

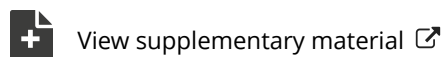


Effects of different drying techniques on the quality and bioactive compounds of plant-based products: a critical review on current trends

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Effects of different drying techniques on the quality and bioactive compounds of plant-based products: a critical review on current trends

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ABSTRACT

Drying is one of the foremost and important steps during the processing of agricultural crops, medicinal plants and herbs to preserve their properties. The present review provides a detailed overview regarding the effect of drying techniques on the physio-chemical properties (microstructure, color, aroma composition) and bioactive compounds (phenolic compounds, carotenoids, essential oils, etc.) of plant materials. Factors affecting different drying processes and their optimization strategies have also been discussed. Furthermore, current trends in the development of drying techniques for plant materials in terms of the retention of their bioactive compounds are critically analyzed. Based on the published research articles, oven drying, and microwave drying are the preferable techniques for most plant parts; while for drying the plant extract, freeze/spray drying methods have gained higher interest. Finally, recommendations are made considering the better use of drying techniques for both plant materials and retention of their bioactive compounds.

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Introduction

Plant-derived bioactive compounds are extensively studied as functional and nutraceutical ingredients.^[1] Most fresh plants and a large variety of food commodities are highly perishable materials due to their high-water content. Many fruits and vegetables hold >80% water, which makes them susceptible to deterioration, such as microbial growth and enzyme-catalyzed chemical reactions, causing fast changes in their composition. Hence, removing and/or reducing water

content from plants or foods has always been one of the most important measures to stabilize such biomaterials both in the food processing area and for further development as functional or nutraceutical products.

Drying is an important process which besides the reduction of moisture content and can be applied to concentrate plant extracts, stabilizing them, or adapting them into final products (Figure 1). It is accomplished through a simultaneous transfer of heat and mass through a matrix characterized by variable

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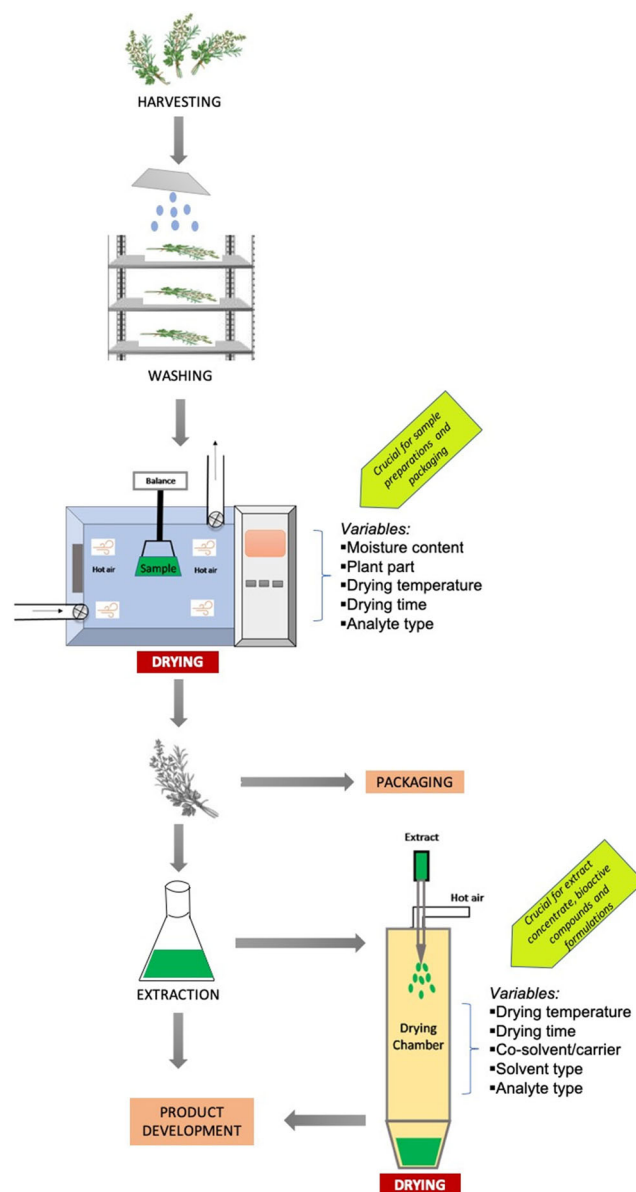


Figure 1. Role and importance of drying technology in the processing of plant and plant-based natural products.

porosity and involves a phase transition.^[2,3] Heat, directly or indirectly applied to the plant material, causes the movement of water from both surface and internal layers.

Drying techniques affect the various aspects of final dried products, *e.g.*, content of bioactive compounds, physicochemical and organoleptic properties, thus largely affecting their biological activities, shelf-life, and safety. The ever-expanding market of plant-based bioactive compounds and supplements needs high-quality raw materials and final products. Therefore, it is necessary to choose the best drying conditions to keep the quality of dried products while also protecting the bioactive compounds of each plant matrix. Furthermore, the drying process has a significant economic impact on the costs involved in the production

of plant-based products, equivalent to 50% or even 90% of total processing costs.^[4] Therefore, drying methods should not only be effective, but also efficient.

Conventional drying (CD) techniques, such as solar and hot air drying (HAD), are often unable to meet high-quality requirements for a dried product, as despite moisture may be highly reduced, their high temperature and prolonged exposure time often lead to the degradation of relevant compounds. Recently, non-thermal drying technologies, such as supercritical drying (SCD), pretreatment with pulsed electric fields (PEF), and osmotic dehydration (OD), have also been proposed.^[2,3] Altogether, development and selection of efficient methods are driven by the need for a drying process with higher efficacy and best quality of

the final product. Therefore, about plant-based products and extracts, the choice of a particular drying technique should meet all the above-mentioned criteria. This work aims to review advances on various drying techniques used to dry plants and/or extracts and plant-based products and to critically evaluate their influence on final product quality, particularly the retention and non-degradation of bioactive compounds. Moreover, this review will also highlight and deepen on current trends, potential chemical changes, factors affecting drying, and optimization strategies considering different plant-based products.

Overview of current drying technologies

Since ancient times, sun, fire, wind, and salt have been used to remove water from the meat, fish, fruits, vegetables, grains, and herbs.^[5] The history of drying plant material goes back to 7,000 BC when the local inhabitants of the Middle East region rolled fruits in palm leaves and then put them in hot sand to dry. From at least 3,500 BC, Incans used sun-dried potatoes (*Solanum tuberosum*), while from 2,800 BC, the ancient Egyptians sun-dried distinct types of fruits. Since 300 AD, tea leaves (*Camellia sinensis*) were dried in India by spreading them on grates. At around 1440 AD, cocoa (*Theobroma cacao*) leaves and beans were sun-dried in Mexico and Peru.^[5] In 1780, the first US patent on a vegetable drying process was registered, in which the vegetables were first boiled and then dried for 30 h. A few years later, in 1795, Masson and Chollet proposed another drying process for vegetables using 45 °C hot air drying (HAD) and applying physical pressure. Since 1870, convection oven drying (OD) technology has been applied to fruits. The spray-drying (SD) process was first developed in 1878 and 23 years later, Robert Stauf registered the first SD patent, applied to milk. Just Hatmaker developed the drum dryer plant in 1902 and 1907, Merrel Soule improved Robert Stauf method by developing the pressurized SD apparatus.^[5] In the 1940s, Coulter developed the high-temperature, high-speed conical spray-dryer, and Flosdorf invented the first vacuum freeze dryer (VFD), based in water sublimation. With the development of microwave-based technology, Percy Spencer developed the microwave drying (MWD) method. Moreover, in 1960, the freeze-drying (FD) of instant coffee was commercialized.^[6] Altogether, several scientific studies have shown that drying is a critical process for the production of a quality end products.^[6,7]

In general, all drying techniques may be grouped as either CD or advanced and innovative drying methods. The latter have been rapidly evolving due to advances in technology. However, it should be noted that such grouping is mostly applicable because of the development of hybrid drying methods, which include CD and helping novel drying techniques. The majority of CD techniques are based on water evaporation at temperatures near the boiling point by heat application to a varying degree.^[8] For instance, the standard method for moisture determination is based on heating the sample at 98 to 100 °C, until its weight does not change. This method was accepted in 1884 by the Association of Official Agricultural Chemists (AOAC).^[9] However, these methods are time-consuming and use high temperature/heat during drying, which may negatively affect the quality of the product such as volume, color, shape, and also the sensitive bioactive contents.^[10] Usually, CD techniques apply conduction and convection methods for heat transfer, which may cause the end product to acquire a poor quality and become susceptible to degradation.^[11]

Novel drying technologies

Due to various limitations of CD, the search is ongoing for new and combined drying techniques of plant materials; so, novel drying techniques have been developed and assessed with various biomaterials during the last few decades. Modification of CD techniques for plants and food drying may help keeping their bioactive constituents as well as improving their physico-chemical and organoleptic properties. The most representative novel drying techniques are depicted in Figure 2. Table 1 gives an overview of the of the pros and cons between advanced drying methods and CD.

