



Innovative physical techniques in freeze-drying

Oksana I. Andreeva^{ID}, Ivan A. Shorstkii*^{ID}

Kuban State Technological University^{ROR}, Krasnodar, Russia

* e-mail: i-shorstky@mail.ru

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Abstract:

Malnutrition is a global problem that is caused by insufficient sources of vitamins, microelements, and other nutrients. This creates a need for developing long-term preservation techniques. One of the solutions is to pre-treat food materials before freeze-drying by applying advanced and safe electrophysical techniques instead of traditional thermomechanical methods.

We reviewed three of the most promising electrophysical techniques (low-temperature plasma, ultrasound, and pulsed electric field) which have proven effective for a wide range of food materials. In particular, we focused on their mechanism of action and the equipment required, drawing on successful laboratory and large-scale studies in Russia and abroad.

The electrophysical techniques under review had an etching effect on the material, caused electroporation, and changed the material's internal structure. In addition to these effects, we described their process and technology, as well as their advantages and disadvantages in industrial applications.

Based on literature analysis, we stressed the importance of developing innovative electrophysical techniques for the food industry. These techniques should ensure high energy efficiency of the freeze-drying process and maintain good quality characteristics of food products.

Keywords: Freeze-drying, physical treatment, food product, ultrasound, pulsed electric field, low-temperature plasma

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INTRODUCTION

Russia's food industry prioritizes a transition to highly productive and green agriculture and aquaculture, efficient processing, and formulation of safe and high-quality products, including functional foods [1]. This requires advanced technology for processing, production, and preservation of foods and raw materials [2, 3].

Drying is one of the oldest techniques to preserve food and its vitamins, trace elements, and other macro- and micronutrients. The quality and safety of the resulting product depend on the drying method applied [4]. Freeze-drying is a highly effective method of dehydrating pre-frozen food by sublimating ice in a vacuum under gentle temperature conditions. The resulting product is of much higher quality than that provided by other conventional drying techniques [5].

Energy saving is of major importance in freeze-drying [6]. The duration and high energy costs of freeze-drying are the main technological barriers to its widespread use [5]. Energy saving is a global trend in all processing industries, including food and agriculture (Industry 4.0).

Today, Russian and international research groups are looking for ways to save energy used in the freeze-drying process. Some are developing better facilities, while others focus on various techniques for both food preparation and the process itself [7–11].

Shorstkij described the main technical solutions to improve freeze-drying such as intensified heat supply, better vapor removal, and the recycling of thermal resources, including secondary ones [2]. These solutions also cover the stage of freezing raw materials.

Physical pre-processing techniques allow for energy saving and better quality of the resulting product. For example, the conventional thermomechanical blanching process inactivates enzymes, removes intracellular air, reduces the loss of color and taste, and increases the drying rate [12]. Another technique is osmotic dehydration, which involves introducing a food matrix into a hypertonic solution [13]. The resulting loss of water reduces the subsequent drying time and the amount of dry matter, thus improving the product's sensory and functional properties [14]. However, these thermomechanical techniques provide insufficient energy efficiency, especially during the freeze-drying of berries [15].

In recent years, new electrophysical techniques have been introduced as a pre-treatment before freeze-drying, including ultrasound, pulsed electric fields, and low-temperature plasma. Without using high temperatures, these innovative techniques help reduce the drying time and improve the quality of the resulting product. They are also quite economical due to low energy consumption.

We aimed to review the current uses of innovative physical techniques for freeze-drying food products.

STUDY OBJECTS AND METHODS

In this review, we presented the most effective physical techniques used in the freeze-drying process, briefly describing their mechanism of action, application, and equipment requirements. For this, we retrospectively analyzed scientific papers published in 2010–2024 and indexed in the Scopus, Web of Science, and eLIBRARY.RU databases. The search was based on the keywords “freeze-drying” and “emerging technology”.

RESULTS AND DISCUSSION

Physical techniques in freeze-drying. Low-temperature plasma (LTP) results from gas ionization by an electric discharge using various media (air, argon, oxygen, etc.). The LTP jet generates cations, anions, free and excited electrons, and a number of volatile atoms and molecules [16]. They can be divided into reactive oxygen species and reactive nitrogen species. Their interaction with a food product causes complex physical and chemical reactions. These reactions produce various effects, including sterilization, enzyme inactivation,

surface changes, as well as an effect on the anatomical integrity of plant cells [17, 18]. The variety of active LTP particles indicates their high chemical (including bactericidal) activity, which is why LTP was initially used to sterilize food products.

As technology developed, LTP began to be used to modify the surface structure of raw materials and produce an “etching” effect, which changed the capillary-porous structure of the material [19]. This treatment was later used to prepare seeds for sowing to improve their germination [20].

