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Synergistic and sustainable approaches for pre-treatment, drying and storage of food waste to preserve nutritional and sensory quality in functional food upcycling.

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Summary

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Thesis Overview and General Aim

The growing focus on healthy and sustainable nutrition has made the fight against food waste a priority, stimulating the search for technological solutions that combine nutritional quality, safety, and reduced environmental impact. In this context, this thesis explores synergistic strategies for the pre-treatment, drying, and storage of food waste and by-products, with the aim of transforming them into high-value functional ingredients.

The first contribution examines apricots subjected to pulsed electric fields and airborne ultrasound, yielding the diffusional parameters needed to optimise hot-air drying. The next two studies focus on whole or sliced fruit, blood oranges and white-fleshed peaches, showing how time/temperature choices and modified-atmosphere packaging preserve colour, aroma and microbiological safety well beyond processing. The fourth paper investigates tray-drying of Sicilian mango, systematically analysing how the ripening stage (green vs fully mature) affects drying behaviour and subsequent MAP storage, ultimately producing stable snacks rich in bioactive compounds. The final two articles move beyond the notion of a mere "finished product". Ultrasound-assisted blackberry powders enrich functional cookies, while mango-peel flour becomes an ingredient in sourdough bread with enhanced nutritional value.

Woven together, these empirical threads pursue a clear overarching aim: to identify, through an integrated programme of experimentation and modelling, the combinations of non-thermal pre-treatments and drying techniques that deliver dehydrated foods of high nutritional quality, microbiological safety and reduced environmental footprint. At the same time, the thesis seeks to demonstrate how dehydrated matrices can be intelligently employed to create innovative functional foods, thereby contributing to waste reduction along the supply chain. Without pre-empting the general conclusions reserved for the final chapter, the results already obtained indicate that integrating "mild technologies" is a promising route to shorter processing times, lower energy demands, better preservation of sensitive nutrients and new market opportunities for currently undervalued raw materials.

CHAPTER 1

1. Introduction

Food sector continues to develop, with individuals requiring increasingly more solutions that meet the modern-day issues of environmental sustainability, food security, and effectiveness of food preservation technologies [1,2]. Nowadays, as the world population increases significantly, access to healthy and safe food is an essential priority. Food production, indeed, will need to face many challenges, among which the minimization of transport and storage costs, enhancement of shelf-life of the products and better management of natural resources. In this condition, food dehydration emerges as one of the most prevalent and established techniques since it has the potential to increase the shelf life of food as well as minimize wastage and ensure good nutritional quality [3]. It is a foundation technology in the processing of the wide array of products, including fruits, vegetables, meat, and bakery products [4]. The primary benefits are the reduction of food's size and weight, thereby making it easier to transport, and the retention of a great deal of nutrients. But the increasing awareness of sustainability has been responsible for looking towards alternative and sustainable techniques. These techniques minimize the use of energy, enhance the quality in terms of nutrition and sensory properties in finished products, and restrict the negative impact on the environment [5–7]. This has been responsible for creating innovative technology for drying like microwave drying, reverse osmosis, vacuum drying, the application of ultrasound (US), and pulsed electric fields (PEF) [3,6,7]. These methods, typically used as pre-treatments, enable a more efficient process, shorter processing time, better retention of bioactive substances, and minimal change in the sensory qualities of food [1,4,6]. The use of ultrasound as a pre-treatment is of interest since it enables moisture to diffuse and alters the structure of plant tissue [8]. This makes the material porous and releases water. As a result, one can obtain a final product that is microbiologically more stable and has better nutritional quality compared to traditional heat treatment. MAP (Modified Atmosphere Packaging) technology has been helpful in extending the shelf life of dried foods, making the colour, odour, and texture better over time [9–11].

Environmental sustainability is one of the priority targets in the food industry today. The food sector accounts for a big share of CO_2 emissions worldwide, with production and preservation being major causes. In this context, minimizing energy consumption involved in drying food is a priority [1,2,12]. The use of technologies that consume less energy, for instance, low-temperature or those that use vacuum and inert gases, helps to lower the impact

on the environment without affecting the quality of the products. Another underlying theme is the preservation of nutritional value of dehydrated foods. Their preparation through high temperatures can damage extremely sensitive substances such as vitamin C and some vitamins of the B group and can even cause the production of unwanted or toxic substances [13]. New technologies, on the other hand, tend to keep intact the main nutrients and minimize the chemical and physical changes that can make it less healthy and less appetizing [4]. Dehydration also plays a huge role in reducing food wastage. Every year, almost one-third of all food produced globally is either wasted or lost [14–16]. A lot of this wastage comes from fresh produce that does not meet aesthetic standards but is still safe for consumption. In this area, dehydration is a great technique for using fruits and vegetables that are misshapen, damaged, or in excess [12]. These can be turned into useful ingredients for new food products. The growing demand for the functional foods has also made drying more vital as a technology. Demand for healthy snacks, supplements, and ready meals that are rich in bioactive materials (for example antioxidants, vitamins, fibre, and minerals) has prompted researchers to come up with technologies that can maintain the stability and integrity of the same type of compounds [13]. Dehydrated products are thus not only longer lasting, but also nutritionally valuable to the modern consumer.

At the same time, policy that promotes sustainable behaviour is also being promoted. Legislation on food safety and the growing demand for quality of processed food urge the sector to invest in technology that can deliver high levels of hygiene and health. New techniques, such as radiation treatments or pulsed electric fields, are being investigated for their ability to inactivate pathogens without affecting the nutritional quality of the products [7,17]. Taking all these into account, the dehydration business is facing a sea change. Traditional technologies, while still common even now, are being replaced by more modern technologies that could incorporate energy efficiency, food safety, nutritional value, and environmental sustainability. This adjustment is a reaction to the growing worldwide demand for sustainable and robust food systems. These food systems can provide nutritious and safe food without degrading the world's resources.

1.1 Evolution of Consumer Trends and Market Drivers

In recent years, there has been a marked change in consumer eating habits and expectations. More people are looking for natural foods that are free from artificial additives, healthy, and have a low environmental impact [18]. At the same time, there is growing attention to sustainability and transparency in food production: consumers want to know not only what is in their food, but also how it was made. A recent survey found that as many as 94% of

consumers believe it is important for food producers and brands to be transparent about ingredients and production processes [19]. This clear demand for trust and clarity has prompted companies to rethink their production practices, favouring recognizable ingredients and "clean label" methods, i.e., simple processes without unnecessary chemicals [20,21]. Numerous studies confirm that health and sustainability trends are leading consumers to examine the ingredients in everyday food products more closely [22–25]. As a result, many consumers are willing to choose organic or certified products, which are perceived as being better for their health and the environment [26]. This scenario outlines a more aware and informed modern consumer, whose preferences are forcing the food industry to take greater responsibility throughout the entire supply chain.

In response to these new requirements, the use of physical and natural preservation methods, including food dehydration, is being reevaluated. Unlike continuous refrigeration or the addition of chemical preservatives, dehydration preserves food by drastically reducing its water content and maintaining much of its original nutritional [27]. Removing water inhibits microorganisms and slows down degradation reactions, effectively extending the shelf life of products without the need for additives [28]. Although sometimes involving some loss of heat-sensitive vitamins, the drying process concentrates many nutrients: for example, dehydrated leafy vegetables remain a concentrated source of useful minerals and micronutrients, despite the partial reduction of heat-sensitive vitamins [29]. An additional advantage is that preservation by drying does not require a cold chain, reducing energy requirements during storage and transport compared to refrigerated products. Furthermore, by removing water, foods become lighter and more compact, with benefits throughout distribution: costs and impacts related to packaging, handling, and transport are lower [27].

In tandem with consumer trends, the global market for dehydrated foods is experiencing steady growth. Industry analyses indicate compound annual growth rates of around 6–7% for natural or "clean label" food products, in line with the expansion forecast for the dehydrated foods sector through 2030 [30]. This dynamic is supported by several market drivers. First, the global popularity of ready meals and functional foods has created strong demand for practical yet nutritious solutions. New urban lifestyles, which are fast-paced and convenience-oriented, are driving demand for foods that save time without compromising on quality. In this context, dehydrated foods are particularly well suited to meeting emerging needs: they offer long shelf life, ease of use, and preservation of nutritional values. For example, it has been shown that dried fruit snacks can be a healthy alternative to snacks high in sugar or fat, providing fibre and bioactive compounds that are beneficial to health [31].

Similarly, dehydrated foods contribute to a more balanced diet, facilitating access to fruit and vegetables out of season or in contexts where fresh produce is less available. The growth of the sector is therefore fuelled by a convergence of nutritional (health consciousness), practical (convenience), and socio-demographic (accelerated pace of life) factors, which make dried products an innovative and welcome solution for contemporary consumers.

Market trends are not the only driver of change: regulatory pressures and global sustainability commitments are pushing food companies toward more environmentally friendly practices, particularly in waste and waste management. Both international bodies and national legislatures are setting ambitious targets (such as reducing food waste by 50% by 2030, in line with the UN Sustainable Development Goals [32]), creating an environment in which companies are incentivized to adopt more sustainable strategies throughout the supply chain. In addition to regulations, there is a growing awareness among consumers themselves: reducing waste has never been so important, thanks in part to more attentive and informed consumers [33]. In this perspective, dehydration emerges as a valuable ally for sustainability in the food sector. Drying makes it possible to make use of food surpluses and waste, converting products that would otherwise be unmarketable (production surpluses, 'ugly' fruit that does not meet standards, processing scraps) into long-life ingredients. For example, by transforming overripe fruit into dried purees or powders, or unsold vegetables into dehydrated mixes, the shelf life of these foods is extended, and premature disposal is avoided. Studies on food waste upcycling report numerous success stories: for example, some restaurants have introduced dishes that use dried vegetable peels as garnishes or snacks (transforming what was waste into something valuable), maintaining taste and quality and significantly reducing waste [34–36]. Reducing water also reduces weight and perishability, which limits losses along the supply chain (fewer products discarded due to expiry or damage) and makes transport more efficient. Overall, the use of dehydration as a circular economy tool offers tangible environmental benefits: it reduces organic waste to be disposed of, recovers nutrients from by-products, and helps conserve finite resources (energy, soil, water) through better utilization of what has already been produced [32].

Innovation in the food industry is focusing on the development of products and solutions that combine convenience, health, and sustainability. There has been a proliferation of new dehydrated foods designed for specific consumer segments. These include healthy snacks such as dried fruit and vegetable chips, freeze-dried or dehydrated instant soups (to which you simply add water to obtain a hot meal), dried fruit and nuts in energy mixes for athletes, and dehydrated ingredients for catering (from spices to aromatic herbs, mushrooms to semi-

finished products) that facilitate the preparation of dishes. These solutions respond to a common need: to have quality, safe, and long-lasting foods ready for use, reducing time in the kitchen without compromising nutritional value. An additional advantage of many new-generation dehydrated products is their low environmental footprint. Thanks to their reduced weight and stability at room temperature, their transport and storage involve lower energy and material consumption (no continuous refrigeration, lighter packaging) [27].

Furthermore, many of these products are made from local raw materials or recovered surpluses, reducing supply distances and implementing circular economy models. Take, for example, packs of dried apple snacks made from surplus batches of fresh apples: they offer consumers a healthy snack while reducing waste upstream. Together, these innovations position dehydration not only as an effective preservation technology, but as a concrete act of sustainability in the face of current challenges. Minimizing waste, increasing the sustainability of supply chains, maintaining the nutritional quality of food, and responding to increasingly responsible demand are all goals that the industry is pursuing through newly designed dehydrated products, a sign of a structural transformation driven by consumer preferences and the needs of the planet.

1.2 Environmental Sustainability Challenges (Food Waste, Carbon Footprint)

The agri-food sector plays a key role in the global climate crisis. Every stage of the food supply chain, from primary production in agriculture, through industrial processing and distribution, to waste disposal, contributes significantly to greenhouse gas (GHG) emissions. Current estimates indicate that the global food system is responsible for about one-third of total anthropogenic GHG emissions [37]. At the same time, about one-third of all food produced worldwide each year is lost or wasted along the supply chain before it is consumed [38]. This involves not only huge economic losses, but also significant environmental impacts: food produced but not eaten means wasting natural resources (land, water, energy) and unnecessary emissions linked to the decomposition and management of that organic waste. In fact, food waste alone is responsible for around 8–10% of global greenhouse gas emissions and uses almost a third of the world's agricultural land without feeding the population, also contributing to the loss of biodiversity [39]. In view of the need to mitigate climate change, the agri-food system is therefore one of the most critical areas for action, as it accounts for a significant proportion of global emissions [40]. Reducing food loss and waste along the supply chain and improving the efficiency of production processes is an internationally recognized strategic priority for reducing the carbon footprint of the food sector.