Novel hybrid techniques based on mechanisms other than HAD principle may efficiently aid CD. MWD is probably the most advanced hybrid technology, that can be exploited with vacuum drying (VD), spouted bed, FD, fluidized bed, foam mat drying (FMD), and fountain-MWD.^[12] The most significant advantage of combining MWD and CD is the substantial reduction in the drying time and energy consumption as well as an increase in the drying rate and overall efficiency.^[13] For example, cold plasma pre-treatment applied for chili peppers before CD; showed that with a best exposure time of 30 s, the drying rate was enhanced. Additionally, a higher carotenoid content was kept, and antioxidant activity of the dried peppers increased. However, longer exposure times

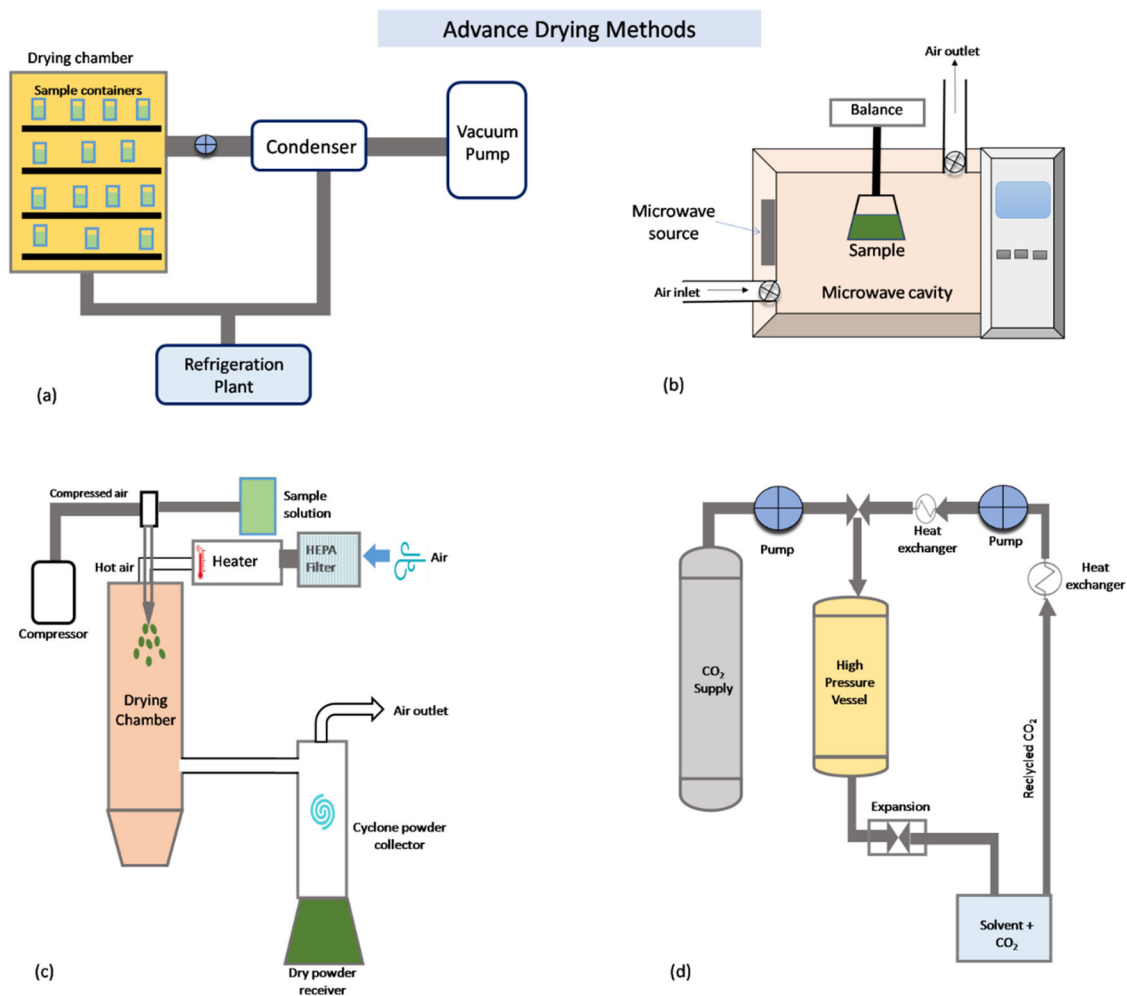


Figure 2. Depiction of some advanced drying techniques. (a) Freeze drying (FD), (b) Microwave drying (MWD), (c) Spray drying (SD), (d) Supercritical drying (SCD).

Table 1. List of advantages and limitations of various drying techniques.

Drying process/Technique	Advantages	Limitations
SoD	Simple Economical	High drying temperature Long processing time Labor intensive
HAD	Simple Economical	High thermal energy Long drying time
MWD	Fast drying Improvement in product quality, volatiles and color retention	Non-homogeneous drying
IRD	High efficiency Short drying time and more energy savings	Penetration power and depth
FD	Low temperature results in the production of a superior quality of end-product	Long drying time High capital and energy costs due to the refrigeration and vacuum systems.
SD	Fast drying ensuring minimum overall loss Versatile and adaptable to a wide range of products Suitable for heat-sensitive materials	Maintenance Issues High capital and overhead costs
VD	Easy to dry heat sensitive materials Average drying temperature is much lower than standard dryers	High operating costs Low efficiency
FBD	Fast, homogeneous drying Suitable for heat sensitive products	Sticky or adhesive materials are difficult to dry
SCD	Higher retention and superior quality Low drying temperature	High cost and complex processing
UAD	Fast process, maintain the texture	High cost
DIC	Maintain the quality of the product, prevent degradation of components, fast process	High cost and complex processing

Abbreviations: SoD, sun drying; HAD, hot air drying; MWD, microwave drying; IRD, infrared drying; FD, freeze drying; SD, spray drying; VD, vacuum drying; FBD, fluidized bed drying; SCD, supercritical drying; UAD, ultrasonic-assisted drying; DIC, instant controlled pressure drop drying.

diminished carotenoid levels.^[14] The authors suggested that this pretreatment may have helped in the release of antioxidant substances and pigments from the matrix surface of the treated samples, which resulted in higher antioxidant activity and pigment content.

Major drivers determining the trends in research and development of novel drying techniques may be summarized as: (i) operational safety, (ii) better product quality, (iii) lower pollution, (iv) higher capacity, (v) better process control, (vi) better economics, (vii) benefits of hybrid drying, and (viii) shorter drying times.^[15] These aspects are primarily associated with the tasks raised for improving industrial drying process. More recently however, they have also become the focus of preserving or concentrating bioactive compounds present in plants and foods.