Surface etching effect. Numerous studies show that the surface effect of low-temperature plasma (LTP) accelerates the freeze-drying of plant materials [21–26]. The mechanism of this effect is shown in Fig. 1. As can be seen, particles of reactive oxygen species and reactive nitrogen species bombard, and interact with, the surface (shell and upper layer) of a food material. The etching effect is mainly due to the decomposition of the material's waxy shell, which causes cracks and channels to form [27]. This waxy shell, commonly present in most foods exposed to freeze-drying (e.g., berries), is a “diffusion barrier” to moisture removal [28]. Exposure to LTP helps remove this barrier and increase drying efficiency. Miraei *et al.* found that the LTP treatment of grapes changed the angle at which water wetted their surface [29]. The etching effect of LTP on the grape's waxy shell increased its hydrophilicity by 40%. This study showed that changing the structure of a plant material's waxy shell leads to higher wettability and moisture absorption. Similarly, Shorstkij and Mounassar reported a rise in moisture absorption when wheat seeds were treated with LTP [30]. In addition, micropores were seen on the surfaces of grapes and wheat seeds, as shown by the scanning electron microscope images. Dharini *et al.* provide more detail about the chemical reactions in the food product's shell [31].

The size of temporary pores formed as a result of LTP treatment may change over time [29]. The size of pores has a direct effect on the efficiency of moisture diffusion onto the surface of the material during freeze-drying. Therefore, further research is needed to maximize the etching effect of LTP. Sosnin and Shorstkij exposed apple slices to LTP, with a pore diameter set at 100 μm [28]. They found that electrically-induced pores

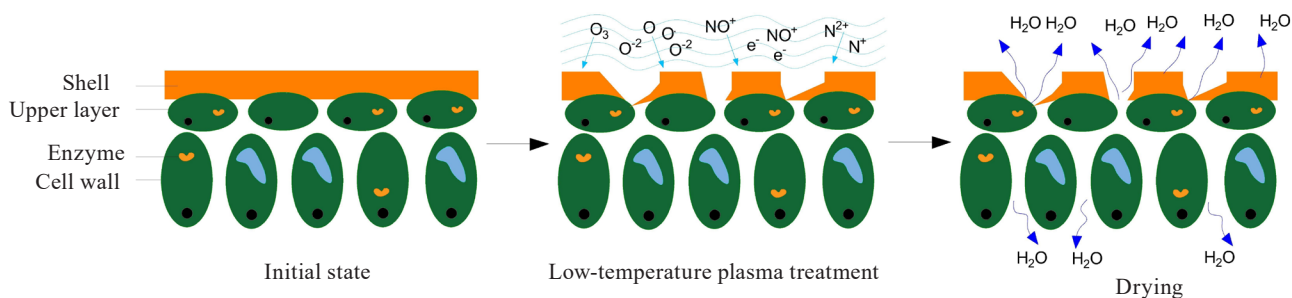


Figure 1 Effect of low-temperature plasma on the surface of plant material: ROS – reactive oxygen species, RNS – reactive nitrogen species

accelerated drying by 15–20%. Similar findings are presented in a study on honeysuckle berries [21]. Also, pores and channels form not only on the surface of fruits but also on other parts of plants, for example, tobacco stem and leaf or common hyssop [32, 33].

The etching effect of LTP treatment directly depends on the material's moisture, as well as the composition and concentration of active substances involved in reactions on the surface of the plant material. Longer treatment and higher moisture significantly increase the number of micropores on the treated surface, which affects the drying time [28]. In other words, the efficiency of etching is one of the main factors for improving the efficiency of freeze-drying.

Changes in the internal structure. As noted above, the surface effects of low-temperature plasma (LTP) can accelerate freeze-drying processes. However, the internal structure of plant materials is modified as well. Since moisture is distributed throughout the volume of the material, it is not enough to only remove the diffuse barrier of the waxy shell. Another barrier to effective mass transfer is the plant cell membrane. There have been few recent studies on improving the drying process by modifying the plant's internal structure [21–26]. Huang *et al.* reported a modified internal structure of grapes, with similar results obtained by Miraei *et al.* using a spark discharge [29, 34].

Li *et al.* reported a high efficiency of treating honeysuckle berries with LTP before freeze-drying [21]. In particular, the LTP pre-treatment for 75 s reduced the freeze-drying time by 27% and increased the permeability of plant cell membranes by 15%. In a study on strawberries, exposure to LTP maintained their quality and improved their sensory characteristics [35]. Yu-Hao *et al.* reported that the cell walls of wolfberry became thinner and more permeable after LTP treatment [36]. Based on the microscopic analysis, the authors assumed that

these changes promoted the release of phenolic compounds through the pores formed after extraction. However, it remains unknown whether this might contribute to a loss of the target component during freeze-drying. Therefore, these changes need to be correlated with varying sizes of the channels formed on the plant's surface and cell membranes. To sum up, changes in the structure of the plant cell membrane are a second factor that affects the efficiency of freeze-drying.

Pectin and cellulose are the main components of a plant cell wall. Based on [37], we can assume that pectin molecules may break down due to the cleavage of the C₄–O covalent bond during oxidation processes induced by LTP. In other words, the LTP treatment of plant materials can promote the breaking of covalent bonds in the cell walls, thereby increasing the rate of water diffusion.

Thus, etching and electroporation of plant cell walls are the main effects of the LTP treatment that have been confirmed by most studies, despite the complexity of LTP's action on multi-component foods. Important factors include the depth of LTP's penetration into the plant structure, the nature of its propagation, as well as the size of pores and channels formed in the material. Saengrayap *et al.* found that the drying efficiency is also determined by the time of the material's relaxation after the treatment [38]. In the study on apple slices, LTP promoted the release of intracellular fluid on the surface of the material, with higher moisture transfer during the drying process [39].