In this context, food dehydration emerges as a technological approach aimed at reducing waste and minimizing the environmental impact of the agri-food industry. By removing most of the water from products, drying processes drastically reduce the weight and volume of food, while extending its shelf life, often without the need for refrigeration [41]. This reduces energy consumption along the logistics chain: dried and stabilized products can be stored and transported more easily, with lighter loads (weight can be reduced by 70–90% through freeze-drying) and less use of refrigerated vehicles. An additional advantage is the elimination of the need for synthetic preservatives, in line with growing consumer demand for minimally processed foods free of chemical preservatives. Proper drying preserves many of the organoleptic and nutritional properties of food, especially through gentle techniques such as freeze-drying, which preserve structure, flavour, and sensitive nutrients such as vitamins and bioactive compounds [42]. In summary, converting fresh food into dried food extends its availability in terms of time and space (thanks to long-term storage without refrigeration), reducing waste and energy requirements for distribution, and improving the sustainability profile of the entire supply chain.

From an ecological point of view, one of the most tangible benefits of dehydration lies in the valorisation of surplus or waste fruit and vegetables, for example, fruit and vegetables that are out of size or aesthetically imperfect, which would otherwise be discarded. Through drying or freeze-drying, these foods can be transformed into long-life, value-added products that can be used as ingredients in other food preparations instead of ending up in landfill. This conversion of potential waste into edible resources helps reduce pressure on disposal systems (by limiting landfill waste) and mitigate climate-changing emissions: it avoids methane emissions resulting from the anaerobic decomposition of organic waste and optimizes the use of resources already used in agriculture (land, water, fertilizers, energy), improving the overall efficiency of the production system. In other words, drying allows food that would otherwise be lost to be recovered, with both environmental and socio-economic benefits (reduction of waste, new income opportunities from the sale of dried products, greater food availability).

In recent years, the technological landscape of food dehydration has evolved considerably. Low-temperature freeze-drying and solar drying have emerged as two prominent alternatives to traditional preservation methods such as freezing or refrigeration, both of which are notoriously energy-intensive. Freeze-drying, although more expensive, allows for excellent preservation of heat-sensitive nutrients and bioactive compounds, keeping the original characteristics of the product almost intact [43]. This method removes water through vacuum

sublimation, preserving structure, flavour, and nutritional value better than any other drying process, and is therefore suitable for foods with high nutritional or functional value. On the other hand, solar drying is a low-cost alternative based on renewable energy, particularly suitable for regions with high solar radiation. In rural areas and developing countries, the use of improved solar dryers offers a sustainable solution for preserving crops while reducing dependence on the electricity grid: recent studies highlight technical advances from passive to hybrid solar systems, with increased drying efficiency and dried product quality [44]. These technologies reduce operating and energy costs for small and medium-sized producers, making post-harvest storage more accessible. For example, the adoption of solar dryers in farming communities can significantly increase farmers' annual profits (by reducing product losses) while mitigating emissions: it is estimated that up to hundreds of thousands of tons of CO_2 could be reduced each year thanks to the reduced use of fossil fuels for drying [44]. The combination of solar systems with supports such as thermal accumulators or hybrid photovoltaic integrations also makes it possible to stabilize the process and increase efficiency even in less-than-optimal climatic conditions [45]. However, not all dehydration technologies have the same energy footprint. For example, microwave drying is recognized for its speed and technical effectiveness, but it could involve significant energy consumption, especially if the electricity used comes from non-renewable sources. In general, industrial drying processes are energy-intensive: it is estimated that drying accounts for 12–20% of total global industrial energy consumption [46].

In specific supply chains, such as that of dried fruit, the drying phase accounts for a significant proportion of the product's overall emissions [46]. These data indicate that the environmental impact of dehydration depends heavily on the efficiency of the process and the energy source used. The advanced technologies available today can certainly improve efficiency: for example, the targeted use of microwaves in combination with traditional methods increases drying speed and product quality while reducing energy consumption and emissions compared to conventional thermal methods [46]. Nevertheless, if this energy comes from fossil fuels, the environmental benefit is reduced; conversely, powering drying processes with renewable energy is crucial to maximizing their ecological benefits. Optimizing energy use (e.g., by modulating the power and thickness of the product being dried) and recovering heat within the plants are further key strategies for reducing the carbon footprint of dehydration technologies [47]. To ensure that dehydration effectively contributes to more sustainable food systems, it is therefore essential to integrate it with renewable energy solutions at processing sites. The installation of solar thermal collectors, photovoltaic

panels, and hybrid energy configurations can drastically reduce the carbon intensity of drying operations, eliminating (or almost eliminating) direct emissions linked to the consumption of electricity or heat from fossil fuels. At the same time, there is growing institutional support for the development and dissemination of low-emission drying technologies. The implementation of sustainability-oriented public policies and incentives is promoting the industrial-scale adoption of low-carbon production processes in the food sector. For example, the fight against food waste, supported by investments in innovative technologies and adequate infrastructure, is recognized as an integral part of climate and sustainable development strategies, with benefits ranging from emissions reductions to improved resource efficiency and food security [48]. At the international level, the UN Sustainable Development Goal SDG 12.3 calls for halving per capita food waste by 2030 [49], while in recent national climate action plans (NDCs), many countries are including measures to decarbonize agri-food supply chains and promote cleaner production processes [50]. These trends indicate a growing institutional and industrial commitment to low-carbon food processing models. In conclusion, the integration of efficient dehydration technologies powered by renewable energy is a crucial step in the ecological transition of the agri-food sector, contributing both to climate change mitigation and waste reduction, towards a more sustainable and resilient food system.

1.3 Dehydration

Food dehydration is a unit operation based on the controlled removal of free water—and, to some extent, bound water—present in food products, to ensure microbiological, enzymatic and chemical stability during storage. Food drying is a simultaneous heat- and mass-transfer process, in which thermal energy causes the evaporation of water and a concentration gradient drives moisture from the interior of the product toward the exterior [51]. The method guarantees a longer shelf life, a substantial reduction in storage costs and improved transportability. Unlike other preservation techniques, such as refrigeration or the use of chemical additives, dehydration requires neither a cold chain nor the addition of preservatives, thus representing a healthier and more eco-friendly form of preservation. Various mathematical models have been developed to describe the kinetics of moisture reduction in foods, both diffusive (derived from Fick's laws) and empirical or semi-empirical. Fick's law provides the theoretical basis: for example, the drying behaviour of thin slices of vegetables can be well represented by solutions of Fick's diffusion equation [52]. From a technical standpoint, dehydration is governed by thermal and diffusive phenomena: heat is transferred into the product and moisture migrates toward the surface,

where it evaporates. Fick's First Law describes steady-state conditions and states that the mass flux (J) is proportional to the concentration gradient (1):

$$J = -D\left(\frac{dC}{dx}\right) \quad (1)$$

where J is the mass flux (kg m^{-2} s^{-1}), D the diffusion coefficient (m^2 s^{-1}) and dC/dx the concentration gradient along the spatial direction.

Because dehydration is a dynamic process, it is more appropriate to use Fick's Second Law, which describes the temporal variation of concentration as a function of diffusion (2) [53]:

$$\frac{\partial C}{\partial t} = D\left(\frac{\partial^2 C}{\partial x^2}\right) \quad (2)$$

In this context, $\partial C/\partial t$ denotes the temporal rate of change of moisture concentration within the material, while D symbolises the diffusivity, specifically identified as the effective diffusivity D_{eff}, measured in m^2 s^{-1}. Furthermore, $\partial^2 C/\partial x^2$ signifies the second-order spatial derivative of concentration, reflecting the variation in moisture distribution. Within this context, D_{eff} is a significant parameter that is greatly affected by temperature, the microstructure of the food matrix and its moisture content [54]. Elevated temperatures tend to increase the mobility of water in the matrix and thus promote diffusion [55]. The moisture content (x), either on a dry basis (kilograms of water per kilogram of dry matter) or on a wet basis (kilograms of water per kilogram of wet product), is a fundamental parameter for assessing the advancement of dehydration. Its variation as a function of time provides critical information regarding the dynamics of the process. Ambient relative humidity and temperature have a large effect on the product–environment differential vapour pressure and thus on the evaporation potential [56]. Drier air or higher temperatures enhance surface water migration and evaporation rates. The heat-transfer coefficient (h) and the external mass-transfer coefficient (k) also dictate thermal and mass-transfer efficiency at the product surface. High values of these coefficients indicate that internal diffusion, rather than surface resistance, is the process-limiting transfer [57,58].

Operational factors and product characteristics influence drying effectiveness. Air temperature is often the crucial parameter: higher temperatures increase the saturation vapour pressure and the moisture gradient, thereby accelerating the drying rate [53]. However, beyond a certain threshold, excessively high temperatures can cause the phenomenon of casehardening (formation of a dry superficial crust) that slows internal water diffusion and further degrades nutrients [52]. Relative humidity (RH) also exerts influence: drier air improves the capacity to remove moisture from the product, but extremely low RH

can accentuate casehardening. Optimisation studies show that there are optimal combinations of temperature and RH to maximise efficiency: for example, for onion slices it was found that about 50 °C and 65 % RH represent optimal conditions that reduce drying times while preserving up to 96 % of total phenolic compounds compared with more extreme conditions [52]. Product size and thickness influence the diffusion path: smaller pieces or thinner slices dry more rapidly. The structure of the product (porosity, presence of cuticle or peel, etc.) and the diffusivity of water within it (which increases with temperature) play a decisive role in the falling-rate period of drying [53,59]. For example, products with a porous or spongy structure (such as herbs) facilitate faster evaporation, whereas fleshy fruits with a waxy peel (e.g., raisins) may require pre-treatments to break diffusion barriers. Air velocity over the product also affects the external mass-transfer coefficient: a faster airflow removes water vapour from the surface more rapidly, maintaining a high concentration gradient and accelerating drying, up to a limit beyond which the effect becomes less pronounced (internal diffusion-controlled regime) [59,60]. In summary, the optimal management of process parameters, moderate temperature, adequate ventilation, controlled humidity, makes it possible to strike a balance between the rate of water removal and the quality of the dried product [61]. The differential equation reported above enables modelling of moisture variation during drying, with initial and boundary conditions related to the geometry of the product (slab, cylinder, sphere) and to its thermo-physical properties. Given the complexity and heterogeneity of food matrices, simplified or semi-empirical models derived from solutions of Fick's Second Law are often used. For convenience, thin-layer kinetic models are applied: the Henderson and Pabis model is the monomolecular analytical solution (first term) of Fick's series for the drying of granular materials [62], whereas the Page model is an empirical variant of the Newton/Lewis model with an adjustable exponent introduced to better fit the drying curves of cereals [63]. Numerous other formulas (Two-term, Logarithmic, Wang & Singh, etc.) and adjustments derive from these. In recent decades the Midilli et al. model (2002) [64] has been proposed, combining linear and exponential terms: many comparative studies show that the Midilli model often provides the best fit of drying data for a wide range of products compared with other classic models [65]. For example, in a study on six different models applied to the drying of edible insects, the Midilli model showed a determination coefficient R^2 of 0.9997, superior to the others, indicating an excellent ability to describe the drying curve [66]. The use of such kinetic models makes it possible to estimate useful parameters such as the effective moisture diffusivity and the optimal process times, thereby facilitating the design and sizing of dryers [53].

The dehydration process can significantly affect food quality from both nutritional and organoleptic perspectives. Increasing the temperature accelerates the process by providing a greater driving force (vapour-pressure difference) for water removal [67,68], but excessive temperatures can cause rapid degradation of heat-sensitive components (e.g., polyphenols) and do not always proportionally reduce drying times because of phenomena such as surface hardening [61]. In general, water removal concentrates nutrients, but process conditions (temperature, duration, method) determine the losses of compounds sensitive to heat and oxidation. Kamiloglu et al. (2016) [69] reviewed the effects of different drying methods on the antioxidant content of fruit and vegetables: marked reductions of vitamin C, carotenoids, polyphenols and consequent declines in total antioxidant activity are often observed with the more aggressive thermal methods. For example, hot-air drying can cause losses of 30–70 % of vitamin C depending on temperature, whereas faster methods such as microwaves or gentler ones such as freeze-drying preserve a higher share [69]. In addition to nutrients, sensory parameters can also deteriorate prolonged heat exposure induces non-enzymatic browning (Maillard reactions), loss of volatile aromas and texture changes (hardening or excessive shrinkage of the dried tissue). Colour and aroma are better preserved by processes such as freeze-drying or low-temperature drying, whereas high-temperature convective drying tends to darken the product and can alter its flavour [70] (e.g., intermittent or vacuum drying) can improve the retention of sensory compounds compared with continuous hot air [71]. In summary, there is a trade-off between maximum drying rate and final quality: process optimisation aims to minimise nutritional and sensory damage while guaranteeing preservative activity.