MWD depends on applying electromagnetic radiation ranging from 300 MHz to 300 GHz. The advantages of this technique include shorter time and lower bulk temperature to remove moisture in foods and herbs as well as its potential to be combined with other drying methods such as VD. The main drawback of MWD is scorching due to the low water content at the end of drying process. Other hybrid technologies are ultrasound (US), supercritical drying (SCD), radiofrequency (RF), and IR-assisted drying (IRD). RF-assisted drying produces waves on the material and results in building electric charge state and vibrational and rotational states. This causes the plant material to heat up without any changes in the temperature of the air surrounding the material. The combination of RF and CD with HAD showed the potential in agricultural crops drying. Novel drying techniques, such as IRD, or explosion puffing drying (HA-EPD) were found to exhibit better quality aspects on the dried product.^[16] On the other hand, IRD uses electromagnetic radiation in the spectral range of 0.75–1000 μm wavelength. The application of IR-HAD hybrid methods on drying plants is a promising novel technology due to a fast penetration and heat transition, reduction in drying time, energy consumption, and improvement in the quality of dried products. On the other side, the penetrating power and depth are still limited.^[17]

SCD is yet another novel drying technique used for drying plant extracts by removing liquid in an exact and well-controlled method. SCD depends on the fact that when the pressure is increased, the interface between the liquid and the gas phase is unstable and when the pressure is larger than the critical pressure, the gas/liquid interface disappears and a mixture of

the two phases appears, which is called a supercritical fluid. This is one of the most effective drying techniques as it achieves a high reduction of moisture.^[18] However, its use is limited due to its process complexity and instrumentation cost. The advantage of supercritical CO_2 fluid is that it is devoid of surface tension, with an excellent mass-transfer, and controllable solvent quality. Hence, it has been applied more recently to avoid the destructive effects of air drying. Furthermore, the SCD technique tends to yield particular compact food textures different to HAD or FD.^[18]

US is a promising technique in the food industry with a lesser impact on the environment, and thus it is also known as a green technology. In addition, US is a non-thermal processing method that uses sound waves with high frequencies, from 20 to 1000 kHz. US induces mechanical, physical and chemical changes through cavitation of the vegetal cell walls and matrix particles. This is applicable to many food processing operations, such as extraction, freezing, drying, emulsification or inactivation of pathogenic bacteria.^[19] The use of US in drying has been carried out by using US pretreatments prior to CD and using air-borne US drying.^[20] These techniques have proven to decrease the total time of drying and improving the water diffusivity.^[21,22] High-intensity US are also used to enhance the drying processes, especially in more complex food matrices. The drying process is dramatically accelerated without causing a noticeable inflation in the temperature of the material. For this reason, US-assisted drying (UAD) method is most useful for retaining thermolabile compounds.

A newly developed technology that could be used as a drying treatment is Instant Controlled Pressure Drop (known in French as “Détente Instantanée contrôlée” as DIC). DIC drying involves elevated temperature and brief time shifts. It consists of a thermomechanical process that applies an instant pressure drop to change the texture of the material. In the first instance, this method requires a short heating step (10–60 s) with an applied high-pressure saturated steam injection (up to 1 MPa) into the previously vacuum-dried matrix. Subsequent vapor condensation and heating increases the moisture (0.1 g $\text{H}_2\text{O}/\text{g}$).^[23] The first vacuum guarantees good interaction between the steam and the sample and hence helps the flow of heat. In certain situations, compressed air may be used in DIC multicycle procedure as a pressurized agent. After the first phase of heating, an abrupt pressure (0.5–1 MPa) decrease in the direction of the vacuum (3–5 kPa) over only 10–60 ms which causes

the product to self-evaporate water, generating vapor and considerable mechanical stress for extending the product. In addition, automatic water evaporation ensures quick cooling that prevents the thermal degradation of sensitive compounds, providing high quality products.^[24] The pressure drop causes the moisture to swell rapidly, and changes the texture leading to higher porosity for improved moisture removal.^[25]

There are also other emerging and less explored drying techniques such as pressure swing drying, rotating jet spouted bed drying, impingement drying, atmospheric FD, photocatalytic drying, ohmic dehydration, desiccant drying, vibration aided drying, sulfur fumigation drying, controlled sudden decompression to VD and pulsed VD. Many studies have reported the advantages of novel and hybrid drying techniques which have been reviewed for various plants and derived extracts during recent years; as an example, different drying techniques and their effects on the quality of biomaterials for which particular drying technique was applied.^[26,27] However, the use of proper drying conditions (individual, hybrid, or novel) is of fundamental importance for achieving the highest quality plant products and their bioactive retention.

Drying effect on the quality of plant-based food ingredients

As mentioned, food quality attributes may be affected by drying technique. Inferior organoleptic and/or structural properties of the improperly dried plant products may negatively affect consumer's selection in purchasing such products.^[28] Although recent advances in drying technologies have opened the possibilities of minimizing quality losses of plant materials; drying may still cause undesirable modifications of food properties, such as discoloration, loss of aroma and nutritional value or undesired texture changes.^[29] Usually, a tight control and optimization of the drying process parameters for each food matrix allows to minimize these undesired outcomes. Below, these changes to food and plant matrices are discussed.

Microstructure (shrinkage and cracking)

Shrinkage and cracking (reduction in external volume and rupture of the cell wall) are common physical changes in plants during drying. Drying due to heating causes loss of water and stress in the cellular structure of the plant leading to change in its shape

and volume. Shrinkage has a negative effect on the quality of dehydrated products and is generally higher the more water the matrix holds.^[30] Nguyen et al. (2018) evaluated shrinkage of potato and carrot during CD at different air temperatures (25, 55 and 65 °C) and air flux speed (1.6 and 0.5 m/s). For all drying conditions, the shrinkage of carrot samples regardless of their cutting shape showed a linear relationship with the moisture content during drying, while potato samples displayed more pronounced shrinkage depending on their cutting shape. Similarly, when comparing HAD or MWD on potential shrinkage of pomegranate arils, it appeared that the drying technique had a different impact on shrinkage, which was more pronounced in HAD while increasing air temperature and microwave power reduced the drying time.^[31]

However, shrinkage behavior depends on the plant material and drying conditions, for instance, in the case of kiwifruits, MWD caused stronger shrinkage than HAD.^[32] Moreover, the structure of apples dried under CD was significantly different compared to microwave-convective or infrared-convective (IR) methods. The MWD and IR drying method reduce the shrinkage, volume, and density but showed increased density when compared to HAD.^[33] HAD, vacuum (VD), and low-pressure superheated steam drying affected microstructure changes of carrots, as evaluated by normalized changes of the fractal dimension and average sample cell diameter, which were correlated to shrinkage and hardness. Both fractal dimension and average cell diameter increased at higher moisture content, equivalent to an increase in the irregularity of the sample microstructure and a decrease in the cell volume.^[34] Drying kinetics in VD proved that the shrinkage of lemon slices followed a linear relation with evaporated moisture content until it approached 60%. Yet, below this point volume remained constant without further shrinkage.^[35]

Some experiments of UAD on different samples have been conducted. It was found that the sample under UAD had a lower hardness than sample dried without ultrasound. Similarly the hardness of dried apples at low temperature was also examined.^[36] The hardness of low-temperature ultrasound-assisted dried cod was also investigated.^[37] It was found that ultrasound promoted the reduction in the hardness of dried and rehydrated cod because it increased the porosity of the matrix. Ultrasound-assisted dried chips were more brittle and slightly crispy as compared to convective HAD dried samples. The effect of US could

be either positive in a low drying temperature or negative in high drying temperature.^[22]

DIC treatment has shown increased porosity in dried matrices. For example, DIC-treated orange peels with additional chemical treatments with Cu, showed increased phenol absorption capabilities.^[25] When applied to carrot chips, DIC produced superior textural properties accompanied by a homogenous pore size distribution. This change was due to alterations to pectic polysaccharides by the DIC treatment. Being a viscoelastic material, their expansion by the pressure alterations resulted in increased porosity.^[38] However, in matrices with higher pectin content such as apple cubes, DIC showed no significant changes in volume but rather in texture.^[39]