Generation of low-temperature plasma and drying efficiency. The effectiveness of low-temperature plasma (LTP) depends on the method of its generation, among other things (Fig. 2). In particular, LTP can be obtained via a dielectric barrier discharge (two oppositely charged plates), an arc discharge, a microplasma jet, a corona discharge, and a microplasma jet supported by thermionic emission [35, 40].

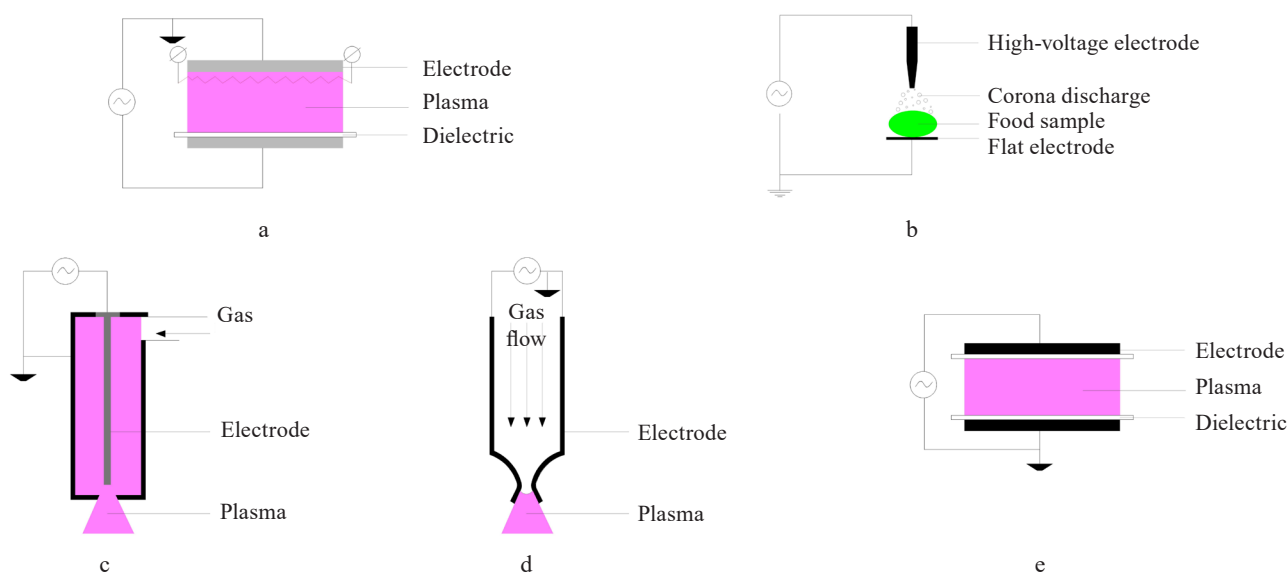


Figure 2 Low-temperature plasma generation schemes: (a) low-temperature plasma supported by thermionic emission; (b) corona discharge; (c) a low-temperature plasma jet with working gas; (d) an arc discharge; and (e) a dielectric barrier discharge

LTP supported by thermionic emission produces a thin string of charged particles that have a directed effect on the object of treatment. Due to thermionic emission, the electric field strength ranges from 6 to 8 kV/cm, in contrast to the range of 15–20 kV/cm for other methods of LTP generation [28]. This method reduces overall energy consumption and provides a better control of the process. The material is hardly heated and the process can be scaled up [28].

With a corona discharge, LTP is generated by using point-plane electrodes set up 3–10 cm apart from each other. Low energy consumption is an obvious advantage of this method. However, the corona discharge does not penetrate into the internal structure of the material, causing only an etching effect on its surface.

A low-temperature plasma jet with pressurized gas supply is the most widely used LTP generation system. The intensity of plasma depends on the generator's power and the flow rate of the gas [41]. Compared to other methods, the plasma jet is more flexible and not limited to a space between the electrodes. Its main disadvantage, however, is a limited impact area and low permeability.

In the dielectric barrier discharge method, the object of treatment is placed between metal electrodes (plane-parallel plates), with at least one of them coated with a dielectric material [42]. To ensure discharge stability, the distance between the two electrodes is limited to a few millimeters, and a high voltage sine wave or switching power supply is required to obtain discharge at atmospheric pressure. The main advantage of this method is that it can be used to treat food materials in dielectric packaging such as polyethylene film. However, given the treatment area of several millimeters, this method can be used mainly for powders.

“Jacob’s Ladder” exemplifies the generation of a traveling arc in the air. When two slightly diverging electrodes are placed vertically and exposed to high-frequency high-voltage power, an arc is first ignited in the narrowest gap between the electrodes and then it slides

along them due to convection [43]. The sliding arc discharge has high energy and gas consumption, with most of the energy used to stimulate chemical reactions rather than heat the gas. Compared to other plasma generating methods, the sliding arc discharge has a larger impact area, which is good for drying [29]. For example, Miraei *et al.* reported that the grapes treated for 50 s at an air flow of 10 L/min had their effective water conductivity at 60°C increased by 30% and their drying time decreased by 26% [29]. However, the devices based on the sliding arc discharge are too complex and sometimes unstable. This disadvantage makes scaling the technology difficult. Yet, this method of generating plasma has high potential in the food industry.

