 ºC). After cooling in an ice bath, the pH was adjusted to 4.5 using a 0.5 M sodium hydroxide solution. The sample was centrifuged at 8400× g for 10 min. The supernatant was collected and analyzed spectrophotometrically at 215 (Vitamin B5), 254 (Vitamin B1), 265 (Vitamin B3) and 716 (Vitamin B6) nm using water as blank. All analyses were carried out in triplicate and results were expressed as the vitamin gain/loss using the fresh fruit as reference.

2.5 Determination of total carotenoid content

The carotenoids were extracted milling 1 g of mango sample with 6 mL of distilled water in a cell homogenizer (Ultraturrax IKA model T25). Hexane (5 mL) was added and the mixture was vigorously stirred in a vortex for 1 min. The supernatant (hexane phase) containing the lipid fraction was collected and analyzed spectrophotometrically at 452 nm to determine the total carotenoids content, using hexane as blank. All analyses were carried out in triplicate.

3. Results and Discussion

3.1. Pulsed UV light pretreatment

Mangoes presented an initial moisture content of 84.2 ± 0.4 g water/100 g fresh fruit (wet basis). The moisture content of the samples decreased during the UV light pretreatment,

reducing the initial moisture content by 4.3 to 15.9%. Figure 1 presents the water loss observed as a function of the energetic density that was applied.

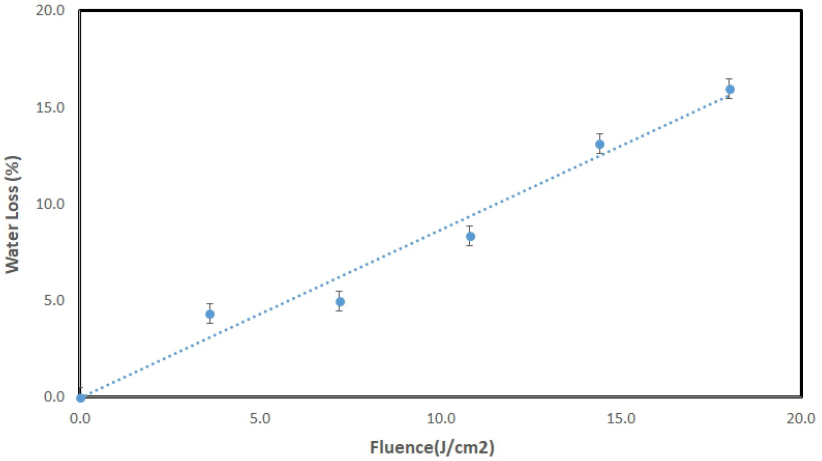

Fig. 1 Water loss during the pulsed UV light pretreatment as a function of the UV dosage applied to the samples.

Overall, pulsed UV light pretreatment contributed to an initial reduction of the moisture content of the samples.

3.2. Air-drying experiments

Figure 2 presents the experimental drying kinetics of mangoes obtained with and without application of pulsed UV light. Drying kinetics of mangoes presented the typical behavior observed for other fruits [7]. Only the falling rate period was observed during the drying process. Drying was carried out until 95% of the initial moisture was removed, corresponding to a final moisture content of 0.08 ± 0.01 g water/g dry matter.

The water apparent diffusivities in mangoes were calculated using Equation 1. The results are presented in Table 1. The diffusion model used in this work was adequate for describing the drying kinetics of mangoes cubes under the different experimental conditions, presenting R^2 values between 0.986 to 0.995.

Despite the initial reduction in moisture content attained during the pretreatment, the pulsed UV light pretreatment did not reduce the air-drying time. In fact, the samples took between 2 and 16% more time to dry than the untreated sample. It must be stated that the required air-drying time to reduce 80% of the initial moisture content was statistically similar among the sample not subject to pretreatment and the samples pretreated with a total energetic density above 10.8 J/cm^2.

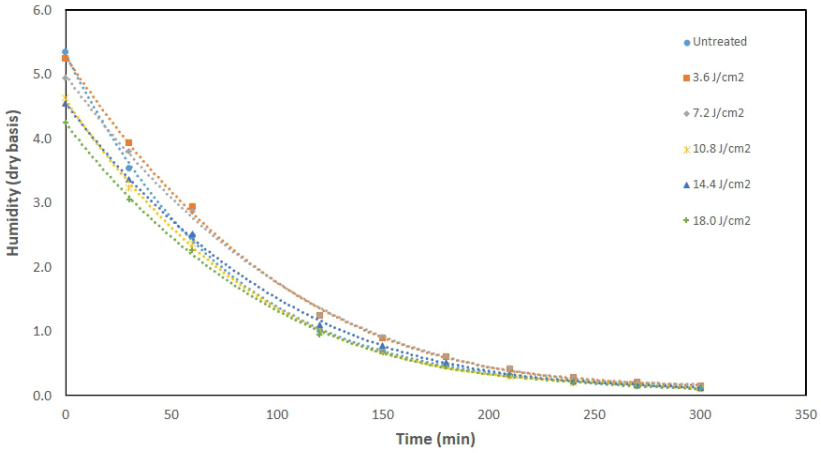

Figure 2. Moisture content (in dry basis) as a function of drying time for the samples with and without pulsed UV light pretreatment.

Table 1. Water apparent diffusivity of mangoes subjected to pulsed UV light pretreatment.

Total energetic density applied (J/cm^2)	Number of UV light pulses (#)	Apparent diffusivity (10^7 m^2/min)	R^2
0.0	0	4.37 ± 0.38 [a]	0.995
3.6	10	3.90 ± 0.03 [b]	0.986
7.2	20	3.76 ± 0.09 [b]	0.986
10.8	30	4.26 ± 0.12 [a]	0.987
14.4	40	3.92 ± 0.27 [a]	0.988
18.0	50	4.10 ± 0.01 [a]	0.991

As such, if the main objective of the pretreatment is to reduce the air-drying time, the pulsed UV light pretreatment would not be a proper technology to achieve this goal.

3.3. Vitamins and carotenoids content

The drying process decreased the content of vitamins and total carotenoids in mangoes. The retention of vitamins and total carotenoids depended on the vitamin, the use or not of pulsed UV light pretreatment and the intensity of the pretreatment.

Figure 3 presents the retention of vitamins and total carotenoids for non-pretreated mangoes subjected to air-drying. Total carotenoids content presented the lowest retention ratio (24.6

%), while vitamin B6 presented the highest retention ratio (97.7 %). The changes observed in Figure 3 represents the effect of heating in the vitamins and total carotenoid content.

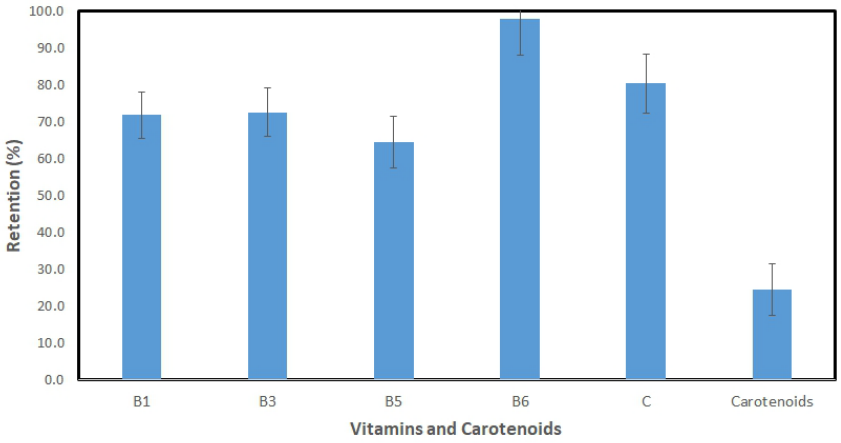

Figure 3. Retention of vitamins and carotenoids in mangoes after air-drying without UV pretreatment.

The pulsed UV light pretreatment showed a positive effect on vitamins B1, B3 and B5 when up to 7.2 J/cm2 where applied to the samples. Under such conditions, the dried product would present between 10.0 to 28.8 % more vitamins B1, B3 and B5 than the untreated dried product. Above the 7.2 J/cm2 level, the effect of the pretreatment is statistical non-significant (at a 95% confidence level), due to a probable degradation of these vitamins by UV light.

Vitamin B1 (thiamin) is the most thermolabile among the B vitamins, so degradation due to thermal effect was expected [8] and was confirmed by the experimental data. The effect of pulsed UV light seemed to be mainly positive and a trend of higher retention of vitamin was observed. The high energy of the pulsed light treatment may have broken the bound of the phosphorylated vitamin, changing it to its free and bioavailable form, thus resulting in a slightly higher content of this vitamin in the treated dried product.

The retention of vitamins B3 and B5 followed a similar trend. As with vitamin B1, the energy delivered by pulsed UV light might have broken the bound between the vitamin and its bounds, releasing an amount of vitamin that otherwise would be unavailable. This is plausible because vitamins, nucleotides and coenzymes absorbs UV energy and could use this energy to break the chemical bond between vitamins and nucleotides, and vitamins and coenzymes. The energy absorbed from pulsed UV light causes physical damages to membranes and other structures due to the release of chemical and physical bonds in these structures [9]. Thus, the same energy that is responsible for bond breaking damage in microorganisms can be responsible for the bond breaking reactions that release the bonded vitamins and transform it in free and bioavailable vitamins.

The pulsed UV light pretreatment had a significant negative effect on vitamin B6. The retention of vitamin B6 that was at 97.7 % for the dried untreated mango, dropped to retentions levels between 48 to 61 %. This vitamin is considered light-sensitive. As such, both UV light and visible light released by the pulses have degraded this vitamin resulting in a significant reduction in its content when the UV pretreatment was applied.

The UV pretreatment did not change significantly the amount of vitamin C in the dried product. The pretreatment was only statistically different (at a 95% level of confidence) when a fluence of 12 J/cm^2 was applied. Under this condition, the amount of vitamin C was 28.6 % higher than the untreated sample. Vitamin C is considered a light sensitive vitamin that present high UV light absorbance in the germicidal range (215 to 260 nm) but does not absorb significant light above 300 nm. The equipment used in our experiments releases light in a spectrum between 310 and 400 nm, thus it has not affected the vitamin C content of the mangoes samples.

The UV pretreatment increased the total carotenoids when a fluence up to 12 J/cm^2 was applied. Overexposure to high intensity UV light tended to degrade the carotenoids. The increase in the total carotenoid content can be attributed to an alteration of the carotenoid-binding protein with a consequent increase in availability of free carotenoids, which has been reported previously for mango juice subjected to UV light treatment [10].

4. Conclusions

Pulsed UV light decreased the initial moisture content of the mangoes samples but did not increase the effective water diffusion of mangoes nor reduced its drying time. The application of pulsed UV light increased the availability of vitamins B1, B3, and B5, vitamin C and carotenoids in the dried product. The light sensible vitamin B6 degraded significantly when compared to the untreated dried mango samples. Overexposure to pulsed UV light (dosages greater than 15 J/cm^2) degraded all vitamins and carotenoids, thus the UV dosage must be carefully optimized. Pulsed UV light can be a potential pretreatment for drying fruits given its simplicity, rapid application and because it confers better nutritional quality to dried mangoes.

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Process intensification and process control in freeze-drying

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Abstract

Widespread use of advanced process control allows reduction of costs, by reducing drying time and energy consumption. The "control of the freezing stage" (by forced nucleation) also appears to be beneficial to process intensification, as it can impact the product structure and modify the product resistance to mass transfer. An alternative way to increase the drying rate is the use of organic solvents as they can lead to larger solvent crystals, hence lower product resistance to vapor flow.

Atmospheric freeze-drying may be a good alternative to vacuum freeze-drying, as a way of increasing process efficiency. A further improvement can be obtained by combining atmospheric or vacuum freeze-drying with new technologies.

A further step towards process intensification is given by continuous plants, as this allows a dramatic increase in throughput and product quality uniformity.

Keywords: *freeze-drying; process intensification; controlled nucleation; continuous process.*

1. Introduction

The freeze-drying process is widely used in pharmaceuticals manufacturing, as well as for drying foodstuffs, as high-quality products can be obtained due to the low operating temperatures. Anyway it is quite an expensive process, and this fact limits applications in food and probiotic industry. Reduction of costs, by reducing drying time and energy consumption, and a better uniformity in end-product quality can be reached by means of process optimization and widespread use of advanced process control, but there is a big potential for process intensification by process modifications.