Numerous technologies are employed to dehydrate foods, each with its own characteristics. Conventional methods include hot-air convective drying (at atmospheric pressure) and vacuum drying, as well as microwave, infrared, osmotic drying and combinations thereof (for example microwave–vacuum drying) [51]. Hot-air drying remains the most widespread technique because of its simplicity and low operating costs; however, it is also the one that produces the greatest negative effects on the nutritional and sensory qualities of the product. To limit these, more advanced techniques have been developed. Vacuum drying, for example, lowers the partial pressure of water vapour, allowing evaporation at lower temperatures and minimising thermal degradation of thermolabile compounds. In freeze-drying (lyophilisation) water is removed by sublimation under vacuum at low temperatures: this method is often considered the most effective for preserving aroma, colour and structure of the product compared with other dehydration methods [70]. The literature also reports

innovative approaches such as the use of ultrasound, electric fields or edible coatings to improve drying efficiency, but hot-air drying remains the most widespread industrial technique for its cost-effectiveness [69]. The use of ultrasound as a pre-treatment is an emerging technology: the mechanical vibrations increase the permeability of the food matrix, facilitating internal moisture diffusion and reducing mass-transfer resistance. Hybrid systems that combine microwaves, infrared and ultrasound with conventional methods are currently under study, with the aim of achieving faster, more uniform and less invasive dehydration processes. Each technique presents specific characteristics that make it suitable for categories of foods.

1.4 Non-Thermal Technologies

In recent years, non-thermal technologies have attracted increasing interest as pre-treatments in dehydration processes, as they enhance processing efficiency without compromising the nutritional and sensory qualities of food. Among the most promising methods are ultrasound (US), pulsed electric fields (PEF), and immersion treatments in solutions containing citric and ascorbic acid (dipping). The application of low-frequency ultrasound (power ultrasound, ~20–40 kHz) as a pre-treatment has shown beneficial effects in the drying process. Ultrasonic waves generate the phenomenon of acoustic cavitation, i.e., the formation and collapse of microbubbles within plant tissue, which causes microfractures and microchannels in the cellular structures [72]. This sponge effect increases the material's permeability and accelerates mass transfer of internal water to the surface, thus shortening drying times [72]. Several experimental studies confirm that ultrasonic pre-treatment can significantly reduce drying time while also improving certain quality aspects of the dried product. For instance, Abbaspour-Gilandeh et al. (2019) and Sledz et al. (2017) report a reduction in drying time for walnut kernels and basil leaves, respectively, when subjected to ultrasound prior to drying [73,74]. In the latter case, ultrasonic treatment also better-preserved antioxidant compounds and leaf structure compared to the untreated sample [75,76]. Similarly, Tao et al. (2016) observed that in the convective drying of mulberry leaves, ultrasonic pre-treatment at 21 kHz reduced drying time and improved parameters such as colour and vitamin C content in the final product [77]. The physical mechanisms involved (cavitation, mechanical vibrations, localized heating) not only favour drying kinetics but also lead to positive outcomes such as greater rehydration capacity of the dried product and fewer changes in colour and texture [75]. Therefore, the use of ultrasound as a pre-treatment is considered a promising non-thermal technique to intensify drying, as also highlighted by recent reviews [73,76,78]. From a technological perspective, ultrasonic

treatments are applied through sonic baths or direct immersion probes, with operational parameters varying according to the type of matrix, applied power, and treatment duration. In the case of pulsed electric fields (PEF), the principle is based on applying short high-voltage pulses (typically 1–10 kV/cm for solid plant tissues, up to ~50 kV/cm for liquids) to the food product to permeabilize the cell membranes. This treatment induces the phenomenon of electroporation, i.e., the formation of transient or permanent pores in biological membranes, which increases cellular permeability and facilitates water release from the cells [79]. In this way, PEF can be used as a pre-treatment to speed up drying: by breaking down cellular barriers, internal moisture more easily migrates outward during subsequent convective or vacuum drying. A key advantage is that PEF is a non-thermal treatment: pulses last only a few micro- to milliseconds, so product heating is minimal [80]. Punthi et al. (2022) report in a review that PEF is effective in accelerating the drying of fresh fruits and vegetables due to electroporation, also improving the rehydration and texture of the resulting dried products [81]. For example, Liu et al. (2020) demonstrated that treating fresh carrots with PEF pulses at 0.6 kV/cm for 0.1 s prior to drying reduced drying time by 55% (at 25 °C) compared to untreated controls [82]. The treated tissue showed a more open structure that allowed for faster drying without raising the process temperature, and the rehydrated product exhibited better sensory properties (brighter colour, less hardness) than carrots dried without PEF [82]. These findings indicate that PEF is a highly useful pre-treatment to accelerate drying kinetics and potentially improve the quality of dehydrated products, also reducing overall process energy consumption due to shorter times [83]. It should be noted that the effectiveness of electroporation depends on field parameters (intensity, number, and duration of pulses) and on food characteristics [82]; therefore, optimization of the PEF treatment must be tailored case by case to maximize benefit without causing excessive tissue damage or undesirable effects on quality [84,85]. Enzymatic browning, due to the action of polyphenol oxidase (PPO) on phenols and catechins, is a common problem during the processing and drying of fruits and vegetables (e.g., apples, pears, potatoes). A widely used pre-treatment to inhibit it is immersion of the product in solutions of ascorbic acid (vitamin C) and citric acid prior to drying. These additives act through different but complementary mechanisms: citric acid lowers tissue pH below the enzyme's optimum and chelates copper ions in the active site of PPO, inactivating it [86,87]; ascorbic acid, instead, functions as a reducing agent that immediately converts quinones (oxidized by PPO) back into phenols, preventing their polymerization into brown melanin [88]. Additionally, ascorbic acid consumes oxygen and acts as a direct antioxidant, further

slowing browning reactions. Thanks to these effects (acidification, anti-browning, and metal sequestration), the combined ascorbic/citric treatment is highly effective in preventing surface enzymatic browning [89]. For example, it has been observed that fruit or vegetable juices treated with ascorbate and citrate retain a light colour if the pH remains sufficiently low (~3–4) to inhibit PPO [90]. In apple slices and other minimally processed products, short immersions in ascorbic acid (0.5–1%) and citric acid (1–3%) solutions before drying prevent darkening during drying and early storage, better preserving natural colour [88]. This approach, documented in numerous studies, is widely practiced in the dried fruit and vegetable industry (often in combination with sulphites or other antioxidants) because it limits browning without significantly altering the final product's flavour.

In addition to ultrasound and PEF, recent research is exploring new non-thermal technologies to pretreat foods to improve their stability and quality without applying heat. Below are some of these emerging technologies, along with their principles and applications.

1.4.1 High Hydrostatic Pressure (HPP)

This consists of applying very high pressures (4000–6000 bar) to foods in a liquid environment at room or refrigerated temperature. This treatment inactivates pathogenic and spoilage microorganisms (bacteria, yeasts, molds) and some enzymes, extending shelf life in a pasteurization-like manner but without heat [80]. HPP preserves the nutritional and sensory qualities of fresh foods very well since it does not break down heat-sensitive compounds; for example, fruit juices, purees, and products like guacamole treated with high pressure retain fresh colour and flavour while being microbiologically stable [80]. HPP is also being studied as a pre-treatment before drying to soften plant tissues (with slight gelatinization effects on pectins and cell walls) and facilitate water release, although it is not yet widely used for this specific purpose compared to its application as a cold stabilization technology.

1.4.2 UV-C Radiation

Irradiation with ultraviolet-C light (~254 nm wavelength) is a non-thermal method for surface sanitization of foods. UV-C rays damage the nucleic acids of bacteria, yeasts, and molds, preventing replication, and can also inactivate surface enzymes like PPO, contributing to reduced browning [90]. Already applied to sanitize air and water, UV-C is used in the food sector to treat whole or minimally processed fresh fruits (e.g., cut) to reduce microbial load and slow spoilage. Recent studies have shown that appropriate UV-C treatments can suppress enzymatic browning in products like cloudy apple juice or fruit pieces by lowering PPO activity without using chemical additives [90]. Since UV-C does

not penetrate deeply, its effectiveness is limited to exposed surfaces; hence, it is considered a useful pre-treatment for thin fruit/vegetable slices before drying, to reduce initial microbial load and preserve surface colour. The dose (J/m²) must be calibrated to avoid undesirable effects (e.g., photooxidative odours), but within legal limits UV-C is a rapid intervention (few seconds or minutes) compatible with minimal processing food production.

1.4.3 Atmospheric Cold Plasma

Non-thermal plasma is an ionized gas typically generated by applying high voltage to an air or gas flow (e.g., helium) at atmospheric pressure. The plasma contains various reactive species (oxygen and nitrogen radicals, ozone, electrons, ions) capable of destroying microbial contaminants on food surfaces and degrading undesirable molecules, all at room temperature (25–65 °C) [91]. This emerging technology allows for effective sanitization of fruits, vegetables, and seeds without heating: for example, a few minutes of exposure to cold plasma reduced E. coli and Saccharomyces counts in juices by 5–7 log units without significantly altering the product's colour and flavour [92]. Cold plasma has also been proposed as a post-harvest treatment to extend the shelf life of fresh products; one study showed that combining antimicrobial washing with in-package plasma treatment of citrus (mandarins) resulted in strong reduction of spoilage fungi (*Penicillium digitatum*) without negative effects on texture, sensory quality, or fruit nutrients [93]. Mechanistically, plasma reactive species oxidize microbial membranes and DNA, causing cell death [94]; since the process occurs at low temperature, there is no heat damage typical of thermal treatments. Cold plasma is thus a powerful non-thermal tool for decontamination and potentially for inactivating surface-degrading enzymes, improving food safety and stability before drying or packaging [95]. Ongoing research aims to integrate plasma into in-line production systems (e.g., conveyor belts) and combine plasma with other hurdles (UV light, natural sanitizers) to maximize effectiveness while preserving quality.

Overall, the integration of non-thermal pre-treatments - ranging from US and PEF to HPP, UV-C and cold-plasma signals a shift toward milder, more sustainable dehydration routes that safeguard heat-labile nutrients while accelerating water removal. By selectively disrupting cellular barriers or inactivating spoilage enzymes, these techniques can trim energy use, shorten drying times and elevate both sensory and functional quality. Their scaled-up adoption, however, hinges on meticulous optimisation of process variables and on equipment capable of delivering uniform treatment across heterogeneous food matrices. Growing evidence also points to synergistic “hurdle” strategies that combine two or more non-thermal approaches for even greater efficacy. Taken together, these developments

outline a versatile toolbox for producing safer, higher-value dried foods that address modern consumers' expectations for health, convenience and environmental stewardship.

1.5 Research Question

In recent years, the imperative to reconcile food safety, waste reduction and the mitigation of environmental impact has renewed the focus on dehydration, a traditional yet still strategic operation for stabilising highly perishable fruits and vegetables. Conventional techniques, from hot-air drying to freeze-drying, already extend the commercial life of fresh produce, but they are burdened by well-known drawbacks: lengthy processing times, substantial energy consumption and, at times, severe degradation of heat-labile nutrients and aromatic compounds. In parallel, industry and academia have been testing non-thermal pre-treatments, such as low-frequency power ultrasound and pulsed electric fields, capable of permeabilising cell membranes or inactivating enzymes and micro-organisms without the use of heat, with the dual aim of accelerating water removal and preserving the original qualitative attributes of foods. Against this backdrop, the present thesis asks how the combination of such pre-treatments with traditional drying schemes affects, in a synergistic manner, not only the rate of moisture removal but also, crucially, the preservation of nutritional quality, the microbiological safety over shelf life and the environmental footprint of the entire process. The overarching research question may therefore be formulated as follows: which process configurations, integrating dehydration technologies with non-thermal pre-treatments, can simultaneously optimise kinetic efficiency, nutrient and sensory retention, microbial load reduction and the containment of energy use and CO_2 emissions, and which physico-chemical principles govern their effectiveness?