Liu *et al.* showed that treating brown rice with LTP generated by a dielectric barrier discharge had a positive effect on the product’s stability during a short storage period [23]. The treatment maintained the levels of aldehydes and alkanes and increased the content of volatile substances. In another study, LTP pre-treatment (200 and 800 Hz) improved the safety of tucum fruits during storage, causing changes in their tissue structure and improving dehydration [24].

Zhang *et al.* observed no significant changes in the color of red pepper depending on the time of LTP treatment [26]. Yet, the treatment of 30 s improved the recovery of color pigments during further processing. Table 1 summarizes the effects of pre-treating plant materials with LTP on the efficiency of their freeze-drying. As can be seen, most studies aim to assess the energy efficiency of the freeze-drying process with LTP pre-treatment. For example, Miraei *et al.* reported electrically induced pores on the surface of grapes, with no numerical correlations [29].

Industrial application of low-temperature plasma for freeze-drying. Figure 3 shows a tray-type commercial unit for pre-treating raw materials with low-temperature plasma (LTP) created by the Kuban State University of Technology in partnership with manufacturing companies [45].

Table 1 Effects of low-temperature plasma pre-treatment of food materials on the efficiency of their freeze-drying

Material and mode of treatment	Treatment efficiency	References
Honeysuckle Low-temperature plasma jet in the reactor	Drying rate increased by 27%, cell permeability increased by 15%	[21]
Strawberry Low-temperature plasma jet in the reactor (3 kV, 1 MHz)	Enhanced aroma at zero/average pressure	[22]
Brown rice Low-temperature plasma jet (30–100 kV)	Free fatty acids reduced by 25.2%	[23]
Tucum Corona discharge (200, 500, and 800 Hz)	Higher drying and rehydration rates, more phenolic compounds retained at 500 and 800 Hz	[24]
Mushrooms Corona discharge	Higher drying rate, accelerated mass transfer, the largest number of phenolic compounds preserved (463.30 mg/100 g)	[25]
Chili pepper Plasma flow (3 L/min, 20 kHz, 750 W, treatment time: 15, 30, 45, and 60 s)	Higher drying rate; longer treatment may result in pigment loss due to degradation of bioactive compounds	[26]
Plums	Drying time reduced by 5–6 h	[44]



Figure 3 A commercial unit for low-temperature plasma treatment based at Kuban State Technological University



Figure 4 An industrial machine for low-temperature plasma treatment of foods for sterilization [47]: 1 – a discharge chamber, 2 – an electrode unit, 3 – a control panel, 4 – an ozonation chamber, 5 – a belt conveyor

This LTP unit can treat up to 100 kg of raw materials per hour, with their subsequent transfer to the freeze-drier. Recent years have seen a steady rise in the number of patents on the use of LTP in the food industry [46]. For example, Pańka *et al.* presented a pilot machine based on a belt conveyor to treat food products (such as ground spices) with LTP for sterilization [47]. This unit can be easily adapted for treatment prior to freeze-drying (Fig. 4).

Advantages and disadvantages of low-temperature plasma. The main advantages of treating plant materials with low-temperature plasma (LTP) to improve the efficiency of their drying are as follows:

1. LTP treatment reduces the diffusion barrier of the food's shell and produces an etching effect that facilitates subsequent freeze-drying.
2. The product retains high sensory characteristics because LTP treatment reduces the drying time.
3. Various methods of generating LTP to treat materials under atmospheric pressure reduce the cost of processing (no need for vacuum units) and allow for conveyor-type processing with a continuous flow of materials.

However, there are a few downsides to LTP application, namely:

1. LTP equipment used for drying is expensive and unstable. The cost of applying LTP to dry food products

starts from 50 000 US dollars [17]. There is a lack of commercial LTP equipment.

2. Scientists do not have a full understanding of how pre-treating raw materials with LTP affects the properties of the final product. Drying efficiency varies greatly depending on different parameters. Finding the optimal dose of LTP and controlling the pretreatment process are still challenging.

3. LTP has a low penetrating ability. This limits its application to mainly overcoming the surface diffusion barrier.

4. The quality of LTP treatment depends on the surface of the material. If the surface is not homogeneous and has some irregularities or protrusions, some areas may remain untreated.

To introduce LTP treatment into commercial food technologies, we need combined efforts of interdisciplinary scientists, manufacturers, and suppliers of agricultural products. This can also be helped by advanced research and equipment design.

Ultrasonic treatment. Effect of ultrasonic treatment on the structure of plant materials. Ultrasonic treatment is a technology for non-thermal processing of the food matrix that is used in a wide range of processes. Ultrasound propagates due to a series of compression and extension cycles induced by an ultrasonic wave at a frequency from 2×10^4 to 1×10^9 Hz. Cavitation bubbles form when the power of an ultrasonic wave exceeds the power of attraction between the molecules in a medium [48]. When the natural frequency of vibration of the bubbles coincides with the frequency of the ultrasonic wave, the bubbles collapse quickly at the compression stage and release energy with a subsequent increase in pressure (Fig. 5). High pressure from the collapse of the bubbles, which can reach 100 MPa, can rupture the cell membranes and damage the cell microstructure [49, 50]. This process may also cause water molecules to dissociate and free radicals (H^+ and OH^-) to form. These free radicals are capable of reacting with other molecules.