Color

Color may be evaluated without contacting the product; therefore, together with product appearance, it is one of the most important quality attributes, deciding consumer's purchasing preferences. Pigment degradation, enzymatic or non-enzymatic browning play a significant role in food color alterations.^[40] Avoiding color loss or browning is especially relevant since it is an indicator of deterioration. This may be measured by brightness (*L*) or yellowness (*a*) indexes. The effect of various drying techniques and conditions on color alterations has been extensively studied. As mentioned, removing water partially avoids generation of reactive oxygen radicals and oxidation. Therefore, although a color loss may result from drying, dried foods will also keep for much longer periods their original colors. For instance, by employing stepwise-varying drying air temperature in 2-stage heat pump drier (HPD) with optimized parameters, it was possible to reduce drying time while improving the color of banana slices.^[41] It was also seen that color degradation was averted by 40% and 23% for a step-up and step-down mode of drying, respectively, in a temperature range between 20 and 35 °C. A significant difference was seen in the overall color change of the banana samples dried by HPD under stepwise temperature variation when compared to other drying systems. Moreover, properly selected stepwise temperature profiles reduced drying time by 75% and minimized degradation of color by 27.6%.^[42] Interestingly, drying may also intensify the color of the dried matrix. In a study, anthocyanin retention was increased in blueberries by HAD at 90 °C, which resulted in a color retention of almost 1.7-fold compared to non-dried blueberries extracts.^[43] However,

in case of hawthorn fruits, at different temperatures and air velocities for HAD, lower drying air temperature and velocity required longer drying times, which resulted in color loss.^[44] Similar results were also reported by other authors, for which HAD decreased the values of lightness while increasing those of the yellowness for blueberry fruits,^[45] mushrooms^[46] or apricots.^[47] These color changes depend on Maillard (non-enzymatic) browning reactions that may be triggered by elevated temperature. For example, shreds of chayote were reported to acquire significantly more intense brown color when drying temperature was increased from 55 °C to 75 °C by HAD.^[48]

The stage of fruit ripening (harvesting time) should also be considered in selecting drying conditions. For example, the browning index of raisins dried by pulsed VD from unripe grapes was higher because unripe material required longer drying time, which was preferable for extended browning reaction.^[49] The brightness of fluidized bed-dried lime leaves was similar to the fresh leaves among all other dried samples, particularly in comparison with HAD, where longer drying time resulted in increased browning.^[50] Similarly, it has also been reported that UAD did not affect red pepper,^[51] carrot or green pepper color^[52] in contrast with MWD. A combination of AD and UAD at -10 °C was found to result in highly significant color retention in salted cod, in comparison to just salted or AD-dried cod.^[37] The authors suggested that ultrasound induced an increase in the lightness and yellow shades of the cod.

In a recent study, DIC was used to keep or increase the color of Zaghoul date snacks. It was reported that brightness was increased may be due to the volume increasing, which traps more air as a result of swelling/expansion of SD.^[40] CD usually need heat and excessive temperature that may result in browning and color loss by degradation of pigments such as anthocyanins and carotenoids and may result in loss of color. However, novel methods employing not requiring such temperature changes may be most useful toward preserving color.

Aroma

Aroma exerts a significant impact on the sensory properties of plants and foods. Besides color, aroma is a key attribute of food quality which may be evaluated without direct contact with the product and therefore may strongly influence consumers' preferences. It can be considered as one of the most sensitive characteristics in the quality reduction of dried products, due to

the loss and/or degradation of volatile organic compounds, since many of these are prone to oxidation and/or thermolabile.^[53] Besides, undesired volatile compounds may also appear because of degradation and oxidation, for which drying may be a solution. Studies proved the role of drying on preservation of aroma constituents. Tian et al (2016) compared HAD, VD, MWD, and MWD-VD of shiitake mushrooms. Their results showed that MW-VD was the best method, with a better structure, color, nutrients and retention of taste-related amino acids. Another study reported that drying by different techniques such as IR, HAD, FD or MWD altered the volatile and phenolic composition of ginger roots.^[54] HAD and IR showed an increase of volatile compounds, being sesquiterpenes like zingiberene, β -sesquiphellandrene, geranial or α -farnesene. Additionally, esters like propionic acid or 2-methyl-, 3,7-dimethyl-2,6-octadienyl ester were formed by HAD, enhancing the ginger profile. However, monoterpenes (β -phellandrene and camphene) decreased in non-thermal methods. Most importantly, IR and FD retained ginger particular ketones like 6-, 8- and 10-gingerol and 6-shogoal by more than 50% in comparison with other methods tested.^[54] Moreover, FD poses a better choice to keep aromatic volatile compounds, as no heat is required. For example, FD preserved the characteristic aroma of fresh guava fruit, as FD powder contained higher contents of (Z)-3-hexenyl acetate, hexanal, and (E)-2-hexenal, which are responsible for guava flavor.^[55] Regarding aromatic profile, it appears that novel techniques that use less heat are more suitable for the preservation of volatile components and avoid their degradation.

Effect of drying on bioactive compounds

The quantity and integrity of bioactive compounds in processed plant materials are essential as they manage the desired biological effects as function and/or nutraceutical ingredients. Drying techniques have been extensively used to improve the shelf-life of the products, supplying safer food; however, these techniques often have a negative effect on bioactive compounds influencing the nutritional quality of the final product. Some of the most relevant tests on target bioactive molecules are listed in Table 2. The effect of different drying techniques on the recovery of bioactive compounds and their chemical modifications for food processing will be discussed in the following section.

Phenolic compounds

Phenolic compounds (PC) are plant secondary metabolites characterized by having aromatic rings and numerous hydroxyl groups in their structure. This confers them antioxidant properties, for which they have been thoroughly studied and made them natural molecules of interest. Several classes of phenolic compounds have been described; such as phenolic acids, flavonoids, tannins or stilbenes, among others.^[77] Numerous studies have been conducted to figure out the effect of drying techniques on the recovery and content alteration of phenolic compounds. For instance, it was reported that pistachios dried by MW-OD, exhibited higher total phenolic content (TPC) than raw nuts.^[78] This could be due to a release of these polar molecules by the MW radiation. Similarly, syringic acid, ferulic acid, protocatechuic acid, and piceatannol contents of MW-HD-berries were more pronounced in HD and MWD. However, the joint method of MWD-HAD was found as a preferable technique in terms of phytochemical contents in the dried rose hip. This was due to a shorter process time by the use of MW, which helps to preserve phenolic compounds, and the better moisture reduction provided by HAD.^[58] This is related to a better protection of phenolics in MWD-samples than HAD-samples attributed to a shorter exposure time to oxygen and lower enzymatic browning despite the higher temperature in MWD. This leads to an overall lower oxidation.^[13,79] In addition, MWD, such as observed in microwave-assisted extractions, can induce the release of phenolic compounds, which are bound to macromolecules such as polymeric carbohydrates or proteins and allow them to act as antioxidants once unbound.^[80]

Higher content and availability of bioactive compounds was also reported in DIC-dried products. Because of the DIC treatment, the texture and the open porous structure enables these compounds to become more available. For instance, the content of quercetin in DIC textured apples was concentrated as much as 7 times compared to fresh samples, which correlated with a higher antioxidant activity.^[81] DIC has also shown to be an effective method for aiding on essential oil extraction. Essential oils are oily fractions that may be extracted from flowers and other plant parts, rich in lipidic and antioxidant molecules. Using an 80% ethanol solvent extraction, essential oil extracts from DIC-treated rosemary leaves were reported to double rosmarinic acid content in in comparison with untreated leaves.^[82] Similarly, convection MVD, resulted in high quality of dried chokeberry fruits as

Table 2. Effect of drying techniques and its conditions on the recovery of bioactive compounds from plants.