Addressing this question entails tackling a multidimensional problem. On one hand, it requires quantifying the heat- and mass-transfer parameters that dictate drying velocity; on the other, it calls for experimental assessment of how different treatments influence vitamin, polyphenol and pigment losses, the formation of undesired compounds and microbial inactivation. A thorough answer will not only deepen our understanding of dehydration mechanisms but also provide concrete guidelines for future industrial scale-up, translating the concept of "sustainability by design" into operational solutions for the agri-food sector.

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CHAPTER 2

Pulsed electric fields pre-treatment and ultrasound-assisted hot air drying for the dehydration kinetics and nutritional quality enhancement in apricots

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Abstract

In recent years, the growing demand for healthy and natural snacks has driven innovation in dried fruit production processes, with particular attention to the nutritional and sensory quality of the products. In this context, the present study aims to enhance the hot air drying (HAD) process of apricots by combining pulsed electric fields (PEF) pre-treatments and the application of airborne ultrasound (US) during drying. The experimental design included six treatments combining two PEF pre-treatments (50 and 200 pulses; 1.5 kV/cm) with HAD (60 ºC; 1 m/s) assisted or not with US (20.5 kW/m3; 21 kHz). From modelling of the drying kinetics, effective diffusivity (D_{eff}) and external mass transfer coefficient k were calculated for each treatment. The quality of the dried product was assessed in terms of shrinkage, total polyphenol content (TPC), antioxidant capacity (FRAP) and colour (ΔE). Results showed that the combination of PEF pre-treatments and US-assisted drying provided the shortest process. Samples treated with a moderate PEF treatment exhibited lower losses in antioxidant capacity and total polyphenol content, while the application of US not significantly affected them. Moreover, the combination of the PEF treatment and ultrasound provided the the lower alteration of fresh colour (ΔE) and had no significant influence on shrinkage measurements, confirming that it was the best treatment among tested for preserving the nutritional and visual quality. This approach proves to the combination of PEF pre-treatment and ultrasonic-assisted drying could be promising alternative for the dried fruit industry, offering not only a significant shortening of the processing time but also an improved quality of the final product.

Keywords: PEF; Ultrasound; Drying kinetics; Food quality; Antioxidant analysis.

1. Introduction

The food industry produces large volumes of by-products containing valuable bioactive compounds, they degraded quickly due to their high-water content [1]. Drying is widely used to stabilize them materials by reducing their water activity, which extends shelf life and reduces storage and transportation costs [2]. Apricot (*Prunus armeniaca* L.) is an example of a fruit with high nutritional value but also high perishability. Fresh apricots are rich in carotenoids, phenolic compounds, and vitamins with antioxidant, anti-inflammatory, and other beneficial properties [3]. However, its 85 % of moisture content combined with its thin skin and delicate tissue makes it extremely susceptible to microbial decay and quality loss after harvesting [4]. The apricots drying is a common way to prevent spoilage while retaining some of their nutritional value [5]. However, the achieving an efficient drying without compromising quality remains a challenge.

Conventional hot air drying (HAD) is the most used method for dehydrating apricots due to its practicality and low cost [6]. However, this process often requires long processing times and the use of high temperatures, which can lead to significant quality degradation reactions, such as browning, shrinkage, texture changes, and loss of heat-sensitive nutrients (e.g., vitamin C) [7]. In practice, pre-treatments are often employed to accelerate moisture removal during drying and mitigate these quality losses. Traditional pre-treatment techniques include, among others, blanching, osmotic dehydration, or freeze-thaw cycles, while more recent approaches have explored the application of innovative technologies such as cold plasma, infrared heating, high-intensity ultrasound, or pulsed electric fields [8]. These pre-treatments mainly aim to induce microstructural changes (cell permeabilization, tissue softening, etc.) that facilitate faster water diffusion during drying, and thus, to improve drying efficiency and product quality.

Among the emerging non-thermal pre-treatments, pulsed electric fields (PEF) have shown promise for fruits and vegetables. PEF involves exposing plant tissue to short, high-voltage pulses, which induce the formation of pores in cell membranes, a phenomenon known as electroporation [9,10]. By permeabilizing cell membranes, PEF pre-treatment improves the release of water and solutes, which can enhance water transfer in a subsequent dehydration process [11]. Furthermore, PEF pre-treatment has been shown to improve the quality of dried fruit by preserving higher levels of antioxidants and other bioactive compounds [12]. In this sense, recent studies on apricots have reported that PEF-treated samples retained greater

amount of phenols and higher antioxidant activity after drying than untreated ones [13–16]. However, it should be noted that the application of an excessive PEF intensity can highly damage cell structure; induce an excessive soften of the fruit matrix and cause an intense cell collapse, which paradoxically could hinder moisture diffusion [8,17]. Optimizing PEF treatment is therefore critical to maximize the benefits of drying while avoiding quality deterioration.

Ultrasound (US) is another innovative technique for intensifying drying processes. Power ultrasound has often been applied as a pre-treatment in liquid media by immersing the product in an ultrasonic bath. The pre-treatment can improve the internal diffusion of water under moderate heating conditions [18], shortening the later drying and helping to preserve heat-labile compounds [19]. However, immersing fruits in a liquid can led to the leaching of soluble constituents and induce some potential contamination or sensory changes in the product [20]. An alternative to avoid these problems is the air-borne ultrasound-assisted drying, which consist of to introduce ultrasonic waves directly into the air surrounding the product during drying [21–24]. The high-intensity acoustic waves create rapid pressure oscillations and micro-flows in the boundary layer of air on the surface of the food, leading to a thinning of the stationary moisture layer and increased water evaporation [1]. Moreover, ultrasound induce the successive compression and expansion of food, phenomenon known as sponge effect, that facilitate the water movement inside the solid matrix. All these mechanisms have been shown to significantly accelerate drying: reducing drying times by 20-30% under moderate temperature conditions [25]. Furthermore, ultrasound assistance can better preserve the microstructure of the product and heat-sensitive nutrients than conventional hot air drying, as reported in various types of fruit and vegetables [26,27]. Airborne ultrasound therefore represents a promising non-thermal means of intensifying drying efficiency while maintaining quality.

The combination of PEF pre-treatments and ultrasound-assisted drying has hardly been explored, especially for whole fruits [8] and, to our knowledge, no published work about their application in the convective drying of apricots has yet been published. The aim of the present research was to fill this gap. Specifically, the influence of PEF intensity and applying airborne ultrasound during drying on the drying kinetics and quality of dried apricots was addressed. The key quality attributes included shrinkage, color, and antioxidant properties (vitamin C, polyphenols) retention were assessed.

2. Materials and Methods

2.1 Sample preparation

Fresh apricots (*Prunus armeniaca* cv. Orange Red) were purchased from a local producer in Valencia (Spain), stored at 4 °C and processed within 24 h of harvest. The fruits exhibited an initial moisture content of 7.30 ± 0.15 g H_2O g^{-1} dry basis (87.9 % wet basis), determined by differential weighting after oven drying at 105 °C for 24 h (AOAC, 2019). Prior to treatment, the apricots were peeled, destoned and sliced to 9.15 ± 1.38 mm thickness using a stainless-steel guillotine slicer. Ten central slices from different fruits were selected for each drying run, which were equilibrated at 22 ± 1 °C and 55 % for relative humidity for 30 min before processing.

2.2 Drying experiments

Six different drying treatments were considered: conventional hot-air drying (HAD, control), ultrasound-assisted hot-air drying (US-HAD), PEF pre-treatment at moderate intensity followed by hot-air drying (PEF50-HAD), PEF pre-treatment at moderate intensity followed by ultrasound-assisted drying (PEF50-US-HAD), PEF pre-treatment at high intensity followed by hot-air drying (PEF200-HAD) and PEF pre-treatment at high intensity followed by ultrasound-assisted drying (PEF200-US-HAD).

2.3 Pulsed Electric Field (PEF) pre-treatment

Apricot slices were pre-treated individually in a rectangular treatment cell equipped with two stainless-steel electrodes (64 cm^2) mounted 8.0 cm apart and completely submerged in tap water (1 035 ± 30 µS cm^{-1} at 25 °C) to ensure electrical contact. A batch generator (EPULSUS-PM1, Energy Pulse Systems, Lisbon, Portugal) delivered quasi-rectangular monopolar pulses of 25 µs and 1.5 kV cm^{-1} applied at 10 Hz. Two different conditions were considered, one of them of moderate intensity by applying 50 pulses (PEF50), which correspond to a specific energy input of 0.4 kJ kg^{-1} of wet matter, and another more intense of 200 pulses (PEF200), which provide a specific energy inputs of 16 kJ kg^{-1} of wet matter. After the PEF treatment the apricot samples were drained, blotted with absorbent paper to remove surface water and immediately transferred to the drying system.

2.4 Ultrasound-assisted hot-air drying

Drying experiments were carried out in a convective drier equipped with a high-intensity airborne-ultrasound system, previously described by Polachini et al. (2023) [28]. For each drying run ten central slices from different fruits were placed on a stainless holder that guaranteed uniform airflow and ultrasonic intensity across the product. The process operated

at an air velocity of 1 m s^{-1} and a fixed temperature of 60 °C. High-intensity ultrasound (21.8 kHz) was applied via a cylindrical drying chamber excited by a piezoelectric transducer. Drying experiments were performed without (HAD) and with (US-HAD) ultrasound application; this last case performed applying an electrical input to the ultrasonic transducer of 50 W to provide an acoustic power level of 20.5 kW m^{-3}. Sample weight was automatically recorded every 5 min. The drying procedure was completed when no change in mass was detected in four consecutive measurements. After processing, apricot slices were stored in a hermetic container with silica-gel desiccant and protected from light until quality parameters were analysed.

2.5 Drying-kinetic modelling

To quantify the influence of PEF pre-treatment and airborne ultrasound application on the moisture removal kinetics, a model based in Fick's second law for one-dimensional diffusion in a finite slab was considered (Eq. 1):

$$\frac{\partial W\,(x,t)}{\partial t} = D_{eff}\left[\frac{\partial^2 W\,(x,t)}{\partial x^2}\right] \quad (1)$$

where W is the moisture content (kg water kg^{-1} dry matter), D_{eff} (m^2 s^{-1}) the effective diffusivity, t (s) the drying time and x (m) the spatial coordinate of the moisture transport. The following assumptions were adopted: (i) D_{eff} remained constant during the run, (ii) the moisture was initially uniformly distributed (Eq. 2) and (iii) the samples behaved as a symmetric infinite slab of half-thickness L (Eq. 3).

$$W(x,0) = W_0 \quad (2)$$

$$\frac{\partial W\,(0,t)}{\partial x} = 0 \quad (3)$$

External mass-transfer resistance was accounted for by imposing a convective boundary condition at the product–air interface (Eq. 4):

$$-D_{eff}\,\rho_{ss}\frac{\partial W\,(L,t)}{\partial x} = k(a_w(L,t) - \varphi_{air}) \quad (4)$$

where ρ_{ss} is the dry-solid density (kg m^{-3}), k (m s^{-1}) the external mass-transfer coefficient, a_w the surface water activity, L the half-thickness of the sample and φ the air the relative humidity of the drying air. Equilibrium moisture (Weq) was estimated from the apricot desorption isotherm reported by Moraga, Martínez-Navarrete & Chiralt (2006) [29].

The partial differential equation was solved applying an explicit finite-difference method in Matlab R2024a (The MathWorks, Natick, MA, USA). D_{eff} and k were identified by minimizing the sum of squared differences between experimental and simulated moisture

contents using the Nelder–Mead simplex algorithm (*fminsearch*). The goodness of fit of the models was verified by calculating the coefficient of determination (R^2) (Eq. 5), the mean absolute error (*MAE*) (Eq.6), the mean relative error (*MRE*) (Eq.7) and the percentage variance explained (*%VAR*) (Eq. 8):

$$R^2_{adj} = 1 - \left(\frac{N-1}{N-p}\right)\frac{\sum_{i=1}^{N}(y_i^{exp} - y_i^{pred})^2}{\sum_{i=1}^{N}(y_i^{exp} - \bar{y})^2} \quad (5)$$

$$MAE = \frac{1}{N}\sum_{i=1}^{N}\left|y_i^{exp} - y_i^{pred}\right| \quad (6)$$

$$MRE = \frac{1}{N}\sum_{i=1}^{N}\left|\frac{y_i^{exp} - y_i^{pred}}{y_i^{exp}}\right| \quad (7)$$

$$\%VAR = \left[1 - \left(\frac{S^2_{xy}}{S^2_y}\right)\right] \times 100 \quad (8)$$

where N is the number of observations, p is the number of estimated parameters, S^2_{xy} is the variance of the residuals and S^2_y the variance of the experimental data. All indices were obtained directly from Minitab output.