The above effects of ultrasonic treatment can cause a number of physical and chemical effects that can be used to prepare food materials for freeze-drying. Unlike low-temperature plasma (LTP), ultrasonic treatment can affect the size of ice crystals during the freezing stage. This accelerates the freezing rate and, subsequently, reduces the total duration of freeze-drying [50].

Effects of ultrasonic treatment on freeze-drying. Ultrasonic treatment was used to accelerate the freeze-drying of bell pepper in [51]. The authors found that ultrasonic water removal required lower temperatures and shorter time. In particular, ultrasonic treatment lasting 10% of the total freeze-drying time reduced the drying time by 11.5%, ensuring a high-quality product. The findings were based on the bulk density, color characteristics, ascorbic acid content, and rehydration parameters.

In another study, the ultrasonic treatment of strawberry slices decreased their drying time by 15.25–50.00%, compared to the untreated samples [52]. Semenov *et al.* reported that applying mechanical vibrations was as ef-

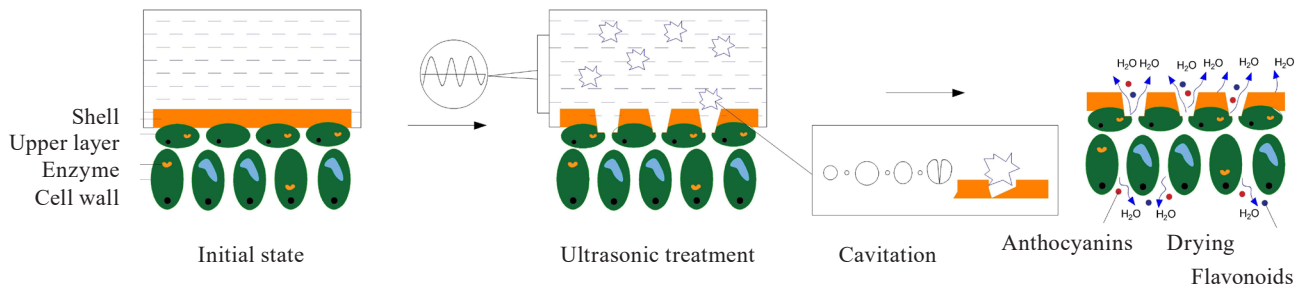


Figure 5 The process of ultrasonic treatment

Table 2 Effects of ultrasonic pre-treatment of food materials on the efficiency of their freeze-drying

Material and mode of treatment	Treatment efficiency	References
Red beetroot 40 kHz, 180 W, 5–10 min	Lower water activity, greater shrinkage of the resulting product	[55]
Barley tops 10, 30, 45, and 60 W/L, 10 min	Energy consumption reduced by 19%	[49]
Pear 20 kHz, 360 and 960 W	Moisture content reduced to 0.12–0.08 g/g dry material	[56]
Bell pepper 76, 90, and 110 W	Total drying time reduced by 11.5%, the product's color preserved	[51]
Hawthorn 37 kHz, 70 W, 20 min	Shorter drying time	[57]
Potato 30 kHz, 200, 400, and 600 W, 30 min	Drying time reduced by 7.2–17.8%, energy consumption reduced by 12–18%, taste and nutritional properties preserved	[58]
Melon 25 kHz, intensity 4870 W/m ²	Overall carotenoid loss reduced, softer texture, and better color retention	[59]
Carrot 25 kHz, 41 W/L, 30–60 min	Drying time reduced due to cell destruction, microchannel formation, and cell swelling	[60]
Apples 400 W/L, 10 min	Internal water diffusion increased by 93%, external mass transfer increased by 10%	[61]
Strawberry 20 and 40 kHz	Drying time reduced by 15.25–50.00%	[52]
Apples 1 kW, 40 kHz, 26–40°C	Minimum moisture (5% by weight), rehydration rate increased by 45%	[62]
Rowan Piezoelectric emitter, 8 kW	Drying time reduced 1.28 times	[63]

efficient as ultrasonic treatment [53]. They also found that ultrasound treatment at 20 and 40 kHz reduced the drying time more greatly than at a single frequency. This most likely indicated the collapse of bubbles of various sizes. Finally, the ultrasonically treated samples had better quality indicators, including vitamin C content, rehydration parameters, density, color, aroma, total anthocyanins and phenols, as well as antioxidant activity.

According to [54], the average rate of freeze-drying was higher for ultrasonically pre-treated apple samples than for the control group. In particular, the pre-treatment at 100 kHz for 5 min at 25°C increased the average drying rate 1.25 times, compared to the control. This might be because ultrasound destroys cell membranes, increasing their permeability to water. As a result, moisture is more easily removed from the cells, accelerating the freeze-drying process.

Table 2 summarizes the effects of ultrasonic pretreatment on the efficiency of freeze-drying of food materials.

To improve the penetration of ultrasonic radiation into the product, the wave resistance needs to be reduced at the medium-product interface. For this purpose, ultrasonic pre-treatment is often carried out in a liquid medium (distilled water or osmotic solution). As a result, the product may lose or gain water during ultrasonic pre-treatment depending on the direction of the concentration gradient at the interface. For example, Oliveira *et al.* found that apples absorbed water from the external medium during their ultrasonic pre-treatment in distilled water and an osmotic solution [64]. When sugar was added, water absorption decreased. This was because sugar increased the resistance to moisture transfer on the surface of the apple. Therefore, the medium parameters are just as important as the frequency, duration, and power of ultrasonic treatment.