Plant/Part	Compounds	Drying techniques	Type of changes	Ref.
Papaya leaves	Polyphenols	OD, SD and FD	OD better preserve TPC at 40 °C, FD better preserve vitamin C	[56]
Cashew nut	Polyphenols	MWD	Gallic acid and (+)-catechin higher in concentration, while major reduction in catechol concentration	[57]
	Oil content		Heated at 720 W, degrade palmitic and stearic acids; while it better preserve linoleic and linolenic acids	
Myrtle berries	Polyphenols	HAD, MWD and MW-HAD	MWD better preserve gallic acid and cyanidin-3-O-glucoside, while syringic acid, ferulic acid, protocatechuic acid and piceatannol contents are better restored in berries dried under MW-HAD	[58]
Birch leaves	Flavonoid glycosides	FD and OD	Flavonoid glycosides better restored under FD	[59]
Chokeberry	Phenolic content	CVMD, CD and MVD	Phenolics better restored in berries dried under CVMD	[60]
Guduchi	Polyphenols	SD	Retention of polyphenol improved along with higher solubility of powder sample	[61]
Guava fruits	Polyphenols	OD and FD	Loss of soluble phenolics during drying	[62]
Pumpkin	Polyphenols	OD and FD	TPC better preserve in OD	[63]
Lime peels and pulps	Polyphenols	FD and TD	FD recovers lower level of hesperidin, eriocitrin and naringenin	[64]
Various fruits	Polyphenols	OD and FD	Phenolics better preserve in FD	[65]
Jambolan juice	Polyphenols	FD and OD	Gallotannins degrade under OD, while ellagitannins was better preserved in FD (70 °C) and OD (80 °C), Myricetin degrade under both	[66]
	Cyanidin-3-glucoside	FMD and FD	Cyanidin-3-glucoside degradation higher under FMD at 80 °C	
Cranberry juice extract	Chlorogenic acid	FD, VD and SD	VD showed higher recovery of chlorogenic acid, better recovery of flavan-3-ols under FD and SD	[67]
	Amino acids		Formation of 2-furoylmethyl amino acids detected after FD and VD	
Meadowsweet	Flavonoids and condensed tannins	FD, OD and TD	FD better preserve flavonoids while condensed tannin was higher in TD (70 °C) and OD (70 °C)	[68]
Willow leaves	Salicin	FD, OD and TD	Salicin better preserve at higher temperature	[69]
Neptune grass	Flavonoids	FD and OD	Quercetin glycosides degrade under FD	[70]
Sour cherries	Polyphenols and ascorbic acid	CD and MW-OD	MW-OD better recover TPC and vitamin C	[71]
Green tea	Polysaccharides	FD, VD, SD and MVD	SD showed lowest recovery of polysaccharides	[72]
Citron	Polysaccharides	OD, VD and FD	FD better preserve polysaccharides	
Aloe gel	Acemannan	SD, IRD, and RWD	Drying degrade acemannan by loss of (1,3,4)-linked mannosyl residues	[73]
Finger citron fruits	Polysaccharide	FD, HAD and VD	FD better prevent degradation of polysaccharides	[72]
Chinese ground orchid	Polysaccharides	VD and VFD	Higher solubility of VFD sample powder	[74]
Sugar beet pulp	Pectin	HAD, VD, FD and SD	HAD decreases the degree of esterification in pectin	[75]
<i>Hohenbuehelia serotina</i> (fungi)	Polysaccharides	VFD, VD, FD and HAD	Intrinsic viscosity of polysaccharides was higher in VFD	[76]
			(Z)-3-hexenyl acetate degrade while terpenes were better preserved after drying	[62]
Guava fruits	Volatile compounds	FD and OD	(Z)-3-hexenyl acetate, hexanal and (E)-2-hexenal better restored in FD sample, while limonene and β -caryophyllene are found only in OD samples	[55]

Abbreviations: FBD, fluidized bed drying; FD, freeze drying; IRD, infrared drying; HAD, hot air drying; MWD, microwave drying; OD, oven drying; RWD, refractance window drying; RWD, radiant zone drying; SCD, supercritical drying; SD, spray drying; TPC: total phenolic compounds; VD, vacuum drying; VFD, vacuum freeze drying.

compared to CD and MVD.^[83] Also, the longer drying time of chokeberry fruit at 50 °C in CD resulted in higher degradation of polyphenolic compounds, higher than in short drying at 70 °C. UAD-FD, another

innovative drying technique, which was recently developed and tested for TPC in eggplants using various air temperature, velocity, ultrasound power, and sample dimension variables and give negative and positive

results.^[84] As such, the TPC increased with decreasing air temperature from -5°C to -10°C and air velocity from 5 to 2 m/s; however, increasing the ultrasonic frequency has a negative effect.^[84]

Regarding the effect of drying on flavonoids and tannins, it was reported that the content of flavonoid glycosides, including quercetin glycosides were significantly higher in FD birch samples than in OD samples, and particularly dried at 80°C compared to 40°C gives better results.^[59] However, Harbourne et al. did not find any significant differences in the levels of condensed or hydrolyzable tannins among FD and air-dried meadowsweet samples. Nonetheless, flavonoid levels were lower after 70°C OD, condensed tannins, on the contrary, were more abundant in tray-HAD dried and 70°C OD samples but lowest in 30°C OD.^[68] The decrease of flavonoids and increase of condensed tannins proportions in tray-dried and OD samples was explained by the polymerization of flavonoids to condensed tannins during drying. Likewise, rosehip berries dried under HAD, MWD, and MW-HAD joint displayed diverse relation of phenolic compounds. The MWD-dried berries contained significantly higher levels of gallic acid and the flavan-3-ol cyanidin 3-*O*-glucoside, than HAD or MWD-HAD-dried berries.^[58]

Reports showed that the drying technique gives different results depending on the analyzed plant species, its anatomical part, and the chemical properties of bioactive compounds. For instance, FD samples of Neptune grass had a significantly lower concentration of quercetin glycosides than OD samples dried at 40°C .^[70] De Carvalho et al also reported that the concentration of gallotannins was lower in foam mat dried FMD jambolan (*Syzygium cumini*) samples than in FD.^[66] However, the concentrations of ellagitannins in FMD (70°C and 80°C OD) samples were recorded higher; with significant reduction was observed when samples at 60°C . These results suggested that the drying of the foams at 70 or 80°C may be suitable for obtaining dried powders from jambolan juice. Moreover, it was also noticed that the content of myricetin holding 3-hydroxyl groups in B ring was highly reduced in the dehydrated jambolan powder, which was explained by the increase in the proportion of its more stable methylated derivative laricitrin at 80°C . The drying method affected the percentage of quercetin and its four glycosides (3-*O*-galactoside, 3-*O*-pentoside, 3-*O*-rhamnoside, and benzoyl-galactoside) in cranberry powders.^[67] The highest amount of flavonols in powders made from sugar-free juice extract was obtained by FD and more cost-efficient SD, while

in VD increasing temperature resulted in a slight (4% concentration) degradation of quercetin-3-*O*-galactoside, quercetin 3-*O*-pentoside and quercetin 3-*O*-rhamnoside (aglycone quercetin did not decrease by increasing temperature). Heating may hydrolyze the flavonol glycosides, as it was demonstrated in the case of cranberry pomace, which accumulated the highest content of quercetin after drying at 60°C .^[85]

In summary, the changes of phenolics during drying may depend on factors, namely their degradation, particularly during prolonged drying time at higher temperatures; the release of bound phenolics; the release of phenolic acid derivatives, and many others. The detailed changes in the composition of phenolic compounds at different drying conditions were reported for guava fruits (Nunes et al., 2016), which suggested that flavonoids were more stable than non-flavonoids, while FD caused less significant changes than OD during guava drying.

Pigments

Anthocyanins are flavonoid-like pigments found in dark-colored fruits and very sensitive to heat and pH shifts. In recent years these molecules have been linked to numerous health benefits.^[43] Therefore, their sources require careful processing to keep their stability. For instance, increasing drying temperature above 60°C of grape and blueberry pomace resulted in a significant reduction in total anthocyanin content.^[86] Comparison of FMD at 50, 65, and 80°C of sour cherry juices, revealed that the highest concentration of anthocyanins in the powder was obtained at 65°C .^[87] In a recent drying study, it was reported that FD and SD of cranberry juice were superior in preserving cyanidin-3-*O*-galactoside and 3-*O*-arabinoside, peonidin 3-*O*-galactoside, 3-*O*-glucoside and 3-*O*-arabinoside from degradation as compared to VD. Moreover, the higher temperature used in VD increased the degradation of anthocyanin content. A negative effect of applying higher temperature was also reported for strawberries drying, which results in significantly decrease anthocyanin concentration comparing to lower temperature of 50°C .^[88] Moreover, the degradation kinetics of anthocyanins showed that it occurs rapidly in the first 100 min of drying. This was followed by a period of stability that stopped when the water content became less than 0.5 g water/g dw at 60°C and less than 1.1 g water/g dw at 50°C . Then, further degradation of the anthocyanin took place in the last 50–100 min at both temperatures.^[88]