2.6 Quality Analyses

2.7 Sample shrinkage

Shrinkage was evaluated by measuring the three principal axes of each apricot sample, longitudinal diameter (LD), transverse diameter (TD) and thickness (THICK), before and after drying (Figure 1).

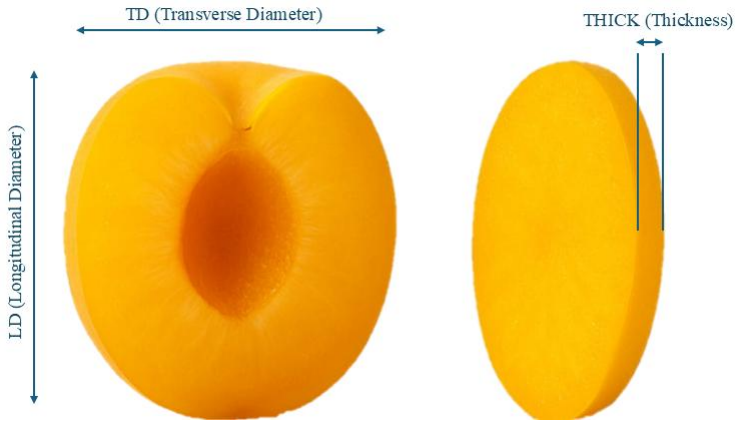

Figure 1. Three principal variables of apricot slices used to evaluate samples shrinkage: longitudinal diameter (LD), transverse diameter (TD), and thickness (THICK).

To do this, every sample was placed on a flat glass plate and measured at three equidistant points per axis using a digital calliper (Mitutoyo 500-196-30, resolution 0.01 mm). Nine slices per treatment were analysed. Mean initial dimensions (x_o) and final dimensions (x_f) were obtained by averaging the three readings for each axis. Shrinkage (%) was then calculated as [30]:

$$Shrinkage\ (\%) = \left[\frac{x_0 - x_f}{x_0}\right] \times 100 \quad (6)$$

where x represents LD, TD or THICK measurements. Results are reported as mean ± standard deviation.

2.8 Antioxidant properties

Antioxidant properties of fresh and dried apricots were assessed in ethanolic extract. To obtain them, a 1.000 ± 0.002 g aliquot of sample was mixed with 20 mL of 96 % ethanol and homogenised with an Ultra-Turrax T25 (IKA, Germany) for 1 min at 13 500 rpm. The slurry was kept at 4 °C for 24 h, centrifuged at 4 000 rpm for 10 min and filtered through glass-microfibre paper (1.2 µm). The filtrate was used in the following analysis

2.8.1 Total polyphenol content (TPC).

The Total polyphenol content (TPC) was assessed using the Folin–Ciocalteu assay [31]. This was performed by mixing 100 µL of extract with 200 µL of Folin–Ciocalteu reagent (Sigma-Aldrich, Spain) and 2 mL of distilled water. After 3 min at room temperature, 1 mL of 20 % (w/v) Na_2CO_3 was added and the mixture incubated in the dark for 1 h. Absorbance was read at 765 nm (Helios Gamma, Thermo Spectronic, UK). TPC was quantified from a previously determined gallic acid calibration curve and expressed as mg gallic acid equivalents (GAE) 100 g^{-1} dry. The results were expressed as TPC loss (%) in the dehydrated samples compared to the respective fresh samples.

2.8.2 Antioxidant capacity.

The Ferric reducing antioxidant power (FRAP) method [32] was applied to determine the antioxidant capacity of samples. FRAP reagent, composed of 0.3 M acetate buffer, 20 mM $FeCl_3 \cdot 6H_2O$ and 10 mM TPTZ in 40 mM HCl, was pre-incubated for 30 min at 37 °C. A volume of 900 µL of it was mixed with 30 µL of extract and 30 µL of distilled water, incubated at 37 °C for 30 min and the absorbance measured at 595 nm. FRAP was calculated as mg Trolox/100 g^{-1} dry matter following a previously determined calibration curve and expressed as FRAP loss (%) in the dehydrated samples compared to the respective fresh samples.

2.9 Colour

Pulp colour before and after drying was measured in CIELab coordinates with a CM-2500d spectrophotometer (Konica Minolta, Tokyo, Japan) under D65 illumination and 10° observer angle, specular component excluded (SCE). Parameters L* (lightness), a* (red–green) and

b* (yellow–blue) were recorded and total colour difference (ΔE) was calculated between fresh and dried samples according to Equation [33]:

$$\Delta E = \sqrt{(L^* - L_0^*)^2 + (a^* - a_0^*)^2 + (b^* - b_0^*)^2} \quad (8)$$

where, L^*_0, a^*_0, and b^*_0 represent the values of the colour parameters measured after drying.

2.10 Experimental design and statistical analysis

The influence of PEF pre-treatments and the ultrasound application during drying in each response variable effective diffusivity (D_{eff}), surface mass transfer coefficient (k), longitudinal/transverse/thickness shrinkage (LD/TD/THICK), total phenolic content (TPC), antioxidant activity (FRAP), and colour variation (ΔE), was analysed using a two-factor General Linear Model (GLM) in Minitab 21 (Minitab LLC, State College, PA, USA). The fixed factors were the number of PEF pulses (0, 50, 200) and the ultrasonic power (0, 20.5 kW m^{-3}) applied.

The main and interaction effects were evaluated with a confidence interval of 95 % and, if significant, explored using Tukey-HSD. In addition, differences among treatments were assessed by one-way ANOVA followed by Tukey's post-hoc test ($p < 0.05$). Factorial mean graphs and interaction plots permitted to visualize the combined influence of PEF and ultrasound. All experiments were performed, at least, by triplicate and the results are reported as mean and standard deviation.

3. Results ad discussion

3.1 Drying experiments

The experimented evolution of moisture content during drying was affected by the PEF pre-treatments and the application of airborne ultrasound (Figure 2).

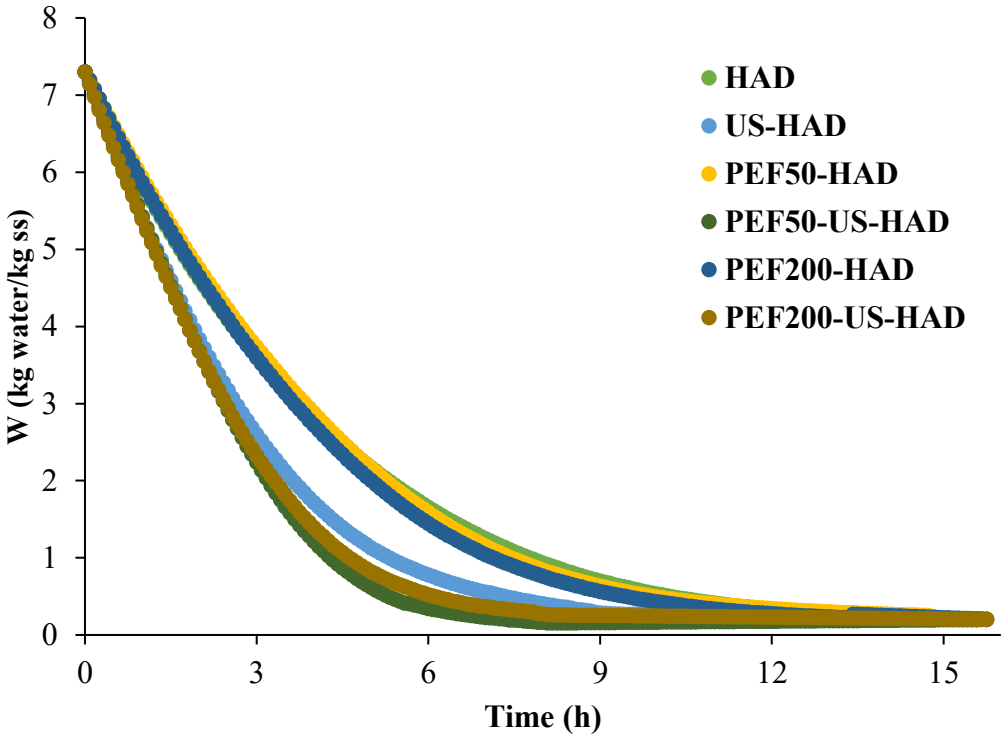

Figure 2. Experimental drying kinetics of apricot slices. Drying experiments carried out with samples pretreated or not with PEF at 50 (PEF50) and 200 (PEF200) pulses and without (HAD) or with ultrasound (US-HAD) application during drying.

In the case of the reference drying process (HAD), the equilibrium moisture content (0.20 kg kg^{-1} s.s.) was reached after 15.0 ± 0.4 h of drying, in line with the drying times reported for products with a similar parenchymatic structure [34]. The application of ultrasound during drying significantly accelerated the process. Although the initial segment of the curve appears approximately linear, coinciding with a constant-speed phase, the drying speed profiles do not show a sustained plateau, and the process quickly enters the decreasing-speed regime. The steeper initial slope therefore reflects a higher initial drying rate and mass transfer facilitated by ultrasound. This fact could be attributable to the sponge effect and surface micro-turbulence generated by the acoustic wave front [35]. In practical terms, US-HAD reduced the process time by about 12% (13 h) compared to HAD experiments, a value in line with that observed on apples and quinces by Puente-Domínguez et al. (2024), confirming the effectiveness of ultrasonic assistance on fruit matrices with high pectin content. As for the application of PEF pre-treatments, the PEF50 pre-treatment produced a moderate shortening of the drying time (-6%) compared to HAD. This can be linked to the

formation of pores that moderately reduce internal resistance to water transport [16]. On the other hand, increasing the number of applied pulses up to 200 shots (PEF200-HAD) resulted in 18% faster drying. This fact indicted that the application of a more intensity PEF pre-treatment could increase the level of the electroporation induced and then make the movement of water inside samples easier [16].

The combination of both technologies, PEF and US, produced the greater shortening of drying. Thus, PEF200-US-HAD experiments needed 10.5 ± 0.4 h to reach the equilibrium moisture, which means a drying process shortening of, 30% compared to HAD). In the case of PEF50-US-HAD, the process was completed in < 10 h, -35 % less than HAD experiments. This confirmed the possible synergy between the effects induced by both technologies. Therefore, PEF-induced electroporation and the resulting structural changes can create preferential pathways for water transport and enhance the effects of ultrasound. In the case of airborne ultrasound applied to convective drying, it has been shown that pressure oscillations and micro-current at the interface thin the diffusion boundary layer, reducing external resistance; at the same time, the sponge effect and micro-deformations of the tissue facilitate internal diffusion [36,37]. These mechanisms together lead to increases in effective diffusivity (D_{eff}) and mass transfer coefficient (k) and shorter drying times [38]. Evidence is reported, for example, for strawberries [39] and mangoes [40] dried with airborne ultrasound.

3.2 Diffusive modelling

The fitting of the proposed diffusive model (Eq. (3) to the experimental data permitted to quantify the effects of the conditions tested on the drying kinetics. Table 1 summarizes the mean values and the standard deviation of the effective diffusivity (D_{eff}), and the mass transfer coefficient (k) identified. The model fit was adequate as showed the values of statistics calculated ($R^2 \geq 0.998$; % var ≥ 99.8%; MRE< 0.13).

Table 1 Effective diffusivity (D_{eff}) and mass transfer coefficient (k) identified for the drying of apricot with and without of PEF pre-treatments and with and without ultrasound application during drying. R^2 represents the fraction of the variability in the data accounted for by the model, with values closer to 1 indicating a better fit.

Samples	$D_{eff} \cdot 10^9$ (m^2 s^{-1})	$k \cdot 10^3$ (kg m^{-2} s^{-1})	R^2	% VAR	MAE· 10^5 (m^2 s^{-1})	MRE
HAD	2.67 ± 0.38	3.87 ± 0.10	0.9989	0.9988	0.21	0.08
US-HAD	4.65 ± 1.08	5.45 ± 0.59	0.9994	0.9993	0.17	0.04
PEF50-HAD	2.95 ± 0.44	3.61 ± 0.27	0.9980	0.9980	0.38	0.13
PEF50-US-HAD	6.18 ± 0.81	5.17 ± 0.13	0.9994	0.9995	0.28	0.06

PEF200-HAD	3.37 ± 0.59	3.78 ± 0.61	0.9985	0.9984	0.34	0.10
PEF200-US-HAD	4.94 ± 1.04	5.29 ± 0.65	0.9993	0.9992	0.30	0.06

The use of ultrasound had a clear impact on the parameters. Thus, compared to HAD experiments, US-HAD showed an 74% average increase in D_{eff} (from 2.67 to 4.65 × 10^{-9} m^2 s^{-1}) and 41% in k. The effects induced by the acoustic field can generate sponge effect and internal microcracks that shorten the internal diffusion, as show the greater D_{eff} value. Furthermore, the induced micro stirring at the sample-air interface, thin the outer boundary layer, as show the increase of the k value. This fact as already reported for other pulp-rich fruits [35].