According to Stankiewicz and Moulijn [1], process intensification can be obtained by modifying equipment, to optimize critical parameters (e.g., heat transfer and mass transfer), or by process intensifying methods, changing the process or using alternative energy sources. Proposed control approaches, and in particular advanced process control, will be first discussed in this review. Other possibilities for process intensification will be then discussed, focusing in particular on controlled nucleation and process modifications in production of pharmaceuticals.

2. Process optimization and widespread use of advanced process control

Improvement of the process control has been recognized as a development need for the pharmaceutical industry over the last thirty years, but relatively few changes occurred at the production scale. It must be evidenced that today even the most advanced industrial freeze-dryers have no robust process control. An open-loop control approach is generally adopted, but rarely the cycle has been really optimized, and cycle transfer between different pieces of equipment or scale up is generally challenging [2-5].

Effective monitoring and control systems are required to manage the process in such a way that product quality is not jeopardized at the end of the process. Besides, as the duration of the freeze-drying process is an important concern, the control systems should be able to optimize in-line the operating conditions, namely the pressure in the drying chamber and the heating power, i.e. the temperature of the heating source. In the following different the methods proposed will be analyzed.

A first group of methods is based on the measurement of product temperature obtained through a thermocouple (or another temperature sensor); they optimize only the temperature of the heating element. In this case it is possible to use just the temperature measurement, with a fuzzy logic-based algorithm. Alternatively, a mathematical model can be coupled to the experimental measurements, thus obtaining a soft-sensor; this can be used

to calculate the optimal control actions using, for example, a PI algorithm, or calculating in line the design space of the process [6-8].

The second group of methods is based on the pressure rise test [9]; this is a technique for the in-line process identification that allows estimating both the state of the product (temperature and residual amount of ice) and the values of some model parameters. By this way a mathematical model can be used to optimize the process using, for example, a classic proportional algorithm, or an advanced Model Predictive Control algorithm [10,11]. Control logics based on measurement of heat or sublimation flux will be also discussed.

Control procedures are available also for the secondary drying step. The key feature of the method is the coupling of the measurement of the desorption rate, obtained by means of the PRT or other devices, with a mathematical model of the process [12].

3. Control of the freezing stage

Freezing plays a fundamental role in the freeze-drying process, as it determines product morphology and, therefore, drying performances and drug stability [13]. Therefore, it has become essential to increase the knowledge and control over this process. A brief overview of the tools which allow control of the impact of freezing on product morphology, and, consequently, on drying efficiency and product quality will be presented.

During freezing, the stochastic nature of nucleation is regarded as a demerit, because it is directly linked to vial-to-vial variability in terms of product morphology, drying behavior and, ultimately, product stability. Hence, lots of attention was given to the control of the temperature at which nucleation occurs, trying at least to make it as uniform as possible over a batch of vials. In fact, it is not actually possible to really “control the nucleation” even if this term is often used for simplicity; as said before, what is possible to do, is to force nucleation to take place at a given temperature and control the batch temperature holding it until nucleation is completed. The higher the nucleation temperature, the larger the ice crystal size, and thus the lower the cake resistance. In recent years, almost all the principal freeze-dryers manufacturers developed their proprietary technology to induce ice crystallization and to reduce the time span for completing the freezing in all the vials of the batch. Many patents were deposited, and different technical solutions were made commercially available, especially at the pilot-scale; even if many difficulties still limit somehow the application to large production scales of some methods, controlled ice nucleation starts moving also into manufacturing [14,15].

Ultrasound nucleation has been the object of an extensive research, but the passage from lab to commercial scale faced strong difficulties. The main problems were related to the difficulty to efficiently propagate the ultrasonic waves, and to scale the process, as the

optimum ultrasonic frequency depends on the set up. Actually commercially available equipment adopts either a variant of the ice fog, or the depressurization method, or the vacuum induced nucleation. The ice fog concept is probably the first method proposed to control nucleation (as it is known since 1990), but only recently the technical developments made it really suitable for practical applications.

The easiest and cheapest way to control nucleation is surely the vacuum induced surface freezing, VISF (also known as vacuum induced nucleation, VIN), as this technology requires no hardware, any equipment can be easily retrofitted, and there are limited sterility concerns (related to sterilization of large valves required for rapid depressurization) [16]. It is important to evidence that notwithstanding the concern for possible product denaturation, in particular with the depressurization and the VIN method (for the possible presence of small bubbles), all the discussed controlled nucleation technologies gave particularly good results; in fact, as a consequence of the larger ice crystals (and thus of their reduced solid surface), an improved stability even for very sensitive products was observed.

Thus the described methods are effective approaches to process intensification, but to guarantee the required final quality and uniformity of the batch, they must be coupled with methods that assure the temperature uniformity of all the vials. In all cases, the depressurization rate is the element that may limit the applicability to large units, as it poses constrains to the geometric characteristics of the apparatus and of the depressurization circuit.

4. Process modifications

The use of a strictly organic or organic/water system is beneficial to both product quality and process optimization. Potential advantages and disadvantages of use of organic solvents and cosolvents (mixed with water, that up to now has been the most common solvent used) in freeze-drying are widely discussed by Teagarden and Baker [17]. The main advantage in addition to the increase in solubility of the product, is the increase in rate of sublimation and hence decrease of drying time. However, before a specific solvent is used in the manufacture of a parenteral product, lyophilization professionals have to carefully weigh advantages and disadvantages.

The use of organic liquids, either as solvent or co-solvent, produces larger ice crystals and, thus, increases the average diameter of the pores created during ice sublimation. As a result, the product resistance to vapor flow decreases and, if product temperature does not change, the rate of sublimation increases, as said above. In addition to the reduction in product resistance, most of the cosolvents used in freeze-drying applications increase the rate of sublimation because they have a higher vapor pressure than water and, hence, they increase the driving force for mass transfer. In addition, a further reduction in energy consumption

results from the fact that the sublimation enthalpy of organic solvents is smaller than that of water ice. Unfortunately, organic solvents are rarely used in the manufacture of a lyophilized pharmaceutical product, mainly because of safety concerns. Specifically, attention has to be paid to how the organic solvents can be safely handled, e.g., preventing fires or explosions during their manipulation or avoiding contamination of the vacuum pump oil, which can decrease the pump efficiency and, hence, impede adequate control of pressure inside the equipment. Furthermore, in the case of mixture of solvents, monitoring the state of progress of drying might be very complex, because the various solvents can show significantly different rates of sublimation. Therefore, it is necessary to track the separation of the individual solvents, e.g., using sensors that are sensitive to only specific solvents and completely insensitive to others [18].

In food processing, vacuum freeze-drying produces dried products that retain almost all their original characteristics, e.g. color, flavor, and taste. The high specific surface area generally allows an easy and fast rehydratation. The drawback is represented by the cost of the operation: fixed costs can be high due to vacuum requirement, and the energy cost can be significantly higher with respect to other drying processes (the specific moisture extraction rate in a vacuum freeze-drying process is in the range of 0.4 kg of water per kWh). In order to reduce the cost and the energy consumption of the process, thus improving its sustainability, the atmospheric freeze-drying (drying with cold air or nitrogen at normal pressure) has been proposed [19,20]: in this case it is possible to achieve a specific moisture extraction rate ranging from 1.5 to 4.6 kg of water per kWh. For example it has been claimed that up to 35% of energy savings could be achieved when using atmospheric freeze-drying instead of vacuum freeze-drying for potato slices.

Most of the literature deals with atmospheric freeze-drying in fluidized bed dryers and in spray freeze-dryers. When the atmospheric freeze-drying is carried out in a fluidized bed it can take advantage of the high values of the heat and mass transfer coefficients; the product has to be frozen and granulated before drying. A drawback of the process is represented by the size reduction caused by mechanical cracking. As an alternative it is possible to carry out the process in a tunnel dryer, even if the heat and mass transfer is not so good.

A detailed comparison of vacuum and atmospheric freeze-drying has not been carried yet, and work is currently ongoing also in the labs at Politecnico di Torino. Surely drawbacks related to the limitation in process temperature, to avoid ice melting, and the necessity to recirculate and dry large volumes of gas at low temperature must be taken into account. In particular, atmospheric freeze-drying produces higher quality products (in comparison to traditional drying) but still entails long drying times. Energy consumption can be reduced by new technologies which use alternative forms and sources of energy for processing. These technologies can be applied to either enhance heat transfer between product and heat source, such as microwave, radiofrequency and infrared radiation, or simply intensify the rate of dehydration without increasing the amount of heat supplied to the product, e.g. by

using high intensity sonic and ultrasonic waves.

Spray freeze-drying seems to be a valuable alternative to produce a free-flowing powder, with high surface area, porous end product, and good instant characteristics, with enhanced solubility and a uniform and ultrafine particle size. Spray freeze-drying into liquids, gases (e.g. a refrigerated air stream), and into gases over a fluidized bed have been reported in the literature. This technology has also a good potential for continuous processes. Spray freeze-drying allows the massive production of pharmaceuticals for dry powder inhalation, and a more precise control of particle size compared to spray drying. Furthermore, the rapid cooling rates promote the formation of glassy water that is beneficial in preventing the aggregation of proteins during the cryo-concentration phase.

5. Continuous plants

Recently, many pharmaceutical manufacturers are trying to convert their processes in favor of continuous production. To achieve this objective, it is necessary to integrate those production steps, that are performed sequentially in a conventional batch configuration, in a continuous process, leading to more compact units with a higher degree of automation and fewer manual interventions. This is particularly true for the lyophilization of pharmaceuticals and biopharmaceuticals in unit-doses which, although it is a robust and well-established technology, still remains inefficient and expensive. For example, the drying behavior within a batch, as well as from batch to batch, is still a problem of deep concern, despite the elaborate equipment design and the sophisticated control systems recently introduced.

It must be noted that continuous freeze-drying find currently application mainly for soluble coffee production. Many patents have been deposited for food technology applications, but realizations are still very limited. In the pharmaceutical industry there are much more severe constrains, and just a relatively few patents have been deposited. In particular, so far, only two technologies have been proposed for the production of end-to-use lyophilized products in a continuous way [21,22]. In both technologies, processing time and equipment size is dramatically reduced, up to 10 times, and all manual interventions and breaks have been minimized reducing the risk of product contamination. Furthermore, in-line control can easily be implemented, and scale-up simply consists of adding parallel modules. Despite their numerous advantages, the application of these technologies to real cases in industry still requires time. The above proposed technologies are still in development, and their capability to work under GMP conditions, meeting all the stringent requirements of a pharmaceutical production, has not been completely demonstrated yet. Nevertheless, these solutions are concrete steps toward continuous manufacturing of lyophilized pharmaceuticals, similarly to what food industry did years ago.

6. Conclusions

Different approaches to process intensification, coupled with improved process control for freeze-drying have been discussed. In the last years, many progress have been carried out in process monitoring and control, including the control of the freezing step. Current research should favor the transition towards a more robust design of freeze-drying cycles, based on deep knowledge of phenomena involved rather than on empirical observations. This would be extremely beneficial, especially for the pharmaceutical industry, where particular emphasis is placed on product homogeneity and process control. Process modifications, like atmospheric freeze drying and spray-freeze drying are interesting alternatives whose real potentiality are currently under investigation. The most ambitious goal is certainly the realization of a continuous process at the industrial scale for pharmaceuticals.

The intensification of the process and the reduction of the processing costs, on the other way, may open new possibilities of wider application of freeze drying in processing of valuable foods and in particular of probiotics, for which the market is requiring higher qualitative standards.

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Supercritical CO_2 drying of food matrices

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Abstract

This work explore the use of supercritical CO2 drying as alternative technique for the obtainment of pasteurized and high quality dried product. Several tests were conducted on animal, vegetable and fruit matrixes in order to investigate the effectiveness of SC-CO2 drying process at different process conditions. Design of experiment was performed to find the optimal process conditions for vegetable and fruit matrices, using the final water activity of the products as key indicator for the drying efficiency. The inactivation of naturally present microorganisms and inoculated pathogens demonstrated the capability of SC-CO2 drying process to assure a safe product. Moreover, retention of nutrients was compared with conventional drying methods. Results suggest that supercritical drying is a promising alternative technology for food drying.