Studies have also reported the effects of different drying procedures on the carotenoids, one of the major compound classes that have various biological activities and also contributing to the color of plant material/food products. Cui et al. studied the effects of MVD drying alone or combined with either HAD or VD on the carotenoid contents of carrot slices. Carrot slices were first dried by MVD until the moisture content reached about 20% and then dried further dried by either HAD or VD. As compared with conventional HAD or FD, these methods provided better retention of carotenoids than hot air drying and similar performance with FD.^[89] For example, it was reported that the direct sunlight drying of mango fruit and cowpea leaves resulted in the high loss of carotenoids in both of these plants while using a visqueen-covered solar dryer could reduce the loss of carotenoids.^[90] In a similar study, the effects of direct sun or tent-drying with UV-resistant or non-UV resistant polyethylene on the total carotenoid contents of sweet potato chips was assessed.^[91] These drying methods did not differ on the loss of carotenoid content which showed a low reduction ($\leq 15\%$). Another work evaluated the effects of FD and HAD on the carotenoid profile of “Ataulfo” mango by-products (paste and peel). It was reported that the FD paste had greater carotenoid concentration than peel, however, the HAD peel displayed greater carotenoid concentration than paste. The authors concluded that the drying methods had significant effects on carotenoid contents, but this was also dependent on the food matrix (peel or paste).^[92]

Polysaccharides

Besides their roles in energy storage and of cell wall structure, many polysaccharides have been reported to exert bioactivities and health benefits, such as, antioxidant, anticancer, immunomodulatory and radioprotective activities, which frequently are closely related to their structural characteristics and spatial conformation, which changes during drying, among other processes.^[93,94] Studies have shown that acetyl groups of acemannan, a bioactive *Aloe vera* polysaccharide, may irreversibly change its structure and bioactivity extent during the drying process. This was tested by comparing SD with some novel drying techniques like IRD, Refractance Window drying (RWD) and Radiant Zone drying (RZD) techniques applied at industrial scale.^[73] The study found that all drying techniques reduced the yield of acemannan along with its water retention capacity (WRC); which may lead to the loss

of hypoglycemic activity of *Aloe vera*. WRC reduction may be explained by the solidification of *Aloe vera* layer during drying due to deacetylation.^[95] The degree of acetylation of acemannan reduced during drying, which correlates with the losses of (1,3,4)-linked mannosyl residues.^[73] The deacetylation of the acemannan promoted by different drying processes has also been observed in other studies using Fourier-transformed infrared spectroscopy.^[96] Drying may also alters fat adsorption, which is related to the polysaccharide binding capacity to the organic molecules and hence reducing the cholesterol level, carcinogens, and other toxic compounds.^[93]

For green tea polysaccharides extracted from the leaves dried using FD, VD, SD, and MVD, the lowest yield was obtained in the case of SD.^[72] Polysaccharide from SD material had lower contents of protein and tea polyphenols, possibly due to their degradation at high temperature (160 °C) and also showed weaker antioxidant activity. However, FD matrix showed significantly higher antioxidant activity, while VD had better inhibitory properties for α -glucosidase and α -amylase. This suggest that different drying methods incur in different retention of bioactive components. VFD powder containing polysaccharide from Chinese ground orchid was better soluble than VD powder.^[74] Moreover, the weight of VD powder was higher than that of VFD and signifies that the polysaccharide molecules easily aggregated under VD. A HD, VD, FD, and SD on sugar beet pulp pectin.^[75] Results suggested that increasing HD temperature decreases the degree of esterification, which could be explained by the destruction of the methyl esterified carboxyl groups of the pectin side chains; however, VD and SD, although performed at elevated temperature, did not affect DE. Additionally, the authors saw a decrease in apparent viscosity of VD samples with the increase in temperature from 40 to 60 °C, while SD (190 °C) and FD samples had the highest and the lowest viscosity, respectively, most likely due to the differences in pectin molecular weight.

Alkaloids

Alkaloids are nitrogenated secondary metabolites in plants and present a broad range of structural diversity. This chemical class groups, in which some of them are well-known hazardous molecules, but which can also display medicinal properties, like capsaicin for example. Hossain et al. studied the effect of the different drying processes (HAD, VD, and FD) on the

steroidal alkaloids content of potato shoots, peels, and berries. During HAD, the content of solanine and α -chaconine increased until 7 days but decreased after 9 days. In potato shoots, FD potato shoots showed higher α -chaconine contents.^[97]

The effects of different drying techniques were studied on the content of cathinone in khat (*Catha edulis* Forsk) leaves. FD of the leaves resulted in the highest content (73%) of cathinone however HAD, sun drying, and OD resulted in the decreased yield of cathinone.^[98] As with other bioactive molecules, alkaloids seem to be better preserved by non-thermal drying treatments.

Vitamins

Ascorbic acid (or vitamin C) has been known as one of the most sensitive bioactive compounds to heat and a valuable healthy and antioxidant part of plant foods. Its stability and retention are thus relevant to obtain dried foods of higher quality. As a reactive and easily oxidized molecule, non-thermal methods like FD are a better choice than HAD or sun drying as shown in dried papaya fruits.^[56] Even though it may be disruptive, properly optimized convective MWD of sour cherries coupled with HAD was shown to retain more ascorbic acid content (76 mg/100g) than HAD alone (58 mg/100 g).^[71] But not only ascorbic acid is a valuable molecule to be preserved. Retaining other vitamins in a food sample is of great interest. Optimized UAD-HAD of apple cubes allowed to retain more content of B vitamins like B₁, B₂, B₃, B₅ and B₆ in comparison to HAD alone.^[99] However, tocopherols (vitamin E), were diminished with the US treatment.

Factors affecting the drying process and its optimization strategy

Plants tissues are highly structured and complex as they have heterogeneous, hygroscopic, morphological, and porosity properties. Basically, in each tissue, three different cellular environments: the intercellular space, the cell wall, and the intracellular space, contain very different proportions of water and has a significant effect on the morphological behavior of plant material during drying.^[100] Thus, an understanding of the plant material's microstructure is critical to understand and select the most suitable drying processes for improving the final product quality as well the energy efficiency. However, available studies on the influence of these factors and process conditions are scarce. Most of the available studies are based on theoretical

and semi-theoretical models^[101,102] Furthermore, due to differences in the nature of plant materials, the standardization of process parameters, and methodology, must be considered and may hinder optimization processes.

Plant physical characteristics

From the perspective of drying, the plant material holds a cellular structure through which heat and mass transfer occur. The effectiveness of both processes depends on the resistance against heat transfer or mass diffusion in the tissue. In most of cases, heat-transfer resistance is lower than mass-transfer resistance. Hence, drying kinetics are mainly dictated by mass transfer.^[103] Among the main factors that determine the resistance are the dimensions of the plant material, together with its structure and chemical composition.^[104] Therefore, origin, nature, composition, and developmental stage are factors to be considered when approaching a drying procedure to guarantee the final quality and properties of the dried material. For any tissue to be processed: flowers, fruits, leaves, bark, roots, seeds, rhizomes, etc., the macromolecular composition (water, fiber, starch, sugars, surface cuticle, bioactive compounds, etc.) determine its physical properties and interactions with water to a different extent, which will have a direct impact on the drying process.^[105] For example, crystals, membranes, and lipids all contribute as barriers to moisture migration. The thickness, pore distribution, and the formation of channels are also determining factors for the preferential path for water transport.^[106] The smaller the size of the pores in the matrix of the food domain, the slower the moisture migration. The porosity of the plant material decreases as the concentration of fibers in the tissue increases. Fibers also reduce the size of the plant cell and increased their number.^[107] Rigid components such as cellulose, pectin, mucilage, lignin, and associated substances, (like cutin, suberin or waxes), are also crucial factors deciding pore size and therefore the drying behavior of plant materials.