The application of PEF also affected the internal resistance. Thus, PEF50-HAD increased the D_{eff} by approximately 11% compared to HAD that could be attributable to different degrees of electroporation depending on the intensity of PEF treatment [16,44]. By increasing the pulses to 200 (PEF200-HAD), the increase in D_{eff} rises to 26%, indicating that the electro-permeabilizing effect was greater producing an easier movement of water inside the sample matrix [16]. No significant changes were observed in k. This is a logical result because the main effects of PEF are induced in the internal structure. Therefore, the k coefficient, which measure the external resistance to mass transfer was not significantly affected by PEF.

The combination of the two technologies showed the maximum drying efficiency of treatment tested. Thus, in PEF50-US-HAD samples, the D_{eff} increases by about 132% compared to HAD, while the mass transfer coefficient (k) increased by 34%, consistently exceeds 5 × 10^{-3} kg m^{-2} s^{-1}. By increasing the pre-treatment to 200 pulses (PEF200-US-HAD) the average D_{eff} reaches 4.94 × 10^{-9} m^2 s^{-1}, equal to an 85% increase over the control. This confirms the theory that the changes in the internal structure induced by PEF not only improved the internal moisture transport but also enhanced the effects of ultrasound. Therefore, the combination of both technologies has a synergistic effect significantly reducing the internal mass transfer resistance. In this case the values of PEF-US experiments were similar that US experiments. As commented before, PEF affects mainly at the internal structure. Therefore, the effects on k were due to mainly to the influence of ultrasound, independently if PEF were applied or not.

The diffusive model reveals a highly significant main effect of ultrasound on D_{eff} and k ($p < 0.001$). It also highlights a quadratic effect of PEF on D_{eff} ($p < 0.05$), with a marked increase when the number of pulses exceeds approximately the threshold of 100; finally, it highlights a PEF × US interaction that is also significant ($p < 0.05$), which explains the greater

efficiency observed in combined treatments. The model fit was excellent: $R^2 \geq 0.998$ for all cases, with MAE between 0.17×10^{-5} and 0.38×10^{-5} $m^2 s^{-1}$ and MRE always < 0.13. These values confirm the adequacy of the model in describing the dehydration dynamics of apricots, while ensuring a reliable prediction of diffusion parameters even in combined PEF-US protocols.

3.3 Quality Analyses

3.3.1 Shrinkage

The shrinkage that experimented the sample apricots after drying showed (Figure 3) a clear dependence on the intensity of the pre-treatment with pulsed electric fields, while the application of ultrasound during drying did not statistically significantly alter the final geometry of the product compared to the conventional drying (HAD).

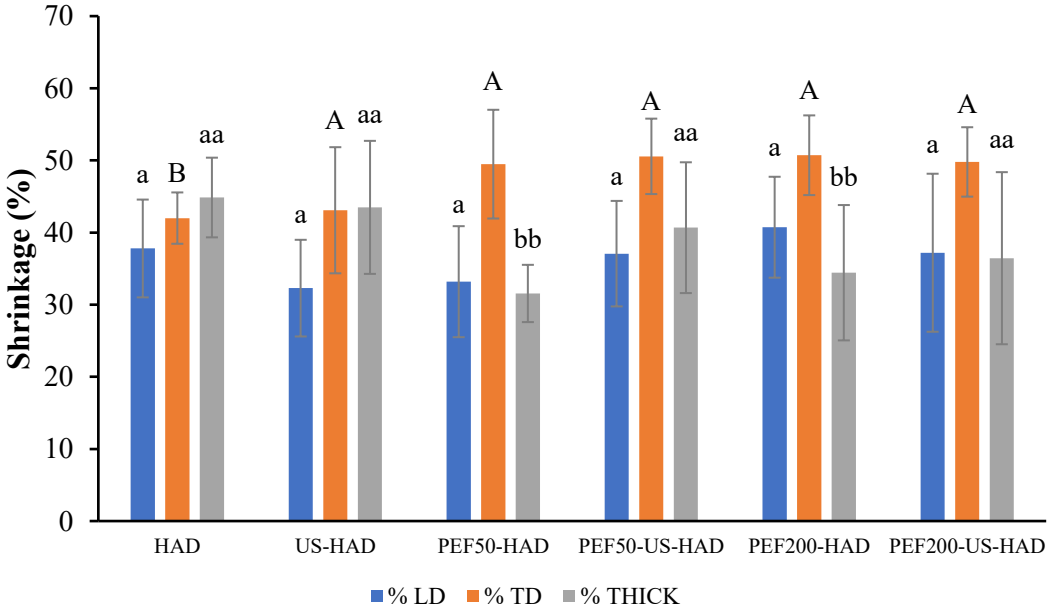

Figure 3 Shrinkage of apricot samples without and with apply a PEF pre-treatment with different number of pulses (50, PEF50; 200, PEF200) and without (HAD) or with (US-HAD) ultrasound application during drying. Percentage of reduction of longitudinal diameter (LD), transverse diameter (TD) and thickness (THICK). The three sets of letters (a, A, aa) represent the statistical significance of the parameter between treatments. Different letters represent significant differences.

Statistical analysis showed a significant effect (p-value < 0.05) of the PEF pre-treatment on transverse shrinkage (TD) and thickness (THICK). On the contrary, the longitudinal shrinkage (LD) was not significantly affected by the PEF (p = 0.27).

Thus, TD changed from 42.0 ± 4.0% in HAD experiments to 50.7 ± 0.5% in PEF200-HAD ones. In the case of THICK, the figures moved from 44.9 ± 1.6% in HAD to 34.4 ± 3.4% in PEF200-HAD. These results indicated that the structural changes induced by PEF promoted a more pronounced radial collapse but, at the same time, attenuated the loss of thickness of samples, likely due to the plastic reorganization of the parenchymal matrix. The stability of

the LD measurements, 37 ± 4% on average across all batches, suggested that the longitudinal vascular fibres maintain a certain elastic integrity. Therefore, although the drying was accelerated by the PEF application, this pre-treatment did not induce macroscopic collapse along the major axis. These results are consistent with those reported for peaches and apples, where high-intensity PEF mainly amplifies radial deformations while leaving the length of the fruit almost unchanged [45]. On the other hand, statistical analysis indicates that ultrasound had no significant effect on contraction in any measurement direction (LD, TD, THICK) and that the PEF × US interaction was not significant ($p \geq 0.05$). This fact could indicate that the acoustic waves, leaves no traces on the cellular microarchitecture that modify the shrinkage pattern of samples during drying [27,46].

Overall, the morphological response of dehydrated apricots showed that the management of electro-permeabilizing energy could be the main tool for modulating the dimensional collapse profile of samples as it can directly affect the final texture of the product. This would in accordance with the hypothesis of greater tissue connectivity and the formation of oriented micro-fractures induced by the electric field [47].

3.3.2 Antioxidant characteristics

- **Total Polyphenol Content (TPC)**

The drying conditions considered affected the TPC content of apricots samples after drying (Figure 4).

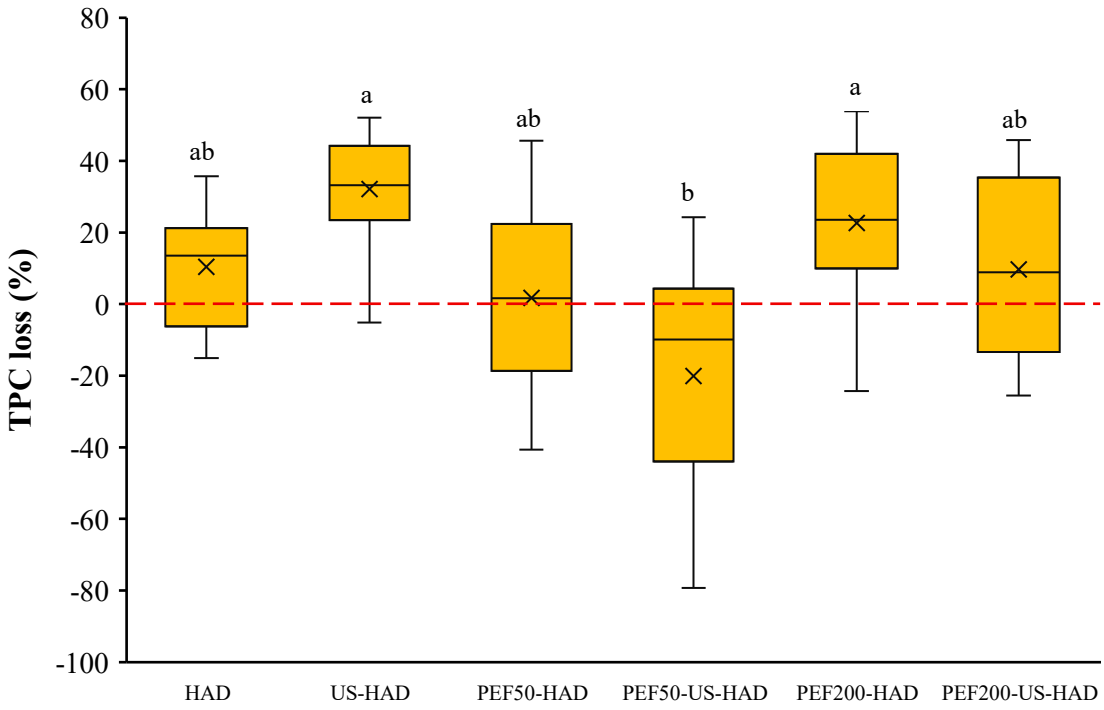

Figure 4 Total phenolic content (TPC) loss (%) of dried apricot without and with apply a PEF pre-treatment with different number of pulses (50, PEF50; 200, PEF200) and without (HAD) or with (US-HAD) ultrasound

application during drying. Letters represent statistical significance between treatments. Different letters indicate significant differences.

Hot air dried (HAD) apricots retained a substantial portion of their polyphenols. Expressed as a percentage change relative to the fresh product (negative values = increase), HAD showed a TPC loss of 10.4 ± 15.8%, providing the reference value in Figure 4. Ultrasound applied during drying intensified the loss (US-HAD 32.1 ± 15.3%). In contrast, PEF pre-treatments improved retention. PEF50-HAD was substantially comparable to the fresh product (1.8 ± 27.0% loss), while PEF200-HAD performed worse (22.7 ± 26.2%). Notably, the combination of moderate PEF with ultrasound produced a net gain. PEF50-US-HAD recorded an average of −20.1 ± 12.7%, which is approximately 20% higher TPC than the fresh product. The highest intensity combination was intermediate, with PEF200-US-HAD recording an average of 9.7 ± 26.4%. Statistical analysis confirmed a treatment effect ($p<0.001$) with PEF50-US-HAD differing significantly from US-HAD and PEF200-HAD, but being statistically indistinguishable from HAD, PEF50-HAD and PEF200-US-HAD. Overall, moderate PEF best preserved, or even improved, TPC, while ultrasound alone promoted losses. The PEF × US interaction depended on PEF intensity, with a beneficial effect only at 50 pulses. Overall, moderate PEF preserved or even improved TPC, while ultrasound alone promoted losses. From a mechanical point of view, moderate PEF probably improves electroporation-induced phenol release and reduces heat exposure, while ultrasound alone promotes leaching. Treatment with higher PEF (200 pulses) and the application of ultrasound may excessively permeabilise the tissues and partially offset the protection provided by PEF.

- **Antioxidant Capacity (FRAP)**

Figure 5 shows the percentage loss of iron reduction antioxidant power (FRAP loss %) for the same samples. This test integrates the combined activity of all redox-active compounds, thus providing a comprehensive picture of the antioxidant potential that survives the drying phase.