Industrial application of ultrasonic treatment for freeze-drying. Continuous-flow ultrasonic treatment is mainly applied to liquid and paste-like materials. Ultra-

sonic treatment is carried out directly in the working chamber or in an ultrasonic bath of a freeze-drying equipment [65, 66].

The industrial application of ultrasonic treatment is described in [67]. The apparatus consists of a stationary ultrasonic bath supplied with all necessary utilities (Fig. 6).

Huang *et al.* combined ultrasonic treatment with the drying process [68]. According to the authors, the formation of a pressure gradient at the gas/liquid phase interface during drying intensified moisture evaporation. As a result, the water went out of the sample without coming back during the positive pressure phase. However, ultrasonic treatment cannot be combined with freeze-drying, since this would require a medium conducting acoustic waves. Vacuum-free freeze-drying can be an alternative to this method [69].

Advantages and disadvantages of ultrasonic treatment. The main advantages of ultrasonic treatment of plant materials are as follows:

1. Ultrasound pre-treatment greatly aids the freeze-drying technology.



Figure 6 An industrial ultrasonic treatment apparatus for food sterilization [67]

2. Ultrasonic waves activate the rate of heat and mass transfer, reducing the drying time.

3. The cost of ultrasound pre-treatment equipment starts from 10 000 US dollars, which is significantly lower than the cost of low-temperature plasma machinery.

However, there are a few downsides to ultrasonic treatment, namely:

1. To reduce wave resistance, ultrasonic treatment should be carried out in a liquid medium. However, this may change the composition of the freeze-dried product since enlarged pores ease the release of the product's target components into the liquid medium, causing their loss.

2. Despite its relatively low cost, ultrasonic treatment is not widely used in the food industry due to several technological challenges. For example, it is important to set the optimal frequency and such medium characteristics as temperature, pH, electrical conductivity, and others. Moreover, the treatment can accidentally destroy the shell of the freeze-dried product, reducing its consumer appeal.

Pulsed electric field treatment. Effects of pulsed electric field on the structure of plant material.

Pulsed electric field (PEF) treatment is an application of short, high-voltage electrical pulses to a liquid or solid product placed between two electrodes in a conductive medium. During this treatment, the cell membrane becomes polarized, which makes the cell wall more permeable. The cell wall can even rupture at certain levels of electric field strength [70]. This mechanism is known as electroporation (Fig. 7). When exposed to an electrical impulse, hydrophilic channels are formed in the lipid bilayer of the cell membrane. Moving along the electric field lines to the membrane boundary, polarized molecules create their internal electrical potential on both sides. The membrane ruptures when the electrical potential reaches a critical level. Thus, this method can enhance mass and heat transfer without causing undesirable changes in the food quality [71]. The electrical conductivity of membranes, as well as pore formation, depends on the critical strength of the electric field (1–2 kV/cm), the size of

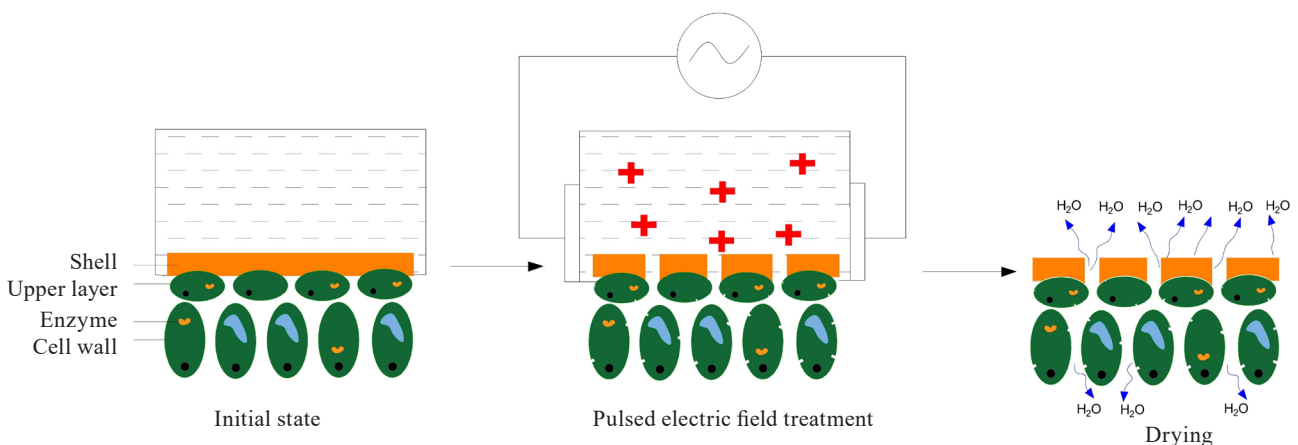


Figure 7 The process of pulsed electric field treatment

a treated cell (40–200 μm), and its electrical characteristics [72]. Electroporation does not occur if the electric field strength and pulse duration are less than critical. Temporary (reversible) pores may form if the electric field strength is close to the critical level, but does not exceed it. In this case, the electrical conductivity of the membranes may change but only temporarily, with a possibility of returning to the original state [73]. This method is used in medicine to introduce target components into the cell. Food scientists, however, are more interested in electroporation caused by the field strength. If the field strength is higher than the critical level, with high specific energy costs, electroporation can become irreversible and the formed electrical pores can cause cell destruction.