Pretreatments

Complex matrices such as whole or fractioned fruits may be difficult to dry effectively using a single or even combined drying method. For instance, the outer surface of fruits is often covered with a thin layer of wax cuticle, which hinders water mass transfer and reduces the drying rate. To break down the wax

cuticular fruit surface and creating microscopic cracks that increase permeability various physical and chemical pretreatments, such as mechanical grinding, ultrasonic or chemical pretreatments can aid the drying process (Table 3). Common chemical pretreatments consist of dipping fresh fruits into solutions of fatty acid esters (such as ethyl or methyl ester emulsions) or alkali solutions (sodium hydroxide and potassium carbonate solutions) for several seconds.^[108] Also, ultrasonic pretreatment increases the effective diffusivity of water into the plant tissues by increasing porosity of the cell walls, thus reducing the drying time by 10–30%.^[109] Further, the pretreatments are also performed in fruits to enhance the retention and stability of certain bioactive compounds. For example, osmotic dehydration, which consists of the immersion of the plant material in an hypertonic solution (of sucrose, glucose, fructose, starch concentrates, corn sirup, or sodium chloride) in a way that the semi-permeable nature of the plant tissues and the lower molecular size of water molecules allow water movement from the tissue and solute gain from the hypertonic solution.^[110] As such, it was reported that osmotic dehydration combined with US increased the anthocyanin levels in strawberries,^[111] and resulted in a high content of total anthocyanins and total flavonoids in cranberries,^[112] while microwave blanching aided in preserving anthocyanin levels of sweet potato.^[113] These pretreatments inactivate endogenous enzymes (peroxidase, polyphenol oxidase, or glycosidases) that can degrade anthocyanins and other antioxidant phenolic compounds.^[114] Moreover, pulsed electric fields pretreatment results in greater retention of carotenoid pigments in carrots, sweet potato, and tomato.^[115,116] High hydrostatic pressure pretreatment preserves the structure of small molecules, such as pigments, volatile compounds, vitamins, and amino acids, due to their relatively simple structures. In contrast, larger molecules, such as enzymes are affected and inactivated, potentially explaining why under the influence of pressure an improved antioxidant activity may be observed.^[117] Recently, a new pretreatment process called plasma processed air was tested as pre-drying treatment of plant tissues of cut apple and potato as a less disruptive alternative to traditional methods, such as pasteurization or addition of antioxidants.^[118] Hence, pretreatments not only improve the efficacy of the drying process, but can also aid on the release of bioactive compounds.^[119] Thus, optimization of pretreatment conditions can reduce the drying time; improve yield, and the functional properties of the dried plant products.

Optimization strategies

Drying processes need to be optimized to achieve efficient operating conditions, considering the moisture contents of foods and the high-quality final products with sustained nutritional value. Generally, nutritional and bioactivity loss increases with the severity in the process conditions during drying processing, which is minimized by applying suitable pretreatments, selecting appropriate drying techniques, and optimization of drying conditions.^[120]

Nevertheless, optimization of drying conditions is always performed to guarantee the best results in terms of high quality of plant material and bioactive compounds preservation.^[56,121] However, energy consumption (and subsequently, economic costs) of a drying process is a key factor affecting its feasibility and implementation. From recent estimates, drying processes use between 10% and 15% of national industrial energy consumption in developed countries, and between 30% and 40% of total energy consumption in developing countries.^[2] When comparing drying methods, specific moisture extraction ratios must be considered for each matrix and method, usually expressed as kWh/kg of water. For example, it was reported that under different conditions, MW dried roman chamomile required several times less energy (1.39 kWh/kg *w*) than VD (88.45 kWh/kg *w*), whereas MW-VD combined achieved a maximum of 5.76 kWh/kg *w*.^[122] A recent, extensive review compiled a great number of optimization studies assessing this topic. The authors found that in general terms, HPD may be more energy-efficient than other methods such as MWD, but also that the latter would be generally more efficient than VD.^[4] Regardless to the energy expenditure, as discussed, the susceptibility of each compound to processing conditions should be evaluated, including temperature (*e.g.*, essential oils, ascorbic acid), UV-rays (*e.g.*, carotenes), enzyme activity (*e.g.*, glycosides), and others.^[123]

For every drying technique, regardless of the CD or novel, optimized parameters should be adapted as per the biomaterial requirement. For instance, leafy and soft plant materials are comparatively easily dehydrated and usually achieve effective drying yields, while the size of hard anatomical parts (roots, rhizomes, bark, etc.) should be carefully selected to achieve sufficient water diffusivity rate from the inner parts of the product under drying. However, one of the main tasks of drying is the protection of constituents, which are sensitive to heat, oxidation, and other undesirable reactions. For instance, it was shown that processing of crushed broccoli at 30–60 °C could

Table 3. Various types of pretreatments for plant material before drying processes.

Chemical pretreatment		Physical pretreatment	
Liquid	Gas	Thermal	Non-thermal
Alkali (NaOH) Acid (H ₂ SO ₄ and HCl) Hyperosmotic solutions (sugars, biopolymers)	Carbon dioxide Ozonation SO ₂	Hot water Infrared heating Microwave Ohmic heating High-Humidity Hot Air Impingement Blanching	Freezing High hydrostatic pressure hydrodynamic treatment High-pressure homogenization Pulsed electric field Ultrasonic field Mechanical disruption

Table 4. Factors affecting various drying techniques.

Drying techniques	Instrumentation based	Plant-based
MWD	Power, time, temperature	Moisture content
VD	Pressure, time, temperature	Plant part
FD	Freezing rate, chamber pressure, sample temperature	Harvesting time
SD	Inlet air temperature, carrier concentration, feed flow rate	Plant structure
FBD	Air temperature, time, weir height, fluidization velocity	Viscosity
UAD	Ultrasonic frequency/power, time, temperature,	
DIC	Pressure, temperature	

Abbreviations: MWD, microwave drying; VD, vacuum drying; FD, freeze drying; SD, spray drying; FBD, fluidized bed drying; UAD, ultrasonic-assisted drying; DIC, instant Controlled pressure drop drying technology

result in the conversion of L-ascorbic acid to dehydroascorbic acid, which could be easily converted to other degradable and pro-oxidant compounds.^[124]

Considering the complexity and the effects of numerous possible variables, mathematical modeling has been widely used for optimizing drying processes^[120] For example, the Design of Experiment approach, which allows a process to be set up in the shortest time for the best yield conditions, has been mostly used.^[120] Based on the individual drying conditions, a range of factors govern the drying process and affect the final product quality needs to consider and optimized for better product quality (Table 4). This can be achieved either using a univariate or multivariate statistical model, such as response surface method. These multivariate models when operated results in the optimization of process variables that allow to maximize a desired yield.^[120,125]

Trends on drying techniques

Innovative drying techniques for plant materials, bioactive compounds/extracts, and its products have been developed in recent years. However, due to expensive instrumentation and high operating costs, the use of CD techniques is still generally preferred by industry and research. A bibliographic search was made in the Scopus database (www.scopus.com, Elsevier) on November of 2020, considering several parameters listed in Table S1. A compilation of the use of different drying techniques used in various food product formulation and extract concentrate was made.

With changing drying techniques and conditions as per the need and desire, various research articles

have been published. Table S2 shows the number of articles dealing with the drying of plants based on its specific parts such as roots, leaves, fruits, rhizomes, and flowers. Among the mentioned drying methods, the FD technique was mostly used followed by OD, while MWD and VD are least used for most plant parts (Figure 3a). FD increases the shelf-life of dried product and thus a preferable choice over other drying techniques. OD is operationally simple, and the equipment required to perform it, relatively more affordable than for other methods. Regarding plant parts subject to drying, fruits are the most represented, given their commercial interest. However, most of the articles only mention the term “drying” without elaborating on the technique employed, which pays off in a considerable number of gaps in articles searching with drying terms and specific techniques term.

Depending on the bioactive compound of interest such as flavonoids, phenolic acids, alkaloids, polysaccharides, etc., the use and trends of drying processes vary (Table S3). However, for all bioactive compounds, except essential oils, FD is the first choice with a higher number of research articles mentioning or studying its use (Figure 3b). FD is a relatively modern technology compared to others and also should be taken into account that many papers did not mention the method of drying, especially in CD techniques like OD and VD. The increased use of FD in recent years may be due to its advantages over other drying techniques, the absence of heat in the process, which results lower potential thermal degradation of the metabolites and plant products. The second choice of drying varies with the