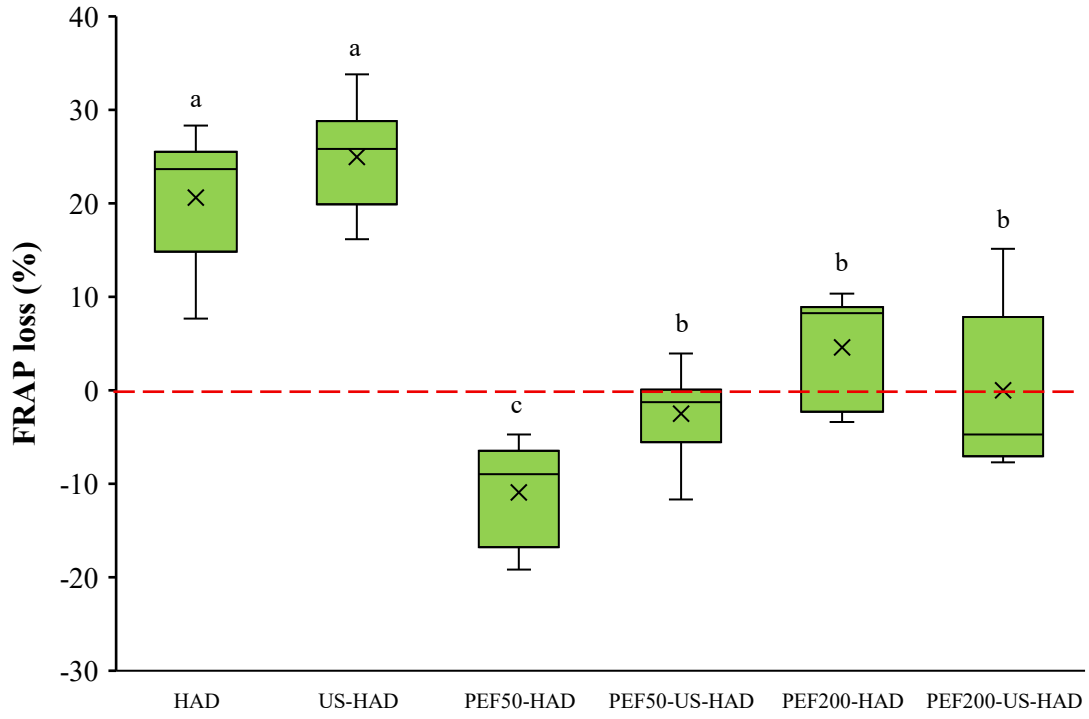

Figure 5 Antioxidant capacity (FRAP) loss (%) of dried apricot without and with apply a PEF pre-treatment with different number of pulses (50, PEF50; 200, PEF200) and without (HAD) or with (US-HAD) ultrasound application during drying. Letters represent statistical significance between treatments. Different letters indicate significant differences.

Antioxidant activity (FRAP) responded to treatments in a distinct but consistent manner with the trend observed for phenols. Expressed as a percentage change relative to fresh (negative values = increase), conventional drying (HAD) resulted in an average loss of 20.6 ± 6.5%, which increased to 25.0 ± 5.4% with ultrasound ($p \geq 0.05$). PEF pre-treatments mitigated the decline. In fact, with PEF50-HAD, a gain in antioxidant activity of −10.9 ± 5.4% was observed, while PEF200-HAD limited the loss to 4.6 ± 5.7%. The PEF x US combinations attenuated the benefits, bringing values close to fresh with PEF50-US-HAD showing values of −2.5 ± 4.7% and PEF200-US-HAD values of −0.0 ± 8.8%. Statistical analysis confirmed a significant effect of the treatment ($p < 0.001$). In fact, the analysis showed that PEF50-HAD and PEF50-US-HAD differ from HAD and US-HAD, while PEF50-US-HAD, PEF200-HAD and PEF200-US-HAD do not differ from each other. In mechanistic terms, moderate PEF (50 pulses) likely promotes the release/extraction of antioxidants and reduces exposure to oxygen and heat. Ultrasound, alone or in combination, tends to negate this advantage. From a mechanistic point of view, these results can be understood by the opposing influences of cell disruption techniques on antioxidant retention. These results can be understood by the opposing influences of cell disruption techniques on antioxidant retention.

PEF creates electroporation in tissues, accelerating drying and reducing thermal oxidative damage to polyphenols [45,48,49]. In fact, PEF-treated dried apricots in this study

maintained higher phenolic levels and showed improved antioxidant parameters, consistent with reports that PEF pre-treatment significantly increases TPC and antioxidant activity in dried fruit [50–52]. Additionally, PEF inactivated oxidative enzymes (e.g. peroxidase) in apricot [15], which likely contributed to the preservation of polyphenols during drying. Ultrasound, on the other hand, has a dual effect. On the one hand, it can reduce drying time and, on the other hand, protect certain heat-sensitive compounds [53]. However, when applied as a pre-treatment by immersion, they also cause cavitation-induced cell rupture, which can lead to the leaching of water-soluble antioxidants and promote their degradation (particularly ascorbic acid) [26]. This could explain why US-HAD samples suffered greater losses in both TPC and FRAP compared to other methods. In summary, the best preservation of antioxidants was achieved with moderate PEF pre-treatment (50 pulses) without ultrasound, which minimized the loss of polyphenols and maintained a good level of the fruit's original antioxidant capacity. This result emphasizes that PEF pre-treatments can effectively preserve functional bioactive compounds in air-dried apricots, as observed in similar studies [10,17,54], while ultrasound pre-treatment alone can compromise antioxidant retention if not carefully managed [55–57].

3.3.3 Colour

The influence of drying conditions on the colour of samples was evaluated thought the estimation of ΔE. This parameter condenses the combined shifts in L*, a* and b* induced by processing into a single metric taking as reference the values measured in the fresh fruit. Values below 5–6 lie below the just-noticeable threshold for most consumers, whereas higher values signal progressively greater discoloration. Thus, it was observed that the sample color was significantly affected by the drying (Figure 6).

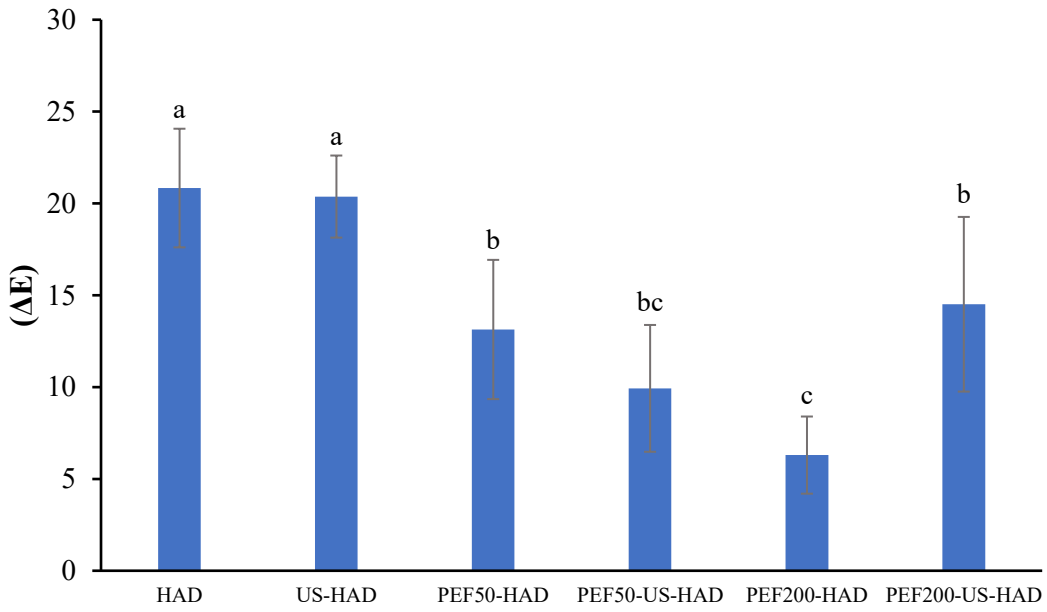

Figure 6 Total colour difference (ΔE) between fresh and dried apricot without and with apply a PEF pre-treatment with different number of pulses (50, PEF50; 200, PEF200) and without (HAD) or with (US-HAD) ultrasound application during drying. Letters represent the statistical significance of the parameter between treatments. Different letters indicate significant differences.

Samples subjected to conventional hot air drying (HAD) showed a ΔE of 21 ± 2, indicating a marked colour change compared to fresh apricots. A ΔE greater than 21 corresponds to a pronounced visible difference from the original colour, implying that HAD alone caused significant colour degradation. This value serves as a baseline for colour change, against which any improvements due to other treatments can be evaluated. The application of low-frequency ultrasound during drying (21 kHz, 20.5 kW m^{-3}) did not significantly affect the overall colour difference of the samples. Apricots dried with US showed a ΔE of 20.1 ± 1.6, which was not statistically significant from the HAD control (21 ± 2, $p = 0.4$). The colour of the dried product remained substantially unchanged, as in the HAD treatment alone.

Unlike ultrasound, PEF pre-treatment had a highly significant impact on colour preservation ($p < 0.01$). Increasing the intensity of PEF before drying led to progressively lower ΔE values, i.e. the colour of the dried product was closer to that of the fresh fruit. With moderate PEF (50 pulses), ΔE decreased to 13.1 ± 1.2, approximately −38% compared to the control (HAD without pre-treatment). With intense PEF (200 pulses), ΔE was 6.2 ± 0.9, approximately −70% compared to the control. This clear inverse relationship between PEF intensity and ΔE indicates that stronger electroporation preserved the original colour of the apricots much better, likely by reducing enzymatic browning and pigment degradation during drying.

The effect of the PEF x US combination depended on the intensity of the PEF. With moderate PEF (50 pulses), the addition of US provided a further improvement in colour retention. The

ΔE decreased to 9.9 ± 1.1 for the combined treatment (PEF50 x US), compared to 13.1 ± 1.2 with PEF50 alone. This ΔE of 9.9 is lower than that obtained with PEF50 or US applied individually, indicating a moderate synergistic effect at intermediate electroporation intensities. In contrast, with high-intensity PEF (200 pulses), the combination with US was less favourable for colour preservation. PEF200-US gave a ΔE of 14.4 ± 1.3, significantly higher (i.e. worse colour retention) than PEF200 alone (6.2 ± 0.9), although still lower than treatments without PEF (ΔE=20). This result suggests that, at very high levels of electroporation, the mechanical stress induced by ultrasound may attenuate some of the protective benefits of PEF on colour. Tissue that has been extensively permeabilised by PEF200 may be more susceptible to pigment leakage and/or accelerated oxidative browning when further agitated by US, partially negating the advantages of more intense PEF.

From a mechanistic standpoint, the superior colour retention at higher PEF intensities can be attributed to several factors that can contribute to the preservation of natural pigments. Thus, the faster moisture removal could limit the duration of the exposure to heat and oxygen, thereby stabilizing carotenoids and slowing enzymatic browning. Similar results have been previously reported for other products [55]. Thus, Rybak et al. [54] reported that PEF pre-treatment of red peppers accelerated freeze-drying and improves the colour of the dried product, with a significantly lower ΔE than drying without pre-treatment [58,59]. Additionally, PEF-induced electroporation may partially inactivate enzymes, such as polyphenol oxidase, responsible of degradation browning reactions [60].

By contrast, the application of power ultrasound alone, although shorten the processes, rarely improves colour of dried fruits. The induced sponge effect and the generation of microcrack, can enhance the driving of pigments toward the surface, where they are degraded by oxidation and darkening the product [61]. Then the shortening of the process induced by ultrasound did not compensate the enhancement of colour degradation, being this reflected in the negligible difference found between HAD and US-HAD apricots. When PEF was combined with ultrasonic-assisted drying, the effects induced were also the result of the interaction of the individual effects. Thus, in PEF50-US-HAD the moderate structural PEF effects combined with the significant reduction of drying time induced by ultrasonic contribute to a better preservation of the colour than the application of both technologies individually. On the contrary the greater structural effects induced by PEF in PEF200-HAD-US combined with ultrasound effects, worsen the individual application of PEF in these conditions. In fact, some studies point out that combining very intense PEF with ultrasound can be counterproductive because an excessive cell rupture can lead to produce microcracks

and intensity Maillard browning, compromising the colour benefits of PEF pre-treatment [26,62]. Overall, the obtained results indicated that the PEF, rather than acoustic one, was the key factor in preserving the colour of dried apricots. Thus, at the more intense treatment tested, 200 pulses, the ΔE remained close to the threshold of 5-6 units, that define the limit of the perception of colour difference of human eye. Therefore, it could be possible to produce dried apricots with an appearance much more like fresh fruit.