Keywords: *supercritical drying; carbon dioxide; food drying; microbial inactivation*

1. Introduction

Fresh food products, in particular ripped fruits and vegetables, are rich sources of nutrients with an important role in human health [1]. However, fresh products are seasonal and an optimal cold chain is needed to prolong their limited shelf life.
An alternative is represented by food dehydration, which is one of the oldest and widely used processes for the long-term maintenance of food products. By reducing the amount of moisture, the microbial and enzymatic activities are inhibited, promoting the extension of the product's shelf-life [2,3]. Conventional hot air-drying is one of the most commonly used dehydration process in food industry. Nevertheless the overall quality of the final product is often reduced by the combination of high temperatures and the presence of oxygen which promotes physical, structural, chemical and nutritional changes [4,5]. Higher retention of those compounds can be achieved using freeze-drying technology [6], however it is an expensive and very slow process, making it suitable only for high value foods [3,7].
Recently the use of carbon dioxide at supercritical conditions (Sc-CO_2) has been investigated as alternative drying food process, specifically for carrots [8], basil [9], mango and persimmon [10] and coriander [11], demonstrating to be a promising process for the retention of the original structure and the preservation of the most valuable compounds.
Within Sc-CO_2 drying the vapour-liquid interface can be avoided meaning that the water is removed as a liquid dissolved in the supercritical fluid. The result is a minor capillary stress for the product, which allows a better preservation of the original structure. Moreover, the critical point, and consequently the critical temperature (31.1°C), is low, which allows to operate at lower temperatures than conventional air drying, helping the prevention of the heat sensitive degradation's reactions and thus giving a final product with higher quality [8,9]. Sc-CO_2 have been largely investigated as alternative food pasteurization at low temperature [12] because it is able to inactivate microorganisms and enzymes.

The present work explore the use of Sc-CO_2 for drying and simultaneous pasteurization of foodstuff. The influence of process parameters (temperature, pressure, flow rate and treatment time) on the final water activity were studied within a Box Behnken Design method. Overall the results demonstate the possibility to obtain a high quality product microbiologically safe.

2. Materials and Methods

2.1 Sample preparation

Different types of food products were daily bought in the local market in Padua (Italy): red bell peppers (*Capsicum annuum, L.*), coriander (*Coriandrum sativum*), strawberry (*Fragaria ananassa*), apple (*Golden delicious*) and chicken breast fillet. The vegetables were cut into slices while coriander leaves were removed from the stem. The chicken breast fillet was cut into small cubes with a weigh of approximately 1g.

2.2 Sc-CO_2 drying apparatus and procedure

The high pressure carbon dioxide apparatus consists of a sapphire high pressure visualization cell (Separex S.A.S., Champigneulles, France) with an internal volume of 50 mL designed to withstand up to 400 bar and 100 °C. The plant includes a CO_2 tank, kept at room temperature, a chiller reservoir (M418-BC MPM Instruments, Milan, Italy), a HPLC pump (307 Gilson, Milan, Italy), and a thermostatic water bath (ME-Julabo, Seelbach, Germany) to keep the vessel at the desired temperature. Further details of the reactor and drying procedure are reported elsewhere [11,13]. Experiments were carried out between 40/60°C and 100/140 bar up to 16 hours of drying for red peppers, while 40°C and 100 bar were chosen for the other food products.

2.3 Experimental design

The Box Behnken Design was used to study the effect of supercritical CO_2 drying process parameters on the final water activity of treated red peppers. To quantify the relationship between the controlled input and the accomplished responses, a second order regression model was used. All the calculations were done using Minitab®.

2.4 Physical and chemical analysis

Water activity was measured (Hygropalm Rotronic, Bassersdorf, Switzerland) at the end of the process. Samples were weighted before (W_{start}) and after (W_{end}) the treatment and the weight loss in terms of percentage ΔW was calculated as $(W_{start}-W_{end})/W_{start}$. Chemical characterization was performed for flavonoids as previously described [11]. For all the HPLC analysis an Agilent 1260 system equipped with Diode array (126 series) and Ion trap Mass spectrometer (Varian/Agilent MS500) were used. For vitamin C 200 mg of grinded powder plant material were xtracted three times for 10 minutes in an ultrasound bath with 8 ml of solution composed of water with 1% (v/v) formic acid. Zorbax SB C3 4.6x 150mm (DTO Servizi, Spinea, Italy) was used for the stationary phase. Isocratic conditions of elution used two solutions: solution A was acetonitrile while solution B was water 1% formic acid. For the quantification, standard solutions of ascorbic acid (Sigma Aldrich, Milano, Italy) were used to build up a calibration curve in the range 3-120 ug/mL.

2.5 Microbial analysis

Mesophilic bacteria, mesophilic bacterial spores and yeasts and molds were counted before and after the treatments by means of the standard plate count technique, as previously described [11]. Breefly, mesophilic bacteria and spores were cultured using total plate count agar (Microbial Diagnostici, Catania, Italy) at 30°C within pour plate, while yeasts and molds were cultured with DRBC agar (Bitec S.r.l., Grosseto, Italy) supplemented with chloramphenicol at 22°C within spread plate. For the enumeration of mesophilic spores, the first dilution tubes were inserted in a thermostatic bath at 80°C for 10 min before plating. The incubation time for mesophilic bacteria and spores was 72 h, while 72-120 h for yeast and

molds. The enumeration was referred to the weight of initial fresh product and expressed in CFU/g. Reductions are expressed as $\log(N_0/N)$ where N_0 was the number of initial microorganisms in the untreated sample and N the number of viable microorganisms after the treatment, in CFU/g of fresh product. The limit of quantification was set to 200 CFU/g for the mesophilic bacteria and mesophilic bacterial spores, 2000 CFU/g for yeast and molds while the limit of detection was < 10 CFU/g < 100 CFU/g respectively. Experiments with inoculated pathogens (*E.coli*, *Salmonella* and *Listeria monocytogenes*) were performed on coriander, apple slices and strawberry slices following the protocol by Bordeaux et al [14]. Results were analyzed with one-way analysis of variance to compare effects of the different treatments with significance at $\alpha = 0.05$.

3. Results and Discussion

The drying kinetics, in terms of water activity and weight loss, were determined by increasing the drying time till a complete water removal. Figure 1 shows the water loss and water activity obtained during the drying of the red pepper. Similar behaviours were observed for the others food samples (data not shown).

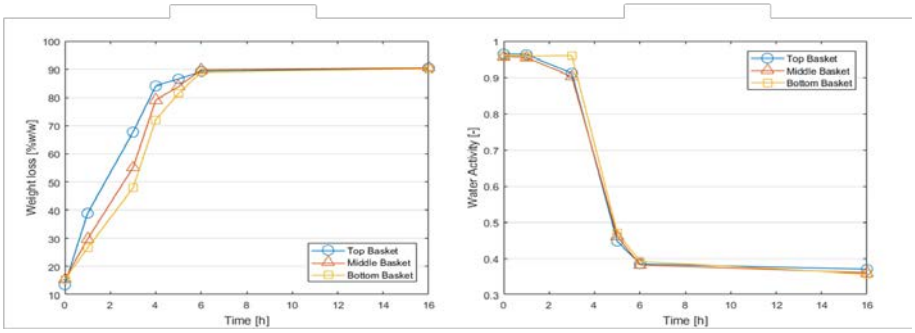

Fig. 1. Water loss (left) amd water activity (right)at different drying times for red pepper. Data are reffered to three different hights of the reactors. Experiments were carried out at 40°C, 100bars and 150kg/h flow rate.

The response surface methodology was chosen to quantify the relationship between the controllable input parameters and the obtained response surfaces, in order to find the influence of the process conditions over the product quality. Fig 2 shows results obtained at 40°C and 16 hours drying that highlight the influence of pressure and pump frequency on the final water activity of the sample. Response surface analysis on strawberry demonstrated a similar behavior (data not shown). To demonstrate the capability of the technology to retain the active components of the fresh products, some chemical analyses were performed on the Sc-CO_2 dried product.

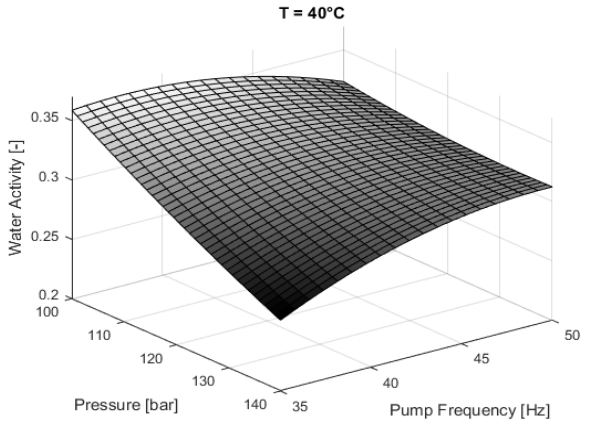

Fig. 2. Response surface for the water activity at 40°C and 16h drying as function of Pressure and Pump frequency.

For red pepper, the amount of flavonoids obtained was about 880 mg/100g of dried sample, reported in Table 1, are consistent with the literature; for instance Deepa et al. (2007) [1] reported phenolics in the range of 20–40 mg/100g of fresh product, which is similar to our results considering a loss of weight of 90% compared to the fresh product.

Table 1. Flavonoid and ascorbic acid content in dried red pepper (40°C. 100bar, 100kg/h flow rate)

Flavonoid [mg/100 g dry product]	**Ascorbic acid [mg/100g dry product]**
880.45 ± 2.4	1163.20 ± 5.3

The average content of ascorbic acid in fresh bell pepper is in the range of 64-220mg/100g of fresh product [1, 15]. As for flavonoids, we measured a higher content of ascorbic acid after Sc-CO_2 drying compared to the fresh product; the data can be explained with an apparent concentration of micronutrients caused by water removal during the process. Considering a water loss of about 90%, data of dried and fresh products are comparable and we can assert that SC-CO_2 drying technique is able to preserve the ascorbic acid content in the red pepper.

Microbiological inactivation was demonstrated on coriander, apple, strawberry and chiken breast fillet for the natural flora and specific pathogens (data not shown). Supercritical drying was able to complete inactivate yeasts and molds in all the samples considered; as regards bacteria, only the most sensitive mesophilia were inactivated on fruits, while a complete inactivation was possible on chicken. *E.coli, Salmonella* and *Lysteria monocytogen* were completely inactivate in all the food samples up to 8 log reduction.

4. Conclusions

Overall the results highlighted the potential of Sc-CO_2 drying technology to obtain a safe and dried products with unaltered nutritional value.

5. Acknowledgements

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Ultrasound enhancement of osmotic dehydration and drying - Process kinetics and quality aspects

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Abstract

The aim of presented studies is to investigate of influence of ultrasonic assistance on both osmotic dehydration and convective drying. A wide range of different materials, as well as several osmotic agents were tested.

The obtained results show that the use of ultrasound always accelerates the investigated processes. The application of ultrasound may reduce the energy consumption of drying. Qualitative studies of dried materials do not give a definite answer about the effect of ultrasound on the quality of the products. Mathematical modelling of the ultrasound assisted drying indicates that so named "vibration effect" plays the biggest role in convective drying acceleration.

Keywords: *osmotic dehydration; convective drying; ultrasound; process kinetics.*

1. Introduction

Drying is one of the food preservation methods. The convective drying is the most commonly used technique. Unfortunately the method is slow and energy-consuming. Therefore, various methods to shorten the duration of the process and improve its energetical efficiency are investigated. One of the possibilities is the use of osmotic dehydratation as drying pretreatment. During the last two decades a great interest is directed to ultrasonic assistance of both osmotic dehydratation [1] and connective drying [1,2]. Also in the Department of Process Engineering, Poznan University of Technology, research in this area has been conducted for several years. The aim of this paper is to present a review of these research.

2. Materials and methods

2.1. Tested materials

A wide collection of various materials has been tested in the work of our team. Most of them are fruits and vegetables: apples [3-11], cherries [12], strawberries [9,13], raspberries [5,14], apricots [16] , carrots [9,17-21], green pepper [22], red pepper [11], potatoes [23], beetroot [24], onion [10]. In addition, kaolin was also tested [11,20].