The critical levels of electric field strength vary greatly, depending on the material's tissue structure, the size of its cells, and their electrical characteristics [74].

Pulsed electric field treatment for freeze-drying of food products. In a research by Lammerskitten *et al.*, freeze-dried strawberry cubes pre-treated with pulsed electric field (PEF) better retained their shape and volume, as well as had a dense and compact structure [75]. In another study, PEF treatment significantly improved the freeze-drying process due to better mass transfer from the electroporation effect [76]. PEF pre-treatment destroyed the anatomical integrity of cell membranes and accelerated further moisture transfer [77]. Importantly, PEF treatment is performed throughout the material, which promotes further scaling.

Table 3 shows the effects of pre-treating food materials with PEF on the efficiency of freeze-drying. As can be seen, in addition to lower energy consumption due to a shorter drying time, PEF treatment can im-

prove the textural characteristics and quality indicators of the resulting product [76]. The extent of shrinkage during freeze-drying and the preservation of useful target components are important for consumer acceptance. Lammerskitten *et al.* reported that PEF treatment preserved the shape and color of the resulting product and contributed to an even distribution of sugar and moisture inside the material [78]. This had a significant impact on the shock freezing stage, since the channels formed were not clogged with sugar. Witrowa-Rajchert and Lewicki noted an improved reducing ability of plant raw materials after PEF treatment and freeze-drying [79]. This confirms that PEF treatment causes changes in the internal structure of the material.

However, the external product evaluation revealed lower shrinkage of the PEF-treated material. What still needs exploring is the relation between PEF's uniform effect on the internal structure of the material and its shrinkage during freeze-drying. For example, Parniakov *et al.* found that PEF-treated samples retained lower temperature in their core for a longer time, compared to untreated samples [80]. This means that frozen water inside the sample retains its original shape, which impairs further heat and mass transfer processes.

Industrial application of pulsed electric field for freeze-drying. According to Google Scholar, there are over 15 000 publications on pulsed electric field (PEF) treatment and over 55 research groups exploring this technology [85]. However, its industrial application has only become possible recently thanks to the advancement of reliable electronics and high-voltage equipment. In particular, this treatment is mainly used in the production of juices (1500 L/h, PulseMaster, the Nether-

Table 3 Effects of pulsed electric field pre-treatment of food raw materials on the efficiency of freeze-drying

Material and mode of treatment	Treatment efficiency	References
Strawberry Unipolar pulses with an interval of 0.5 s, pulse duration 40 μs ; $E = 1 \text{ kV/cm}$	Preserved shape, color, and taste; more porous structure with evenly distributed pores; greater crunchiness	[75]
Strawberry, bell pepper $E = 1.0 \text{ kV/cm}$; specific energy 0.3–6.0 kJ/kg, treatment time 2.0–28.6 ms	Reduced shrinkage, increased rehydration, improved mass transfer	[76]
Apples $E = 800 \text{ V/cm}$	Drying time reduced, pore size increased to 86 μm , rehydration capacity increased by 1.3 times, shape preserved	[80]
Apples $E = 0.3, 0.6, 0.9, \text{ and } 1.2 \text{ kV/cm}$; 5, 10 or 15 pulses	Changes in the integrity of the cellular structure, reduced content of water suitable for freezing	[81]
Apples $E = 1.07 \text{ kV/cm}$, specific energy 0.5, 1, and 5 kJ/kg	Reduced shrinkage, preserved shape, improved porosity, greater retention of phenols, antioxidant activity reduced by 60%	[78]
Red bell pepper 1 and 3 kJ/kg, $E = 1.07 \text{ kV/cm}$	Drying time reduced by 70 %, appearance preserved	[78]
Apples $E = 1000, 1250, \text{ and } 1500 \text{ V/cm}^{-1}$	Energy consumption reduced by 17.74%, freeze-drying time reduced by 22.50%, productivity per unit area increased by 28.50%	[82]
Potato $E = 0.2\text{--}1.1 \text{ kV/cm}$; pulse duration 20 μs , specific energy 1–10 kJ/kg	Uneven changes in cell viability and microstructure, increased cell rupture at a constant frequency and higher electric field strength	[83]
Potato $E = 600 \text{ V/cm}$; total treatment time $t_{\text{PEF}} = 0.1 \text{ s}$	Drying time reduced by 22–27% at $T_d = 40\text{--}70^\circ\text{C}$, significant changes in the texture and microstructure	[84]

lands), French fries (50 t/h, Elea, Germany), and milk (350 L/h, EnergyPulse) [86].

The application of PEF to plant materials (apples, potatoes, red peppers, etc.) during drying processes started in the early 2000s [75–77]. Since then, its efficiency has been experimentally confirmed by many studies. PEF is industrially applied in Germany, Italy, the USA, and some other countries. High-performance PEF apparatuses have been developed for potato processing and juice production [87]. Figure 8 shows one of such systems for preparing various food materials (such as berries and fruits) for freeze-drying. The apparatus has a belt conveyor passing through a bath with a medium where an electrode unit is installed. PEF pre-treatment significantly reduces undesirable shrinkage of plant materials. This improves the rehydration ability of the freeze-dried product, which is especially important for instant soups or whole berries [80, 88].