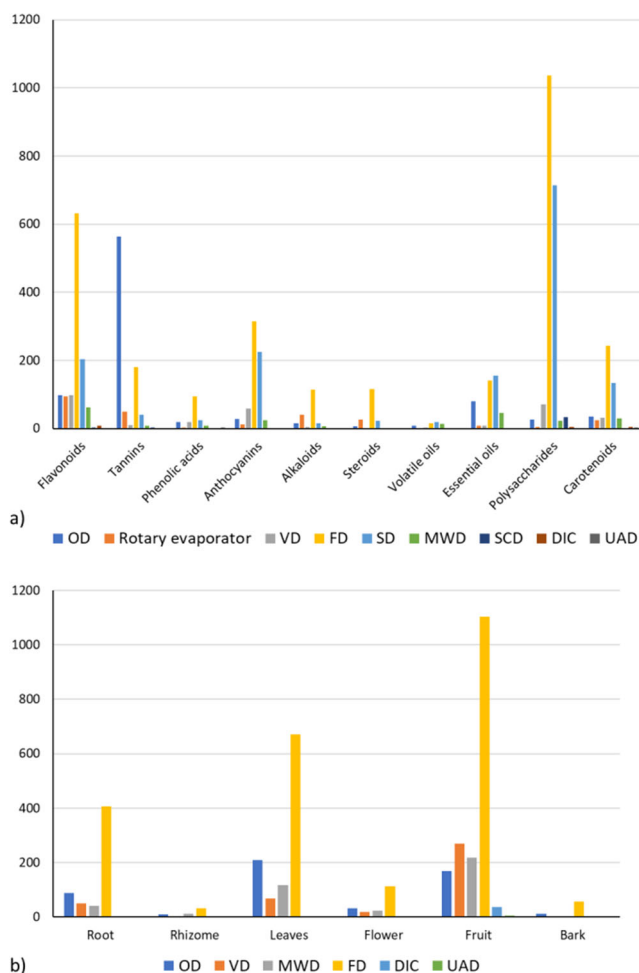


Figure 3. Trends of various drying techniques used for (a) different drying methods used for plant parts, (b) different classes of compounds, and (c) optimization experiments for classes of compounds and (d) plant parts based on the articles retrieved from the bibliographic search.

compound of interest. For instance, for tannins, OD is the second most popular choice, while for anthocyanins, SD seems to be preferred. Rotary evaporator, which is simple to run and used to concentrate the extract before its further drying into powder form is a pre-drying step, used for alkaloids and tannins (Figure 3b). It was found that, SCD is not well-represented, due to its high instrumentation costs and operational complexity.

To obtain the desired dried products of superior quality, different drying techniques and conditions were optimized based on the nature of plant material, bioactive compounds, or finished products. Very few optimization studies have been performed with respect to drying techniques (Table S4). A higher number of optimization studies have been conducted on drying fruits as compared to other plant parts (Figure 4a). Fruits are significant in the food and beverage industry and are prone to early decompose. Articles mentioning leaves and roots also have high numbers of

optimization studies, which may be due to their importance in the pharmaceutical, cosmetic, and food industry. Concerning bioactive compounds, polysaccharides have been most studied as targets for optimization of drying conditions, followed by flavonoids (Figure 4b).

Polysaccharides are widely used in the food and pharmaceutical industry and due to their structural-functional activity, which is affected by the drying process and its conditions. Analyzing from the past 25 years, the research on the optimization of the plant drying process is kept on increasing (Figure 5, Table S5). From 1996 to 2000, only a few papers were published on drying optimization. Yet this was increased significantly every next 5-year period.

The increasing research on drying process optimization supports its relevance over plant processing for sample preparations for analysis and/or product development. Hitherto a critical control should be exerted over the several processes' parameters.

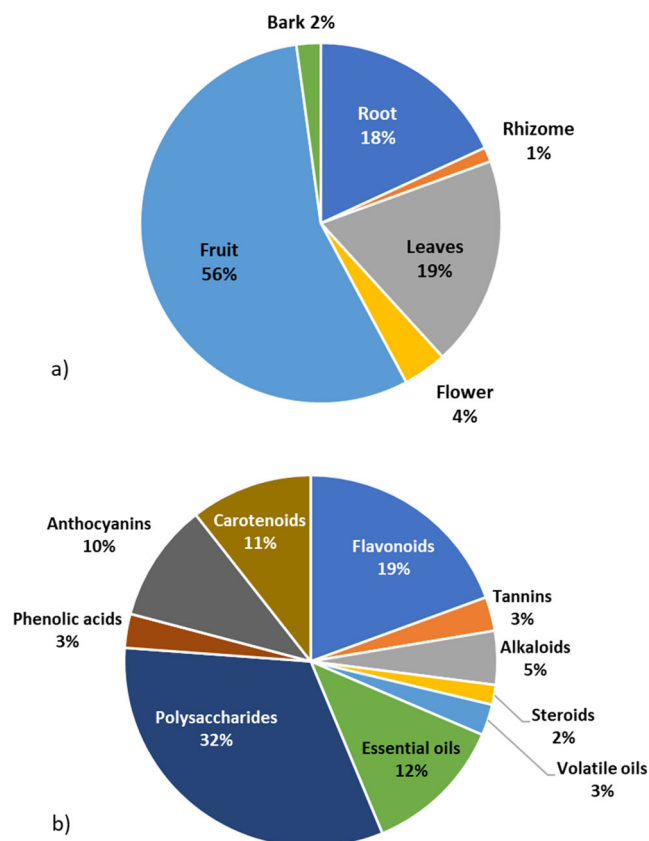


Figure 4. Trends of various drying techniques used for (a) distinct groups of bioactive compounds and (b) different drying methods used for plant parts, based on the articles retrieved from the bibliographic search.

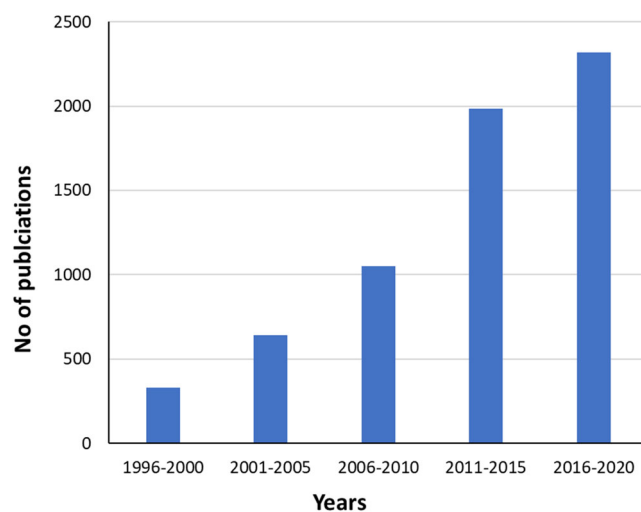


Figure 5. Number of research articles published on drying optimization studies from 1996 to 2020 grouped by 5-year periods based on the articles retrieved from the bibliographic search.

Conclusions and future perspectives

Due to the differences in characteristics of samples to be treated and/or the compound of interest, there are different choices for selecting the drying techniques and conditions. Each drying technique has positive and negative aspects and particular characteristics that may affect the drying kinetics and quality of the final

products. Despite the efforts made to improve traditional drying methods and to develop new techniques, many hurdles still hamper universal application of a single drying method, as these impact organoleptic or functional attributes in different ways. Several studies have been focused on the effects of drying on nutritional characteristics of plants, particularly

health-beneficial phytochemicals but also on other chemicals influencing organoleptic characteristics such as color or aroma.

Given the vast diversity of matrices, compounds of interest and drying methods available, optimization studies regarding drying parameters such as temperature, air velocity, time or power, are still a focus of research for both industry and academia, as well as the nature of the starting materials has a greater influence on its quality and on the degradation of compounds. These parameters are critical on the final retention of bioactive compounds. Depending on their nature and lability, these compounds occurs may be significantly degraded during the dehydration process but also during drying pre-treatments. Thus, it is of great relevance to find and establish optimal drying methods that allow to preserve both the matrix integrity and phytochemical contents. However, as discussed, many of the studies performed lack assessment of parameters in a wider spectrum. Therefore, it is of critical significance that future studies in this topic assess several aspects that greatly influence drying dynamics and bioactive compound retention; as well as detail as much as possible methods and results to allow an accurate replication. These aspects include: (i) drying method, (ii) detailed instrumentation, (iii) detailed process parameters such as temperature, time, frequency, power, etc., and (iv) clear description of the plant material's conditions, such as species and cultivar, color, moisture content, or harvesting time.

While economical suitability and effectiveness must be pursued considering drying processing, effectiveness, efficiency and minimized environmental impact should be drivers for the development and implementation of new processes. This is, also maintaining effective drying performance. As discussed, novel methods such as MWD are effective and require relatively low energy input, but can also likely degrade bioactive compounds of interest, while VD needs far more energy but doesn't show those drawbacks. Nonetheless, further research should describe optimized methods for drying plants and foods aiming for improved preservation of the present natural bioactive molecules as this can dramatically improve the overall quality of the dried material.

Disclosure of interest statement









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