2.2. Osmotic dehydratation – method

The simple osmotic dehydration and ultrasound assisted dehydratation (frequency 25 kHz) were tested to get the effect of ultrasound on the process kinetics and product quality. Aqueous solutions of glucose [12], fructose [6,17,19] and d-sorbitol [6] as working fluids were used. Kinetics and effectiveness of osmotic dehydratation were assessed on the basis of solid gain (*SG*), water loss (*WL*) and osmotic dehydratation rate (*ODR*):

$$\mathrm{SG} = \frac{m_{st} - m_{si}}{m_i}, \quad WL = \frac{m_i - m_t}{m_i} + SG, \quad ODR = \frac{dm_t}{dt} \tag{1}$$

where m_{st}, m_{si} are masses of solid matter of osmotically dehydrated and fresh sample, respectively and m_i, m_t are the initial and actual mass of sample, respectively.

2.3. Drying – methods

Several different dryers were used in the research. The laboratory chamber dryer was used for simple convective drying after osmotic dehydration [12,17]. Hybrid drying (convective – microwave – radiative) after osmotic dehydratation was performed in prototype laboratory hybrid dryer [19]. All ultrasound assisted drying processes were carried out in one of two hybrid (convective – microwave – ultrasonic) laboratory dryers: cabinet dryer [3-5,7,8,10,11,13-16,18,20,22-24] and rotary dryer [6,9,21,24]. The influence of ultrasound assistance on convective drying [3-11,13-16,20-24] and on convective - microwave hybrid drying [11,13-16,18,19,21,22,24] was investigated. Continuous drying processes were studied in all papers, while in some works the intermitten drying was examined [2,11,12,15-17,20,24]. The samples mass and their temperature were measured continuously during

experiment. The moisture ratio $MR = (m_t - m_{eq})/(m_0 - m_{eq})$ as a function of drying time represents drying kinetics, where m_t, m_0 and m_{eq} are the instantaneous (for a given time of the process), initial and equilibrium sample mass, respectively.

Energy consumption was measured for the whole drying system with the electricity meter.

2.4. Quality assesment – methods

It is important to ensure the quality of the dried product. The basic parameters describing quality were: color change during drying (important from the consumer point of view) and water activity (responsible for durability of food).

The preservation of nutrients during the drying process is extremely important from the point of view of the food's value of the product. Retention of several nutrients were measured.

The dried product ability to irrigation was measured in rehydratation tests. Texture of dried apple was determined by compression tests with the acoustic emission measurement. Change of plant tissue microstructure (onion and apple) was observed.

3. Experimental results

3.1. Osmotic dehydratation

Osmotic dehydratation could be treated as predrying process. During simple osmotic dehydratation water loss (*WL*) ranged between 18% [12] to 51% [17] and solid gain (*SG*) ranged between 8% [17] to 12% [6,17]. The use of ultrasound to intensify the process caused *WL* increase by 14% [17] to 44% [19] and *SG* increase by 15% [17] to 45% [6]. It was also shown that osmotic dehydratation rate (*ODR*) increased [19].

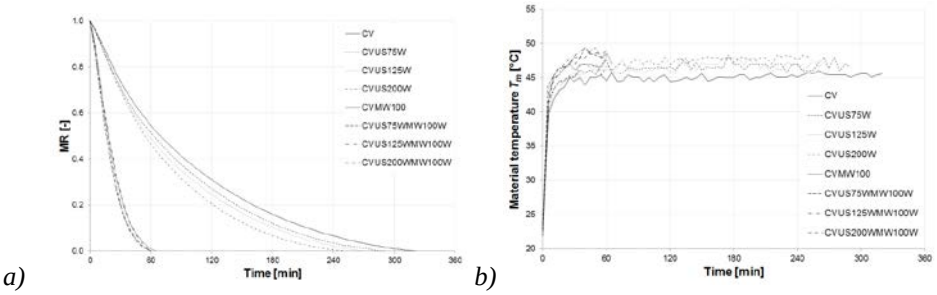
After osmotic dehydratation, the samples were dried. Then the water activity and the color change of the samples were examined. The results of these tests indicate that the effect of ultrasound application on final water activity is negligible. The color change results are ambiguous. In works [12,17] a reduction in color change was obtained due to the use of ultrasound. On the other hand, the works [6,19] indicate an increase of this parameter

3.2. Drying

3.2.1. Drying kinetics and energetic effectiveness

Drying kinetics is described by drying curves (moisture ratio *MR* versus time) and temperature evolution. Figure 1 shows the results of convective drying (CV), convective-microwave drying (CVMV) and both methods ultrasound assisted (CVUS and CVUSMV, respectively). After the shortcut specifying the method (MV and US) the used power in watts was written. The use of ultrasounds during drying results in the process acceleration and in a slight increase in the temperature of the dried material. A clear acceleration in convective drying was obtained, while convective-microwave drying was only slightly accelerated.

Ultrasonic assistance of convective drying shortened the drying process from 11% [5] to 60% [15]. This shortening of drying time resulted in a reduction of total energy consumption from 9% [3] to 21% [14]. A slight acceleration of the convective-microwave drying process was associated with an increase in energy consumption.



a) *b)*

Fig. 1. Drying kinetics of carrots: a) drying curves; b) temperature of dried material [18]

3.2.2. Products quality

The use of ultrasound in each case resulted in a reduction in the color change. This reduction ranged between 12% [21,23] and 40% [13,18].

All studies indicate no impact of ultrasound on water activity. This parameter depends primarily on the final moisture content in the material, regardless of the drying method used.

Rehydratation ratio is defined as ratio of sample mass after rehydratation to initial sample mass (fresh material). Ultrasound application improved rehydratatin ratio from 14% [23] to 21% [16, 22]. This means that the use of ultrasound improves the internal structure.

The retention of vitamin C was improved due to ultrasound assistance (from 44% to 69% in green pepper [22] and from 68% to 70% in red pepper [11]). The obtained results regarding retention of phenolic compounds in carrot [18] are inconclusive. The use of ultrasound of low power caused a reduction in the phenolic compounds content while ultrasound of high power caused an increase in the content. The retention of carotenoids was improved due to ultrasound assistance (from 73,5% to 90% in carrot [18] and from 67% to 76% in red pepper [11]). The retention of betanin in red beetroot was improved from 27% to 33% [24]. The retention of anthocyanins in raspberries [11,15] was improved from 56% to 76%. Antioxidant activity of carrot [18] deteriorated due to the use of ultrasound. The activity decrease ranged between 13% to 33% (depending on US power) compared to simple convective drying.

Ultrasound assistance influenced changes in material texture [5]. Generally streght and Young modulus of material increased due to ultrasound application. Ultrasound dried crisps were more brittle although less crispy that convective dried ones.

The effect of ultrasound on the material structure was examined using an optical microscope [4, 10]. The application of ultrasound caused the increase of pore dimentions, the creation of microchannels and disruption of the tissue. These results were confirmed by SEM photographs of convective and ultrasound-convective dried apple [10].

4. Modeling of US assisted drying

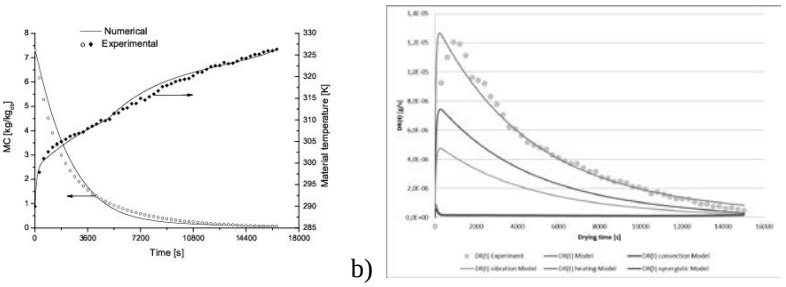
4.1. Lumped capacities model

The lumped capacities model was proposed [3,25] to describe drying kinetics. The final system of coupled ordinary differential equations is as follows:

$$m_s \frac{dX}{dt} = -A_m h_m \ln \frac{\varphi|_{\partial B} p_{vs}(T_m)}{\varphi_a p_{vs}(T_a)} \tag{2}$$

$$m_s \frac{d}{dt}[(c_s + c_l X)T_m] = A_T h_T (T_a - T_m) - A_m l h_m \ln \frac{\varphi|_{\partial B} p_{vs}(T_m)}{\varphi_a p_{vs}(T_a)} + \Delta Q \tag{3}$$

where: A_m, A_T denote surfaces of mass and heat exchange, respectively; h_m, h_T – coefficients of mass and heat exchange, respectively; $\varphi_a, \varphi_{\partial B}$ – relative drying air humidity far and near dry material surface, respectively; p_{vs} – saturated vapor partial pressure (temperature dependent); c_s, c_l – specific heat of a solid and liquid, respectively; l – latent heat of evaporation; ΔQ – volumetric heat source describing ultrasound absorption.



a) b)

Fig. 2. Drying kinetics of carrots - experimental and numerical results: a) drying curves and temperature of dried material [12]; experimental and model drying rate and "vibration effect", "heating effect" and "synergistic effect" as a function of time

The model was applied to describe ultrasound assisted convective drying in [3,9,13,14,20,25]. The model shows very good compatibility with experimental data (Fig. 2a). The results of the process modeling make it possible to analyze the efficiency of ultrasound application. Additional parameters should be specified for this purpose. Drying rate DR expresses the rate of moisture decrease in the material during drying as a function of time and is determined as

$$\mathrm{DR}(t) = -m_s \frac{dX}{dt} = A_m h_m ln \frac{\varphi|_{\partial B} p_{vs}(T_m)}{\varphi_a p_{vs}(T_a)} \tag{4}$$

The ultrasound assistance improves the drying rate. This is due to three mechanisms associated with the use of ultrasound, namely "vibration effect", "heating effect" and "synergistic effect" [13,22]. The model separates these effects and allows to determine their share in the drying rate (Fig. 2b). The results indicate that the vibrating effect has the largest share in the acceleration of drying. The impact of the other two effects is much smaller.

4.2. Packed bed drying models and continuous model

Three models of ultrasound assisted bed drying were proposed [26-28]. All of these models assume lumped capacities of grains and continuous description of the whole bed. Models by Kowalski [26] and Kowalski, Rybicki [27] treat material as not shrinking. Model by Musielak [28] takes into account high shrinkage of grains. All these models describe drying kinetics as well as distributions of moisture content and temperature in the bed.

The continuous model, describing mass and heat transfer in a single body during ultrasound assisted drying is proposed in [7]. The model was developed basing on irreversible thermodynamics. The model allows to calculate and describe the drying kinetics, the distributions of moisture content and temperature in dried body and the shrinkage of the body.

5. Conclusions

The paper is a review of research, carried out in Department of Process Engineering, Poznan University of Technology, on the use of ultrasound to intensify osmotic dehydration and drying. In general, it can be concluded that the use of ultrasound significantly accelerates osmosis and convective drying. Thanks to this, energy efficiency of the processes increases. Ultrasonic assistance of microwave drying causes slight acceleration, therefore it is energy inefficient.

In most cases, the use of ultrasound has improved product quality. This is due to the shortening of the drying time and changes in the structure of the dried material.

Mathematical modeling allowed to describe the kinetics of the process. Thanks to this, the magnitude of the impact of individual phenomena on the intensification of drying could be determined.

6. Acknowledgements

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Using Life Cycle Assessment methodology to minimize the environmental impact of dryers

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Abstract
Drying is known as a high energy consuming unit operation, representing between 12 to 25% of the global industrial energy consumption in developed countries. Consequently, drying contributes to several environmental impacts mainly associated to its heat or electricity requirements. One can cite global warming, emission of particles, acidification, photochemical ozone formation, …

Based on a literature review and some dedicated case studies, this work will illustrate how Life Cycle Assessment (LCA) can be used to evaluate the environmental impacts associated to a drying operation. The results will be presended in a way to indicate some eco-design strategies for dryers.