Advantages and disadvantages of pulsed electric field treatment. The main advantages of pulsed electric field (PEF) treatment of plant materials are as follows:

1. PEF treatment occurs throughout the material, which is why both whole and sliced products can be treated.
2. The use of cooling systems makes up for increasing product temperatures during PEF treatment.
3. The size of channels formed on the surface of plant cell membranes can be controlled by changing PEF's electrophysical characteristics.

However, PEF has a few downsides and technological challenges, namely:



Figure 8 An industrial pulsed electric field apparatus for pre-treatment of solid materials (Elea, Germany) [89]

1. PEF treatment requires a conductive medium, which is usually a liquid electrolyte.
2. PEF treatment consumes a lot of energy due to the use of high-ampere Marx generators.
3. The use of high voltage and high currents during PEF treatment requires more effective protection of equipment and personnel.
4. The liquid medium used for PEF treatment may erode the metal electrodes, with metal traces entering the final product.

Although the cost of a PEF system starts from 200 000 US dollars (10 t/h), this technology boasts wider commercial application than low-temperature plasma and ultrasonic treatment systems described above. Quite a few companies manufacture PEF systems for industrial use.

Promising areas of research. Industry 4.0, or the fourth industrial revolution, and the emerging Industry 5.0 require that we move away from the traditional thermomechanical processing systems and look for alternative techniques. They include autonomous intelligent systems that make use of advanced robotics and smart technologies at all stages of the food supply chain [90]. In addition, we need to ensure the safety of nutritious food products, low energy consumption, and environmentally clean production.

Introducing advanced electrophysical techniques into the food production chain is a promising area for drying technologies (Fig. 9). This approach involves a synergistic effect which can help produce sufficient amounts of high-quality freeze-dried products.

In this paper, we discussed three innovative physical techniques that can be applied to prepare materials for freeze-drying processes, namely low-temperature plasma, ultrasonic treatment, and pulsed electric field. Before these techniques can be used on a wider scale, we need to address a few technological challenges.

For low-temperature plasma to be introduced widely, we need to:

1. understand how chemically active plasma particles interact with, and affect, individual food components (such as lipids, proteins, carbohydrates, etc.);
2. optimize ways of generating low-temperature plasma in order to reduce overall energy costs; develop atmospheric treatment without the use of vacuum; unify methods for calculating energy indicators; and
3. analyze the effect of low-temperature plasma on the size of pores and channels so that this technique can be used to produce products with a desirable appearance.

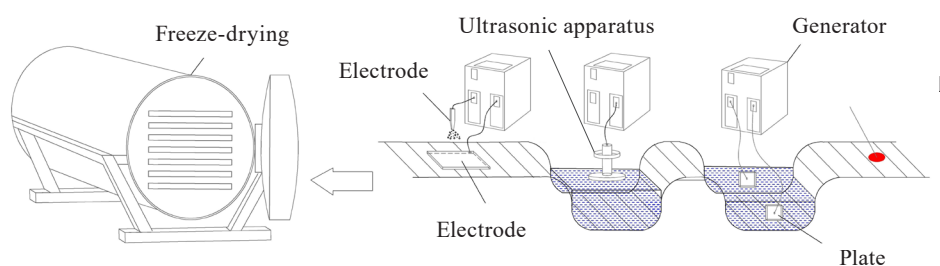


Figure 9 Preparation of food raw materials for freeze-drying within Industry 4.0

To promote ultrasonic pre-treatment, it is important to:

1. establish ranges of ultrasonic frequencies for materials that differ in moisture, size, and composition, as well as for different treatment media;
2. optimize ways of ultrasonic treatment for continuously supplied raw materials; and
3. develop mechanisms to reduce the loss of target components after ultrasonic treatment and further freeze-drying.

For pulsed electric field to be applied more widely, we need to:

1. optimize energy costs in order to increase the efficiency of freeze-drying; determine the effects of pulsed electric field treatment on the freezing stage, where moisture distribution throughout the material is an important factor;
2. reduce the cost of pulsed electric field equipment, as well as search for analogues of the Marx generator for generating high voltage pulses.

CONCLUSION

Vacuum freeze-drying is one of the most effective and one of the most expensive methods of food preservation. Low-temperature plasma, ultrasound, and pulsed electric field are innovative physical techniques that can

be used to pre-treat raw materials before freeze-drying. These techniques can significantly reduce the drying time and enhance the quality of freeze-dried products.

In particular, they can improve heat and mass transfers during the drying process by causing an etching effect and electroporation. They also help preserve the quality characteristics of freeze-dried products. However, pulsed electric field and low-temperature plasma are quite expensive techniques, while ultrasonic treatment needs to be optimized for continuously supplied raw materials.

Freeze-drying is an energy-intensive process, although numerous studies have sought to speed it up while preserving the nutritional and sensory properties of the final products. Therefore, further research is needed to optimize pretreatment conditions for different types of raw materials.

CONTRIBUTION

O.I. Andreeva collected information for the review and designed the graphics. I.A. Shorstkii collected additional information and wrote the manuscript.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interest.

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ORCID IDs

Oksana I. Andreeva  <https://orcid.org/0009-0008-4265-9651>

Ivan A. Shorstkii  <https://orcid.org/0000-0001-5804-7950>