Keywords: *drying; eco-design; Life cycle assessment; environmental impact.*

1. Introduction

Drying is known as a high energy consuming unit operation, representing between 12 to 25% of the global industrial energy consumption in developed countries. Consequently, drying contributes several environmental impacts associated to heat or electricity production. One can think, among others, to global warming via greenhouse gas emissions, acidification or photochemical ozone formation via nitrogen oxides emissions, human toxicity via particule matter emissions, … Besides these impacts directly linked to energy consumption, industrial drying operations may also induce other environmental impacts, depending on the choice and the quantity of the materials it is made of, for example.

Life Cycle Assessment (LCA) is now considered as the most complete methodology to evaluate the potential environmental impacts associated with a process, product or service, following a cradle to grave approach. In addition to International Standards ISO 14040 [1] and 14044 [2], the European Joint Research Center developed guidance rules published in the International Reference Life Cycle Data System (ILCD) Handbook [3]. As mentioned in one guest editorial of A. Mumudar [4], LCA of competeting systems has to carried out before selecting the optimal one.

Based on a literature review and some dedicated case studies, this work will illustrate how this methodology can be used to assess the environmental impacts associated to a drying operation. The results will focus on the main process parameters influencing the environmental impacts in a way to indicate some eco-design strategies for dryers.

2. Materials and Methods

This section will summarize the principles of the LCA methodology allowing to understand the results that will be extracted from the literature and from our case studies.

Following ISO standards, LCA studies include 4 phases. The first step consists in defining the “goal and scope”, namely determining the functional unit, to which all the results will be associated, the system boundaries, cut-off rules, time period, impact categories, etc. A typical functional unit could be ‘the drying of one ton of product’ with some specifications on final quality of the product (dryness, …). The system boundaries specifies the different so-called ‘unit processes’ included in the scope, for example, the supply chain, the feed preparation, the packaging, the maintenance, treatment of exhaust gases, …

The second step is called the Life Cycle Inventory (LCI). This phase involves data collection and modeling of the product system, as well as description and verification of data. The data must be related to the functional unit previously defined. Besides specific data, several databases can be used, as well as the scientific literature. The LCI provides information about inputs and outputs in form of elementary flows from and to environment for all the unit processes included in the system boundaries. In the context of drying, a part of the data can be obtained via process control sofwares or energy audit systems allowing the report of any consumption or emissions.

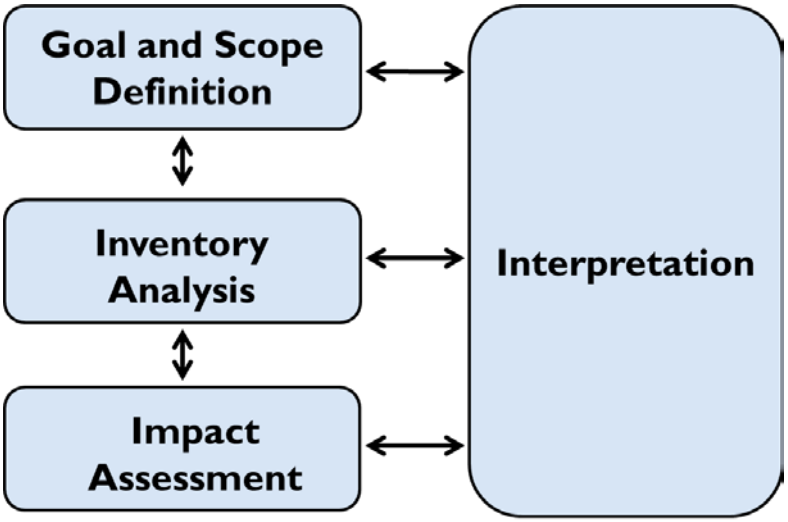

Fig. 1 The four steps of a Life Cycle Analysis.

The third step aims to convert the LCI results into environmental impacts, using several recommended methodologies (following the ISO Standards and the ILCD handbook). Depending on the methodology (ReCiPe, ILCD, Impact World, etc.), the contribution of the functional unit to impact categories such as global warming, eutrophication, acidification, inorganic respiratory effects, tropospheric ozone formation, etc. can be assessed. Fig. 2 illustrate 15 midpoint (problem oriented) impact categories and 3 areas of protection at endpoint. Characterization factors are used to calculate the contribution of each elementary flow of substances to the impacts they are known to be related.

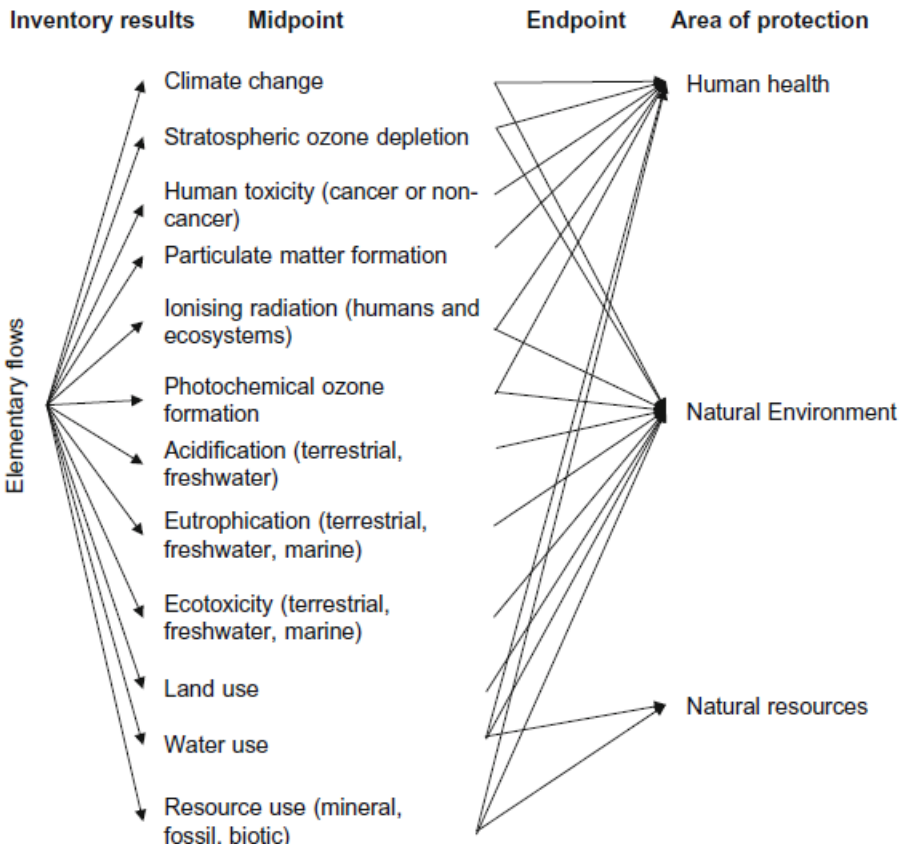

Fig. 2 Impact assessment according to the ILCD handbook.

The last step of a LCA is the interpretation of the results, with three significant steps: the identification of the significant issues, the evaluation of the quality of the study and the drawing of the conclusions, recommendations and reporting.

3. Literature review

Even through an increasing number of papers mention the importance of using an holistic approach to design drying in a sustanaible way, the number of studies really using LCA as an eco-design tool is still low.

Ciesielski and Zbicinski [5] compared two spray dryers, one at laboratory scale and one at industrial scale using LCA. They found that both units generated the greatest environmental load at the usage stage of the life cycle and have an effect mainly in the damage category of resources depletion.

De Marco et al. [6] studied the industrial production of apple powders. Both the drum drying and the storage are the steps that have high impacts (more than 35% each one) on

global warming potential and aquatic eutrophication; for the other midpoint categories, the main contribution (> 67 %) is due to drum drying.

Romdhana et al. [7] developed a general eco-design model of biomass drying. Their idea was to develop an assessment computer-aided process engineering tool that compares environmental impacts of different operating conditions and fuel types to support decision-makers for an improved compliance to environmental criterion. However their optimization only includes carbon footprint, which is not representative of the overall process performance.

Prosapio et al.[8] used LCA to optimize the production of strawberries by freeze-drying. They found that that agricultural steps, packaging and end of life only marginally influenced emissions, whereas processing steps are the main contributors. Their analysis revealed that the process was sensitive to vacuum drying time and rather insensitive to freezing time; They proposed an improved solution using osmotic pre-treatment allowing reduced process times and a decrease of 25% of emissions.

Van Oirschot et al.[9] used LCA to evaluate the system design of seaweed cultivation and drying. They found that the drying step (using light fuel oil in a industrial furnace) had the highest contribution on the environment.

4. Case study

In order to illustrate some information that can be used as decision support tool when designing dryers, a simplified LCA of a sludge dryer has been carried out, varying some parameters. The aim of the study is to compare the environmental impact associated to the evaporation of 1 ton of water, i.e. the functional unit, following the scenarios indicated in Table 1. Scenario 1 defines the base case.

Table 1. Modeling scenarios

Scenario	Thermal energy consumption kWh	Thermal energy production	Electricity consumption kWh	Electricity production
1	700	Gas boiler (EU-28)	80	EU-28 grid mix
2	700	Gas boiler (DE)	80	DE grid mix
3	700	Light Fuel Oil boiler (EU-28)	80	EU-28 grid mix
4	700	Biomass boiler (EU-28)	80	EU-28 grid mix
5	770	Gas boiler (EU-28)	80	EU-28 grid mix
6	700	Gas boiler (EU-28)	80	Wind power (EU-28)

The value of thermal and electrical energy consumptions correspond to the claimed performances of Innodry® 2E (Suez-Degrémont). Depending on the scenario, the thermal

energy is produced via gas, light fuel oil or biomass boiler, which technical characteristics corresponding to Germany (DE) or to the average situation found in EU-28. The electricity is taken from the grid, i.e. the German or average European one. All these scenario also include the transportation of the wet sludge (20% DS) on 100 km using a EURO 6 truck-trailer, up to 28 t gross weight. The dryer infrastructure in itsef has been neglected. GaBi 7 software and associated datasets have been used to carry out the LCA, with ReCiPe 2016 v1.1 Midpoint (H) as impact assessment method.

Fig. 3 shows the results at the characterization stage: for each of the selected impact category, the highest score is put at a value of 100% and other scores are represented using a relative scale. The absolute values are given in Table 2. In order to facilitate the interpretation of the results, only the most relevant impacts are given.

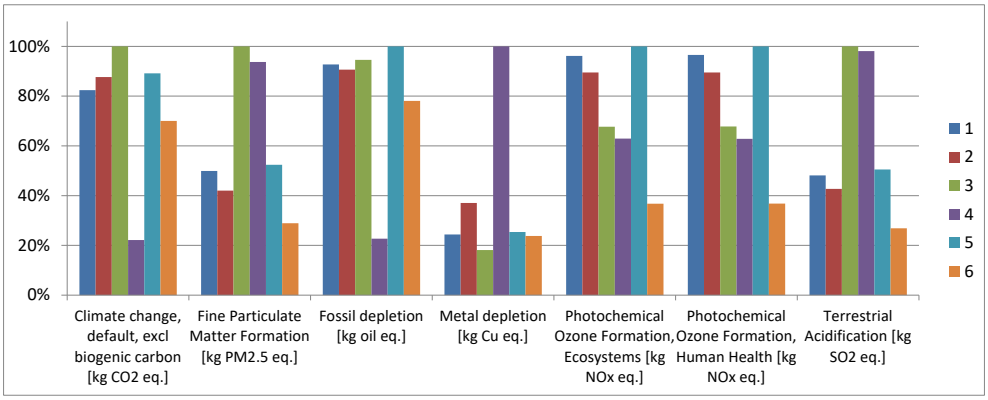

Fig. 3 Impact of the evaporation of 1 ton of water: characterization results.

Table 2. Characterization results

	1	2	3	4	5	6
Climate change, default, excl biogenic carbon [kg CO_2 eq.]	220	234	267	59	238	187
Fine Particulate Matter Formation [kg $PM_{2.5}$ eq.]	0,050	0,042	0,100	0,094	0,052	0,029
Fossil depletion [kg oil eq.]	86,6	84,6	88,3	21,2	93,4	72,9
Metal depletion [kg Cu eq.]	0,089	0,135	0,066	0,364	0,092	0,087
Photochemical Ozone Formation, Ecosystems [kg NOx eq.]	162	151	114	106	168	62
Photochemical Ozone Formation, Human Health [kg NOx eq.]	101	94	71	66	105	39
Terrestrial Acidification [kg SO_2 eq.]	0,153	0,136	0,318	0,312	0,161	0,085

The results show clearly the influence of the choice of the energy source, either thermal or electrical, on the environmental impact. The use of a biomass boiler (4) allows to reduce the climate change by 80% in comparison with light fuel oil (3). This biomass scenario also

gives the lowest impact regarding fossil depletion but the highest one for metal depletion. With respect to the base case, an increase of 10% of the thermal energy consumption (7) induces a similar relative increase in all categories. Bigger changes are obtained when replacing the electricity of the grid by wind power (6), especially for photochemical ozone formation and fine particle matter formation. The comparison between scenarios 1 and 2 also shows the impact of the localization of the drying plant on its environmental footprint.

As a sensitivity study, the transportation distance, initially fixed at 100 km, was set to 50 and 150 km, using energy consumptions of scenario 1. Fig. 4 shows that an increase of the transport distance influences almost all impact categories, except the ones related with photochemical ozone formation. Nevertheless, Table 3 indicates, for example, that an increase of 100 km leads to an increase of 15 kg CO_2 eq. This illustrates that the transportation step is not prevailing in this case study (less than 10%), in regards with a total climate change indicator of 220 for the base case. A more detailed analysis shows, in this case, that the most impacting step is the production of thermal energy, contributing to 78% of the climate change score.

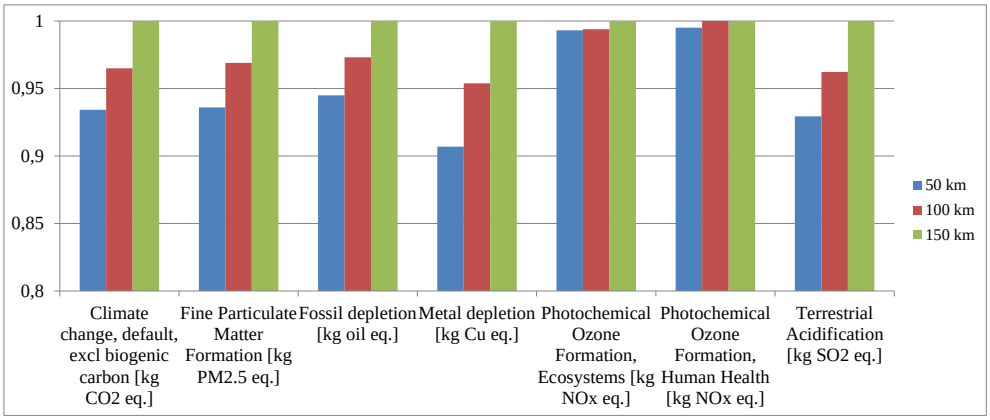

Fig. 4 Influence of the transportation distance on the impact associated to the evaporation of 1 ton of water: characterization results.

Table 3. Influence of transport distance - Characterization results

	50 km	100 km	150 km
Climate change, default, excl biogenic carbon [kg CO_2 eq.]	213	220	228
Fine Particulate Matter Formation [kg $PM_{2.5}$ eq.]	0,0482	0,0499	0,0515
Fossil depletion [kg oil eq.]	84,1	86,6	89
Metal depletion [kg Cu eq.]	0,084	0,089	0,093
Photochemical Ozone Formation, Ecosystems [kg NOx eq.]	162	162	163
Photochemical Ozone Formation, Human Health [kg NOx eq.]	100,4	101	101
Terrestrial Acidification [kg SO_2 eq.]	0,147	0,153	0,159

5. Conclusions

As already mentioned in 2011 by Haque [10] in his guest editorial, hopefully LCA will soon become one of the key tools that drying practitioners and R&D personnel will utilize on a regular basis. This very simple case study illustrates that LCA can be used to evaluate the influence on energy production source on the environmental impact. Besides logistical aspects, LCA could also includes the infrastructure (building material options, …), namely in the case where several configurations or technologies could be used. This tool could allow to predict whether energy intensification strategies are really worth and do not lead to impact shifting.

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Beyond freeze-drying of biologics: vacuum-foam drying and spray freeze-drying

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Abstract

The complexity of biotherapeutics in development continues to increase as our capability in discovery and recombinant technology improves. While safety and efficacy remain the two critical aspects of all therapeutics, ensuring adequate stability is a challenge. Freeze-drying is a commonly-used processing technique to enhance the stability of biotherapeutic products, although the lengthy process time and low energy efficiency have led to the search for, and evaluation of, next-generation drying technologies, including spray freeze-drying and vaccum-foam drying. Both processes result in dosage forms that vary considerably from those produced by lyophilization and possess physical properties that may be deemed superior for their intended applications.

Keywords: *vacuum-foam drying; spray freeze-drying; lyophilization; biotherapeutics; stabilization*

1. Introduction

Most biological materials contain high water content (typically ≥ 80% w/w). Removal of water through drying provides numerous benefits, including ease of handling and storage, reduction in transportation costs, and improved stability, to name a few. Though all drying techniques share a common objective (i.e., dehydration), conceptually they are different and require modification/adaptation based on the properties of the compound.

Numerous commercially-approved products are manufactured by freeze-drying,[1] thus lyophilization represents the gold standard to which novel drying methods must be compared. Despite its prevalent use, novel technologies are continuously being evaluated, including vacuum foam-drying and spray freeze-drying, as will be described herein. Furthermore, there are a great number of drying technologies that are available, if not already in use, in the food, agriculuture, and textile industries.[2] As the sensitivity of pharmaceuticals is unique to the given compound, the selected drying technique may not be universally applicable. By understanding the drying mechanism and the stresses involved, the drying techniques can be tailored for effective use.

2. Materials and Methods

2.1. Vacuum-Foam Drying

2.1.1. T cell sample preparation

Primary human pan-T cells (STEMCELL Technologies Inc, Cambridge, MA) were expanded 3-4 fold and cryopreserved at -150°C. During formulation, frozen T cells were thawed at ambient temperature, centrifuged, supernatant removed, and resuspended in the appropriate formulation matrix.

2.1.2. Cell viability and count

Viability measurements were obtained using a NucleoCounter® 3000 (ChemoMetec A/S, Allerød, DK). Neat samples were diluted 4:1 in PBS and cell viability and count assay was performed using a Vai1-Cassette™. The procedure used membrane penetrating acridine orange (AO) and non-penetrating 4′,6-diamidino-2-phenylindole (DAPI) fluorescent dyes to assess cellular membrane integrity. Dried samples were allowed to recover for 3 hours following reconstitution and diluted in PBS prior to analysis.

2.1.3. Vacuum-Foam Drying and Freeze-Drying

Vacuum-foam drying and freeze-drying were performed using LyoStar lyophilizers (SP Scientific, Warminster, PA). Vacuum-foam drying cycles utilized pressures and shelf

temperatures ranging from 0.05 to 5 Torr and 5 - 30°C, respectively. The freeze-drying cycle utilized -30°C shelf temperature for primary drying at a pressure of 0.05 Torr.

2.2. Spray Freeze-Drying

Two process equipment types were utilized for spray freeze-drying. The first process step, spray freezing, was conducted at ambient pressure in a spray freezing chamber and the subsequent dynamic freeze drying of the frozen bulk was perfomed in a rotary bulk freeze dryer.

2.2.1. Spray Freezing

Spray Freezing in all scales was performed in a spray freezing chamber unit (SprayCon, Meridion, Germany) with frequency driven droplet formation nozzle(s) placed in the top lid. The droplet formation is achieved by controlled laminar jet break up. The cylindrical process chamber is double walled and cooled with gaseous and liquid nitrogen.

For all trials, the spray liquid is a 20% (w/w) sucrose solution, and a 300 µm orifice opening for the nozzle was used. For the freezing step, the main process parameters are: (i) for lab and pilot scale trials: - 150 °C gas temperature; spray rate: 19 g/min (1 nozzle); droplet size 550 ± 10 µm; (ii) for commercial scale trial: - 120 °C gas temperature; spray rate: 26 g/min / nozzle (3 nozzles), droplet size 550 ± 10µm

For lab and pilot scale trials, the process equipment used was a stand-alone equipment with intermediate frozen storage of material at -60°C. For commercial scale, the trial was conducted in a fully contained process line that integrates both the spray freezing equipment and the rotary freeze dryer; e.g., the freezing chamber continously discharges the frozen spheres into the precooled drum of the rotary freeze dryer.

2.2.2. Dynamic Freeze-Drying

Lyophilization of the frozen sucrose pellets was performed in three different scales of rotary freeze dryer (RFD) equipment (all by Meridion, Germany); RFD LyoMotion LAB (lab scale), LyoMotion 30 (pilot scale), and LyoMotion 200 (commercial scale). All scales used a rotating, double walled drum which was positionesd in a vacuum process chamber, to which a condenser was attached. The drum temperature was controlled in a range from – 55 °C up to +50 °C.

In all trials, sublimation energy was conveyed by contact heat, via the double wall of the rotating drum, and by infrared radiation emitted from one or more of the infrared sources that were positioned inside the drum above the moving bulk product surface. The pressure within the drying drum was maintained between 50 and 100 µbar at all three scales.

3. Results and Discussion

3.1. Vacuum-Foam Drying

Vacuum-foam drying (VFD) transforms a solution or suspension into a dried static foam through a vacuum-induced evaporation and boiling process. VFD enables removal of water at low temperatures, which is required for heat labile biotherapeutics, through the use of a strong vacuum (e.g., 1 -10 Torr). For pharmaceutical applications, VFD can be performed using a lyophilizer capable of pressure control at a higher range than a typical freeze-drying (FD) cycle. In VFD, the boiling process results in a final product that has an expanded foam structure. Fig. 1 demonstrates that increasing sucrose concentration from 15 to 40% (w/v), while maintaining a 1 mL fill, correlated with increasing foam volumes in the final product. The reproducibility and heterogeneity of a VFD product appearance is a challenge that should be considered during formulation and process development.

Fig 1 Vacuum-foam dried (VFD) preparations of 15%, 30%, and 40% sucrose (left to right).

Compared to other drying techniques, dry static foams have been reported to provide significant stabilization to biotherapeutics. For example, Abdul-Fattah and coauthors [3, 4] have demonstrated improved storage stability of a monoclonal antibody and live virus vaccine as a dried foam in comparison to those prepared by spray drying and freeze drying. Currently, the use of cryopreservation techniques are required to stabilize cell-based therapeutics since the health of cellular suspensions decreases over a short period of time. The ability to stabilize mammalian cells in the dried state may reduce the logistical challenges of a supply chain for therapeutics requiring extremely low storage temperatures.

The T cells used in this work are primary human pan-T cells that were stored at -150°C prior to preparation. T cell formulations evaluated include CryoStor® freeze media (with 0, 5 or 10% DMSO) and disaccharide-based formulations (20% sucrose/trehalose in PBS at pH 7.4) at 1E6 cells/mL with 1 mg/mL bovine serum albumin. CyroStor freeze media is a commercially available preservation medium utilized for cryopreservation of cells at multiple concentrations of DMSO. Disaccharide-based formulations have been reported to provide significant stabilization to mammalian cells though the drying process and in the dried state

[5, 6]. Viability of pre- and post-dried preparations were measured using a NucleoCounter® 3000 fluorescent cell counting and membrane integrity assay.

Prior to drying, viability of all formulations was measured and no significant difference was observed. The average viability of the liquid controls was 91 ± 3% (Fig. 2). All formulations were vacuum-foam dried using a fixed shelf temperature of 30 °C and the pressure was incrementally decreased from 5 to 0.05 Torr with a total drying time of 90 minutes. Drying was completed prior to an extended secondary drying step to minimize dehydration stress which could lead to additional viability loss. Fig. 2 presents the membrane integrity of T cell formulations following vacuum-foam drying and reconstitution with water. The viability of T cells after drying in CryoStor media was 65%, with the addition of 5 and 10% DMSO resulting in post-drying viability of 57 and 32%, respectively. The 20% sucrose and trehalose formulations resulted in post-drying viability of 63 and 65%, respectively. While greater than 60% T cell viability was retained after drying various DMSO-free formulations, the recovery could be improved further through optimization of formulation, drying process parameters, and residual water content.[2]

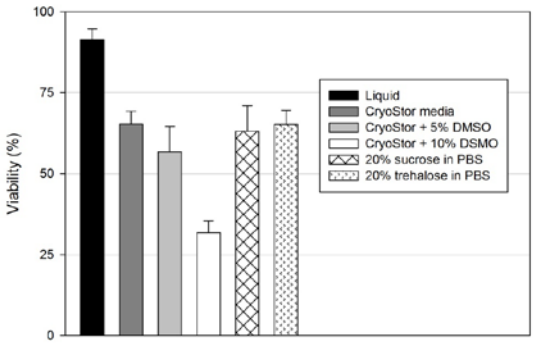

Fig. 2 Viability of human pan-T cell formulations after vacuum-foam drying compared to liquid controls. Data presented as average ± SD.

A follow-up evaluation compared the storage stability of 7E5 T cells/mL formulated in 30% trehalose and 3% BSA in PBS at pH 7.4 as liquid, freeze-dried and foam dried preparations at 5°C (Fig. 3). The freeze and vacuum-foam drying cycles were designed to target a residual water content of 9% (w.b.). There was a higher post-drying viability for the VFD preparation compared to FD, for which the process loss was 61 and 42% after FD and VFD, respectively. In order to decouple processing stress and storage stability, the viability results were normalized based on initial stability samples (post-drying). As shown in Fig. 3, the VFD preparation exhibited superior stability compared to liquid and FD preparations. At the same residual water content as a FD cake, these data provide evidence that T cells vitrified as a dried foam provides improved storage stability.

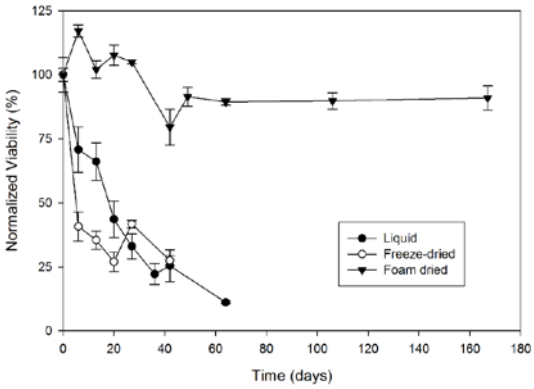

Fig. 3 Normalized viability of a liquid, freeze-dried, and foam dried preparations of T cells on stability at 5°C. Data presented as average ± 83% CI.

3.1. Spray Freeze-Drying

Spray freezing is often described as a process whereby the liquid droplets generated by spray nozzles (e.g., binary air nozzles or pressure nozzles) are directed into a cryogenic medium, often liquid nitrogen.[7] In such a case, the suspended frozen droplets are collected either by sieves or are collected after the cryogen has boiled off. For the spray freeze-drying technology presented herein, two unique processes have been developed and adapted for the use in the manufacture of parenteral pharmaceutical formulations.[8-10]

In the first process step, spray freezing, the frozen microspheres are generated as bulk by dispersing the substrate liquid into single droplets of homogeneous size,[11] utilizing a frequency nozzle, which fall through the cold gas (serving as cryogenic medium) and congeal to form frozen spheres. In contrast, the use of conventional spray nozzles results in a broader range of particle size with a significant amount of fines. The direct use of liquid nitrogen (LN_2) is avoided to minimize internal mechanical stress encountered during freezing, in particular for large particles. The freezing chamber is therefore designed as a cylindrical, double walled column. The process takes place under ambient pressure conditions. Droplet size depends on parameters such as flow rate, frequency, viscosity (based on formulation and temperature), and orifice diameter; for the current application, a (selectable) range for the droplets between 300 to 1000 µm was targeted. For scale-up, multiple nozzles were used, though the height of the freezing column remained unchanged.

The second process step is the freeze drying of the frozen bulk material. The dynamic lyophilization process conducted in a rotary freeze dryer provides process conditions that produce bulk product with high homogeneity (Fig. 4), while avoiding specific aspects of fluid bed processing.[12] Generally speaking, processing conditions such as pressure and temperature are quite comparable to parameters utilized in conventional freeze drying. There

are some differences to be noted, for example: the large surface of the frozen bulk increases heat and mass transfer, which generally allows for shorter drying times. Furthermore, the water vapor diffusion length is significantly reduced. A 10 mm lyo cake in a vial poses a maximum diffusion length of 10,000 µm; in a 1 mm microsphere, the maximum length is 500 µm. In conventional shelf freeze drying, the heat to a large portion is conveyed across the bottom of the glass vial; the drying front is moving from the top to the bottom, i.e. the heat transfer takes place across the frozen product. In dynamic freeze drying, the energy both from radiators and drum surface is transmitted to the surface of a particle, at which the drying front initiates.

Fig. 4 Spray Freeze-Dried microspheres generated for a 20% (w/w) sucrose solution.

The results for spray freeze-dried sucrose conducted at three scales are shown in Table 1. In all scales, residual water content less than 1% can be reached and that yield above 95% is possible in commercial scale. The lower yield in pilot scale is explained by the use of higher rpm of the drum in conjunctions with higher IR power. During drying, a pellet will loose 80-90 % of its weight. High water vapor flows would cause the pellets to get entrapped into the vapor flow, which would cause particles to leave the drum, reducing the yield. Additional factors need to be considered, such as solid content, as higher solid content reduces the loss in weight, and electrostatic phenomena, which may be significant if the particle size is too low. The level of residual water content achieved is comparable to that from conventional freeze drying.

Table 1. Results for 20% (w/w) sucrose solution processed at three scales

	Lab scale	**Pilot scale**	**Commercial scale**
Amount processed (kg)	1	6	107
Drying time (h)	5.5	16	29.25
Yield (%, w/w)	98.6	81.6	97.3
Residual water content (%)	≤ 1.0	≤ 1.0	≤ 0.6
Reconstitution time (min)	≤ 1.0	≤ 1.0	≤ 1.0

4. Conclusions

The complexity of biotherapeutics in development continues to increase as our capability in discovery and recombinant technology improves. While safety and efficacy remain the two critical aspects of all therapeutics, stability, both in terms of shelf-life and to stresses encountered during manufacturing, remains a challenge. Spray freeze-drying is a hybrid

processing technology comprising spray drying and bulk freeze-drying, while vacuum foam drying is a modified freeze-drying process that challenges the conventional processing conditions utilized in lyophilization. The former has matured to a level where the application of the technology even in commercial scale is in reach also for pharmaceutical applications, while the latter has provided enhanced stability to a complex biological beyond that provided by a conventional freeze-drying process. The development of *novel* drying technologies, such as the aforementioned processes, is a culmination of fundamental understanding gained in academia and leveraging the lessons learned through their utilization in orthogonal industries. For implementation, technical evaluation should include the scalability of the process, energy efficiency, as well as the capability to implement the technique in a GMP environment.

5. Acknowledgements

Karin Mayer for supporting the SFD lab and pilot work and Thomas Gebhard and Roland Kaiser for the commercial scale trial, all performed at Meridion, Germany.

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New technique of combined hot air and microwave drying to produce a new fiber ingredient from industrial by-products

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Abstract

The search for solutions to transform the by-products generated by the agri-food sector in high value-added ingredients is a priority. The aim of this research was to develop a microwave coupled with hot air drying technique allowing maximizing profits by reducing time and operational costs and to produce a dietary fiber ingredient with interesting technological properties for the development of healthy foods. The shrinkage-swelling phenomena occurred during drying changed the rehydration properties of the fibre ingredient obtained. An increase in particle size improved the fibre's swelling capacity when hydrated, allowing 50 % fat substitution in potato purees.

Keywords: *hot air-microwave drying; orange peel; thermodynamics; GAB model; dietary fibre*

1. Introduction

Sustainability is nowadays an investment for the future of any economic activity. The current situation of crisis has had an adverse impact in most industries, including the agri-food sector. However, this industry has been relatively the least affected when compared with other industrial sectors. This is mainly attributed to the fact that food products continue to be basic for consumers despite the economic downturn. Therefore, the agri-food sector is a key element in the European economy and can play a crucial role in the achievement of the objectives set in the EU's strategy for 2020: ensuring a sustainable framework of growth of a more competitive economy. The European agri-food industry has focused on energy efficiency and on reducing greenhouse gases emissions, along with better management of their resources as a way to improve its industrial competitiveness. In this sense, the search for solutions to transform the by-products generated in high value-added ingredients, is a priority. In this context, the juice industry, as fundamental sub-sector within the food sector, and large waste generator, must exploit the opportunity to transform their by-products into useful and profitable products for society. This transformation presents some difficulties which impede the profitability of the process. These difficulties are associated with the by-product, such as its compositional variability and its seasonality, and current techniques of transformation as the high energy cost in dehydration processes. This work represents an innovative and sustainable solution for overcoming the disadvantages associated with the high costs of stabilization, turning this by-products into high value-added ingredients, from both, nutritional and technological, points of view. The main aim was to develop a microwave coupled with hot air drying technique (HAD + MW) allowing maximizing profits by using the following strategy: reducing time and operational costs, producing a new ingredient rich in dietary fiber, with interesting technological properties for the development of healthy foods, studying the proposed comprehensive process and analyzing the new generated by-products.

The specific objectives were (i) to develop a thermodynamic model for understanding internal heating and water transport mechanisms occurring from the inside to the outside of orange peels during HAD + MW drying and to predict the chemical and structural transformations, (ii) to determine the sorption isotherms and the isosteric heat of sorption and to study its effect on the macrostructure and microstructure of the orange peel, (iii) to develop and to determine dielectric tools to predict the moisture and water activity by using dielectric spectroscopy and sorption isotherms, (iv) to compare the energy consumption of hot air drying (HAD) versus HAD + MW by analysing the physico-chemical and technological properties of the dietary fibre obtained and (v) to asses the technological and sensory properties of the new fiber obtained by using it as a fat replacer in potato pures.

2. Materials and Methods

Oranges (Citrus sinensis (L.) Osbeck var Washington Navel) were bought from a local supermarket in Valencia (Spain), and their peel was used for the experiments. Sixty orange peel cylinders (20 mm diameter and 3 mm thickness) were obtained using a core borer.

The size and shape of the samples were designed to resemble the small pieces of orange peel left after mechanical extraction of juice and the cuts made by a hammer crusher machine in the processing of orange peel. A diagram of the experimental procedure is shown in Fig. 1.

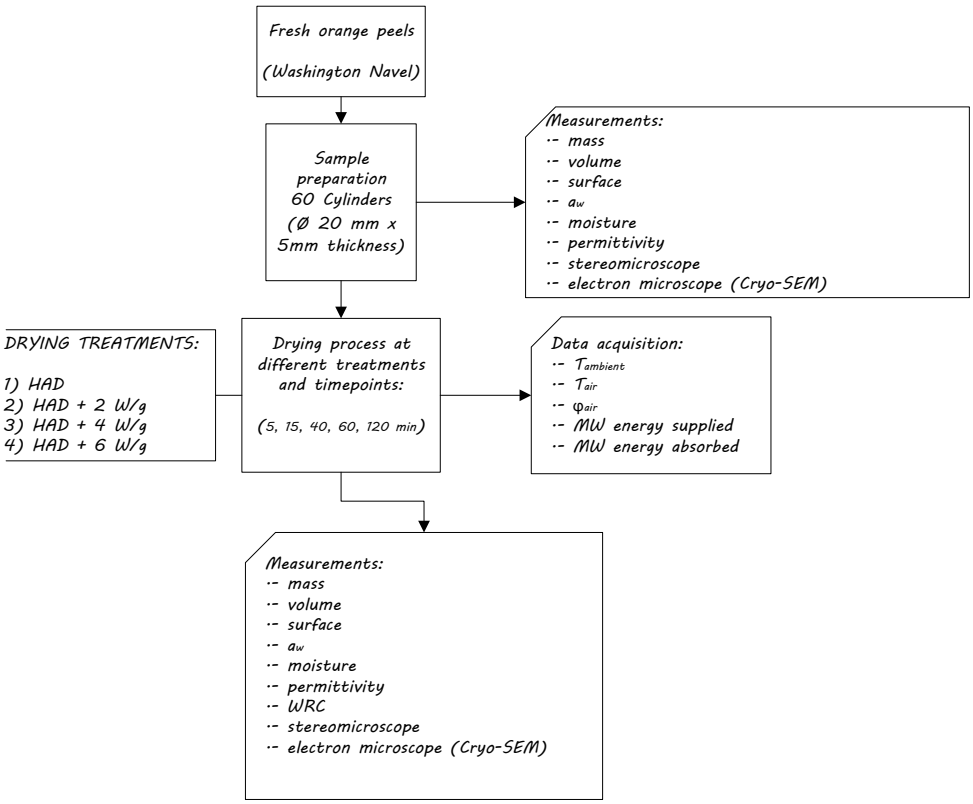

Fig. 1: Experimental procedure

Samples were subjected to HAD + MW, using a specially designed MW-air drying oven with a maximum output of 2000 W at 2450 MHz. For the experiments, the air velocity was 2.5 m/s, hot air temperature, 55 ºC, and the MW energy, 0, 2, 4 or 6 W/g. Four drying experiments were carried out (HAD, HAD + 2 W/g, HAD + 4 W/g and HAD + 6 W/g). Three orange peels samples (triplicate) were used for each drying time (5, 15, 40, 60 and

120 min) in each drying experiment. For fiber production, samples were milled after drying, using an ultracentrifuge mill (ZM 100, Retsch, Haan, Germany) with a sieve of 500 μm. At this stage, powder samples were sealed in plastic bags for further characterization.

2.1. Mass, volume, surface, a_w and moisture content

Samples were weighed using a precision balance Mettler Toledo AB304-S (precision: ± 0.001 g). Surface water activity was determined employing a dew point hygrometer Decagon Aqualab®, series 3 TE (precision: ± 0.003, dimensionless) (Decagon Devices Inc., Pullman, WA, USA). Measurements were performed using structured (not minced) samples; thus, the obtained a_w was considered the surface a_w. The water content of representative fresh orange peel sample and the samples dried for 120 min was determined. The samples were dried in a vacuum oven at 60 ºC until constant weight was reached (AOAC method 934.06 2000). The moisture content of the samples at the intermediate stages was calculated from the weight loss during drying. Volume was determined by image analysis (Sony T90, Carl Zeiss optics), using Adobe Photoshop© software, obtaining the diameter and thickness of the samples in triplicate.

2.2. Microstructure

The microstructure of fresh and dried samples was analysed using Cryo-SEM. A CryoACryostage CT-1500C unit (Oxford Instruments, Witney, UK), coupled to a Jeol JSM-5410 scanning electron microscope (Jeol, Tokyo, Japan), was employed.

The samples were also examined under a Leica MZ APO™ stereomicroscope (Leica Microsystems, Wetzlar, Germany) with a magnification of 8× to 80×.

2.3. Permittivity

The permittivity was measured with an Agilent 85070E open-ended coaxial probe connected to an Agilent E8362B Vector Network Analyser. The system was calibrated by using three different types of loads: air, short-circuit and 25ºC Milli®-Q water. All determinations were made from 500 MHz to 20 GHz.

2.4. Water retention capacity and sweeling capacity

For the determination of WRC, approximately 0.5 g of each sample (precision ± 0.0001 g) was hydrated in 20 mL of distilled water in a 50 mL (adapted from Robertson et al. 2000). Swelling capacity, defined as the ratio of the volume occupied when the sample is immersed in excess of water after equilibration to the sample weight, was measured by the method of Raghavendra, Rastogi, Raghavarao and Tharanathan [39]. To 0.2 g of dry sample placed in a graduated test tube; around 10 mL of water was added to hydrate the sample for 18 h; then the final volume attained by fiber was measured and expressed as volume/g of original sample (dry weight).

2.5. Rheology

The rheological characterization of the samples was carried out using a controlled-stress AR 2000 rheometer (TA Instruments, Leatherhead, United Kingdom). Stainless steel parallel plate geometry of 40 mm diameter was used with a gap of 2 mm.

2.6. Sensory analysis

The sensory analysis of the purées was carried out by a panel formed by 7 trained tasters applying a quantitative descriptive analysis (QDA) according to the UNE-ISO 6658: 2008 and UNE 87025: 1996 standards. Unstructured scales of 10 points were used to analyze 6 sensorial attributes: 2 visually (homogeneity and viscosity) and 4 on the palate (granularity, fat character, creaminess and viscosity). Each taster evaluated 6 samples of potato puree in triplicate: HAD, HAD + 2 W/g, HAD + 4 W/g, commercial fiber, no fiber and the reference puree.

2.7. Statitcal analysis

To determine the statistical significance of the results, an analysis of variance (ANOVA) was carried out with confidence levels of 95 % ($p \leq 0.05$) and 99 % ($p \leq 0.01$) using the Statgraphics Plus 5.1 programme.

For the sensory analysis the statistical analysis was carried out through the R-project program (R version 3.0.1.) applying a one-way ANOVA to determine the significance of the differences between samples for the parameters analyzed. In addition, a contrast test (Tukey test) has been applied to establish among which samples these differences exist.

3. Results and discussion

A thermodynamic model was developed to explain the mechanisms involved in mass and energy transports throughout the combined drying by hot air and microwave. A continuous shrinkage in HAD samples was produced by the internal liquid water losses, and the samples treated by HAD+MW showed an internal swelling caused by the internal evaporation produced by the microwave energy. Depending on the predominant mechanisms (HAD shrinkage and MW swelling) samples suffer volumetric expansions or contractions (Fig. 2).

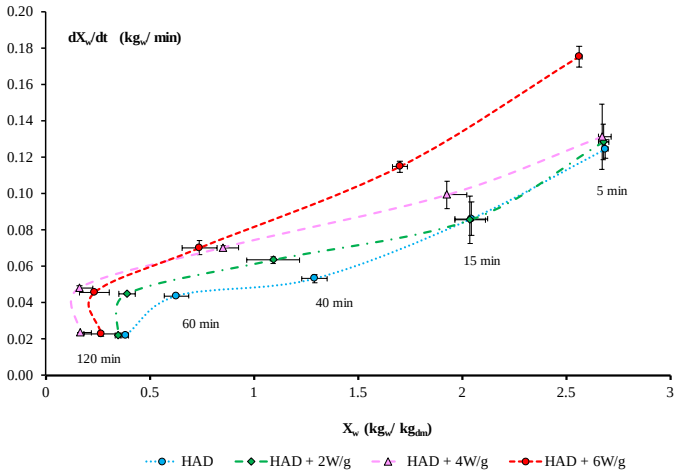

Fig. 2 Drying rate of orange peel samples dried by HAD and HAD + MW at different power intensities. Data represent means and standard deviation of experiments performed in triplicate.

The desorption isotherms of orange peel dried using different treatments (HAD + MW) were obtained and analysed. The results showed that the GAB model could be used to predict the moisture levels using the a_w measurements. The macrostructural and microstructural transformations were demonstrated and discussed in [1], taking into account the interactions of water with the tissue. The observed shrinkage/swelling phenomena clearly depended on the MW power and on the nature of the tissue.

It was possible to develop a dielectric isotherm technique (Fig. 3) by adapting the GAB model to predict the water activity in dried orange peel by using ε' (20 GHz). The physical meaning of the dielectric isotherm parameters (ε_0' and Cd) was studied and explained in [2]. The value of ε_0' at 20 GHz (γ-dispersion) represents the induction effect of the minimum quantity of adsorbed water or the monomolecular moisture layer. The parameter Cd is related with isosteric heat or the adsorption energy of the monomolecular moisture layer, as well as the C parameter of the GAB model. The application of MW power produced an increase of the isosteric heat or adsorption energy of the monomolecular layer, improving the surface tension of samples and thus the hygroscopicity, explaining the reduction of the ε_0' independently of the quantity of the water molecules adsorbed.

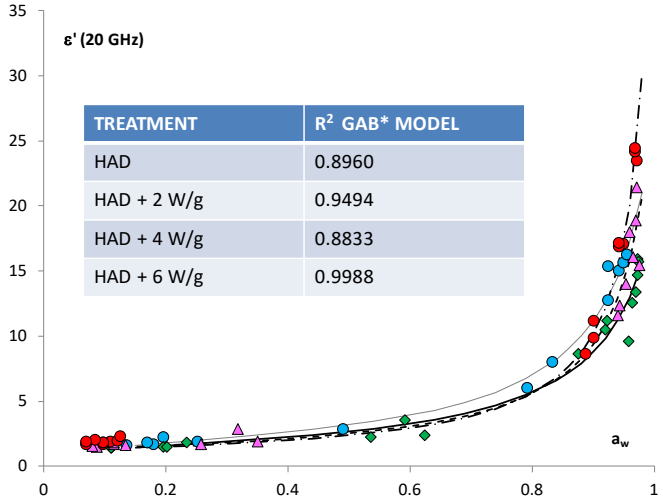

Fig. 3 Dielectric isotherm at 20 GHz of orange peel treated by different drying treatments. Color code same as Fig 2.

An important reduction in processing time (92 %) and energy consumption (77 %) was achieved compared to HAD. The drying treatment did not affect chemical composition or water retention capacities orange fibers. Total dietary fiber content was about 60 % with a ratio of soluble to insoluble fiber of 1:1. Although viscosity of both treatments showed similar values [3], the higher swelling capacity of HAD + MW treated fiber provoked a significant decrease in the viscoelasticity of the samples. An increase in particle size due to an increase in porosity during drying [4], improved fiber swelling capacity (Fig. 4).

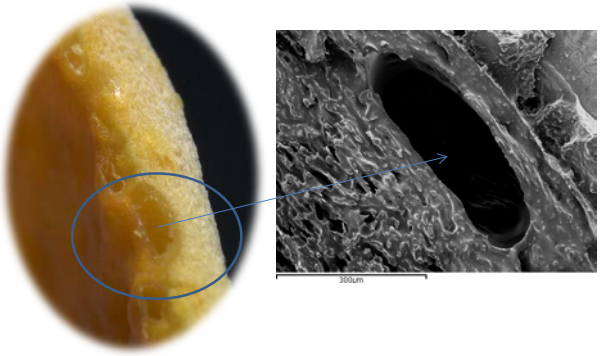

Fig. 4. Micrographs of orange peel samples dried by HAD + 2 W/g.

The fat content of processed potato purées with cream was reduced by 50% using different types of citrus fiber. All the fibers increased the visual viscosity and in mouth of the purées, as well as their behaviour viscoelastic The fibers obtained by air combined drying hot-microwave (4 W / g) presented a swelling capacity similar to commercial fiber. In addition, these fibers were perceived as more granular in the mouth which can be explained due to the greater volume increase when rehydrating.

4. Conclusions

This study has analyzed the microwave coupled with hot air process, developing tools that allow the adequate upscaling of the drying operation by adapting it to the best standards of quality of the final product. A monitoring system that ensures these standards has been designed. This model allows optimizing the traditional hot air drying, by coupling microwave, of orange peel waste as a novel process for citrus by-products valorization, reducing the process time and, therefore, process costs.

The quality and the energy consumption of the dietary fiber production process has been improved. The properties associated with its inclusion in food matrices have been optimized. Therefore, it can be concluded that combining the microwave treatment with hot air drying not only reduced the processing time but it also generated microstructural changes in the dried tissue that increase its water retention capacity. This improved the technological properties of this stabilised by-product, which will be of benefit during its further conversion into the dietary fibre.

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Influence of drying on *in vitro* gastric digestion of beetroot: evaluation of the microstructure

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Abstract

To better understand the influence of processing on the bioaccessibility of bioactive compounds during digestion, the microstructure of beetroot samples was observed prior to and after 180 min of in vitro digestion, by using scanning electron microscopy. Beetroot samples were subjected to convective drying at 60 ºC and 2 m/s and freeze-drying at -50 ºC and 30Pa. Dried beetroots were rehydrated prior to digestion by immersion in distilled water at 37 ºC during 90 min. To extract quantitative information related to cell size from the visual texture of beetroot, grey level granulometric methods from mathematical morphology were applied.

Keywords: *freeze drying; convective drying; scanning electron microscopy; image analysis; image texture analysis.*

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