4. Conclusion

This study evaluated the combined effect of pre-treatment with pulsed electric fields and ultrasonic-assisted drying on drying kinetics and quality of apricots. The application of US during drying affected both external and internal mass transfer resistance, increasing the external mass transfer coefficient (k) and the effective diffusivity (D_{eff}). On the contrary PEF effects were mainly focused on internal resistance, the greater the number of pulses applied the higher the D_{eff} identified. The combination of PEF and US provided the shortest drying process indicating a synergistic effect of both technologies.

The application of PEF affected the shrinkage of samples being significant ($p < 0.05$) its influence on transverse (TD) and thickness (THICK) directions while the longitudinal (LD) one remained stable. No appreciably changes in the final geometry of samples was observed due to the application of ultrasound. For TPC, moderate PEF preserves or even improves phenols, while ultrasound alone promotes their loss. FRAP trends mirror those of TPC but are more moderate: moderate PEF improves or maintains capacity almost equal to that of fresh products, while higher PEF mainly limits losses. Ultrasound alone generally reduces FRAP, and the PEF×US interaction does not show robust synergy beyond the moderate PEF case. In terms of colour, PEF pre-treatments contributed to a significantly better preservation of colour, the higher the intensity of treatment, the lower the colour changes induced. On the contrary, the influence of US was not significant. However, the lower colour change was observed when combining a moderate intensity PEF treatment and the ultrasonic-assisted drying. Overall, the adequate combination of PEF pre-treatment and ultrasonic-assisted drying can provide a fast-drying process and a better preservation of quality attributes of apricots.

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CHAPTER 3

Effect of Hot Air-Drying Technique on the Quality and Stability of Blood Orange Slices in Modified Atmosphere Packaging

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Abstract

The choice of time/temperature combination is critical for ensuring microbiological stability and retaining the characteristic taste of dried blood orange slices. The aim of this study was to investigate the capability of hot air-drying technique to maintain the quality characteristic of dried blood orange slices stored in modified atmosphere pack- aging (MAP). Hot air-drying at 70 ◦C for 12 h preserved shrinkage without altering the longitudinal diameter, though thickness was significantly reduced, especially in samples with passive MAP. Increased hardness and masticability were noted due to water removal, with active MAP maintaining high hardness and colour integrity up to 100 days of storage (D100). Sensory analysis revealed differences in colour intensity and flavour between active and passive MAP-stored slices. Headspace solid-phase microextraction (HS-SPME) chromatography identified key chemical compounds contributing to aroma and flavour, highlighting the complex interplay between temperature, storage conditions, and volatile organic compounds production. The study demonstrates that drying combined with MAP storage enhances organoleptic qualities and nutritional value, offering a method to produce a healthy, tasty, and visually appealing snack.

Keywords: convective hot-air drying; blood orange slices; sensory; microbial safety; aroma

1. Introduction

Sweet orange (*Citrus sinensis* (L.) Osbeck) is a subtropical evergreen fruit tree belonging to the Rutacee family. It is likely native to Southeast Asia and has since spread worldwide [1]. The largest producer of oranges globally is Brazil, while Spain, Italy,

and Greece are the most important growers in Europe [2]. In Italy, Sicily is the main producer of sweet oranges, especially blood oranges (Moro, Tarocco e Sanguinello varieties) [3,4] due to the climatic conditions that promote the synthesis of anthocyanins [5]. Blood oranges are highly valued for their juiciness, aroma, and are also a natural source of vitamin C, hydroxycinnamic acids, and anthocyanins, which are primarily responsible for their distinctive red colour [6–9]. The cyanidin-3-glycoside (C3G), the main component of blood orange anthocyanins, has high antioxidant capability, opposes the activity of free radicals, and appears to have anticancer properties [10,11]. Oranges are mainly consumed as fresh fruit or as byproducts, such as juice, but they can also be frozen and processed into jams, canned, or dried fruit [12].

Among orange preservation techniques, particularly drying, numerous studies have been conducted on Navel late [13] and Valencia [14] sweet orange varieties, finger citron [15], kumquat [14], and lemon [16]. However, there is no research on the effects of drying on blood oranges. Various drying techniques exist [17,18], but convective hot-air drying is the most widely used in the food industry [12]. Drying not only extends the shelf life of food but also increases its value [19] by making it available throughout the year [14]. In recent years, researchers have focused on creating healthy foods from waste products along the supply chain [20]. In the drying process, the main objective is to remove moisture up to 14–16% as quickly as possible at a temperature that does not affect the flavour, texture, and colour of foods, while ensuring microbiological stability [13,21]. If the temperature is too low, microorganisms may grow before the food is properly dried; conversely, if the temperature is too high and the moisture is too low, food may harden on the surface and brown, reducing the market value. Studies suggest that temperatures between 37 ◦C and 71 ◦C effectively kill bacteria and inactivate enzymes [22–25]. Furthermore, drying reduces the weight of products, as well as their transport and storage costs [17]. In addition, a critical factor in maintaining the shelf life of dried products is the method of storage. In recent years, the preservation of minimally processed and dried fruits has garnered significant attention, particularly concerning modified atmosphere packaging (MAP). MAP is essential for extending shelf life by reducing the respiration rate of fruits, thereby maintaining their quality over time [26]. Modified atmosphere must be considered to maintain optimal conditions due to reducing the respiration rate of the fruit [27,28]. The main used gases in modified atmosphere packaging (MAP) are CO_2, O_2, and N_2, which affect the density of bacterial and viral populations, reduce the possibility of fruit slices sticking [17,29], and prevent the packaging material from collapsing onto the product without altering

the food's sensory characteristics [30]. Reducing oxygen and increasing carbon dioxide decreases the respiratory activity of the product, thereby slowing down spoilage [31]. However, the choice of each or their combination depends on the product to be stored. Recent studies on MAP in fruits like strawberries, apples, and other dried citrus fruits have shown its positive impact on preserving colour, aroma, and texture, making it a vital aspect of fruit preservation [32]. Furthermore, MAP has been shown to be effective in maintaining the volatile compounds of fruits, which contribute to their aroma and flavor, even after prolonged storage periods [33].

The aim of the present study was to investigate the capability of the hot air-drying technique to maintain the physicochemical, microbiological, and sensory characteristics and volatile compounds, in dried blood orange slices stored in MAP conditions.

2. Materials and Methods

2.1 Vegetal Material

Fifty kilograms of ripe blood oranges (*Citrus sinensis* L. Osbeck) from the Moro, Tarocco, and Sanguinello varieties were randomly hand-picked from 15 trees grown under open-field conditions in the ancient orchard (38°06′ 25″ N–13°21′07″ E) near the Department of Agricultural, Food and Forestry Sciences at the University of Palermo (Sicily, Italy). The fruits were selected for homogeneity and absence of defects (according to the standards of Codex Alimentarius [34]), using peel colour as a ripening index. Then, they were transported to the post-harvest laboratory and stored for 2 days at room temperature (20±5 °C) [35]. Afterwards, the fruits were washed under tap water to remove field residues.

2.2 Experimental Design

Fruits were sanitized in a solution of NaClO 2% *v*/*w* at 5 ◦C for 20 min [36]. Afterwards, the blood oranges were peeled, sliced into pieces with a thickness of 5±0.6 mm, and placed in a convective hot air dryer (Premium Stainless Steel Food Dehydrator, Cosori, Italia). According to the other authors [13,16], to achieve dried products that remain microbiologically stable, a residual moisture content of about 14–16% is necessary. Several preliminary tests were conducted to determine the optimal time/temperature combination. At 70 ◦C for 8 h [30], the ideal moisture into the sample was not achieved. Therefore, we continued the dehydration process until a constant weight (2 g) was reached, following the Official Methods of Analysis [37]. The final moisture content was measured at 14.6%, in line with previous studies [12]. As a result, blood orange

slices were dried at 70 ◦C for 12 h. After drying, blood orange slices were stored in polyamide/polyethylene bags (80% PA/20% PE, thickness 90 µm, volume 500 cm3, oxygen permeability 47.6 cm2/ (m2 day atm), and water vapor transmission rate 3.9 g/ (m2 day atm). The slices were then subjected to Modified Atmosphere Packaging (MAP) technology. They were stored at two different gas concentrations: 100% N_2 (active MAP) and 21% O_2 + 0.04% CO_2 + 78% N_2 (passive MAP). The dried slices were divided as follows: 50 g of sample per bag × two treatments (active and passive MAP) × 5 days of analysis (D0 immediately after drying, D25, D50, D75, D100) × three replicates. All bags were sealed using a digitally controlled packaging machine (VM 16 Orved S.p.A, Musile di Piave, Venezia, Italy) and then stored at room temperature (20±5 ◦C) for 100 days.

2.3 Physicochemical Analysis

The following analyses were carried out on a sample of 25 fresh slices before the dehydration process:

- weight (g), using a precision electronic scale (Gibertini EU-C 2002 RS, Novate Milanese, Italy);
- longitudinal diameter (LD-mm) and thickness (mm), using a digital calibre (Turoni TR53307, Forlì, Italy);
- hardness (N) using a TA-XT plus texture analyser (Stable Micro Systems, London, UK) with 50 N load cell. Tests were conducted using a three-point bending method with a span setting of 4 cm, where the dried orange slices were deformed until they broke. The hardness (N) value was calculated from the force/displacement curve using Texture Expert Version software. The pre-test speed was set at 5 mm/s, the test speed at 1 mm/s, and the post-test speed at 5 mm/s. The penetration distance was set at 4 mm for a force of 5 g, using the P/4 cylinder;
- colour was carried out with a digital colourimeter (CR-400, Konica Minolta, Tokyo, Japan) based on the CIE L*a*b* system (CIE L*a*b*: lightness (L*); redness/greenness (a*); yellowness/blueness (b*)). Additionally, the browning index (*BI*) of the dried orange slices was calculated using the following Equation (1) of Ruangchakpet and Sajjaanantakul [38].

$$BI = \left(\frac{x-1.75L^*}{0.17}\right) \times 100 \qquad (1)$$

where $x = \left(\frac{a^*+1.75L^*}{5.645L^*+a^*-0.3012b^*}\right)$

After the dehydration process, the same analyses were carried out on a sample of 25 dried slices per treatment at D25, D50, D75, and D100.

2.4 Microbiological Analysis

Blood orange slices obtained immediately after dehydration at 70 ◦C for 12 h, as well as samples taken at specific time points (D25, D50, D75, D100), underwent the decimal serial dilution procedure [39]. Spoilage and pathogenic populations were enumerated using the following media: Plate Count Agar (PCA) for total mesophilic microorganisms and total psychotropic microorganisms, incubated for 2 d at 30 ◦C and for 7 d at 7 ◦C, respectively; Pseudomonas Agar Base (PAB) added with CFC supplement for *Pseudomonadaceae*, incubated for 2 d at 25 ◦C; Violet Red Bile Glucose Agar (VRBGA) for *Enterobacteriaceae*, incubated for 1 d at 37 ◦C; Baird Parker (BP) agar coagulase-positive *staphylococci*, incubated for 2 d at 37 ◦C; Hektoen Enteric Agar (HEA) for *Escherichia coli* and *Salmonella* spp. incubated for 1 d at 37 ◦C; Listeria Selective Agar Base (LSAB) for *Listeria monocytogenes*, incubated for 1 d at 37 ◦C; Yeast extract Peptone Dextrose (YPD) agar for yeasts and filamentous fungi, incubated for 2 d and 7 d at 25 ◦C, respectively. Detection of *L. monocytogenes* and *Salmonella* spp. was performed on 25 g of dried blood orange slices samples following the ISO 11290-1 [40] and ISO 6579-1 [41] guidelines, respectively. These analyses were performed in triplicate using media purchased from Oxoid (Basingstoke, UK).

2.5 VOCs Analysis

Headspace solid-phase microextraction (HS-SPME), coupled with gas chromatography-mass spectrometry (GC-MS), was employed for the identification and quantification of volatile organic compounds (VOCs) in blood orange slices treated as above described. Standard organic compounds were injected throughout the experimental sequence using the same extraction and purification procedure to detect and evaluate each compound. The identification of each compound was facilitated using Kovats indices (KI). KI values are based on the retention time normalized to the adjacent eluted n-alkanes. KI values are independent of the method of analysis; therefore, they were useful for the identification of unknown compounds. An Agilent 5890 GC system coupled to an HP 5973 quadrupole mass spectrometer was used for gas chromatographic analyses. An HP5-MS column (5% diphenyl - 5% dimethyl polysiloxane 30 m × 0.2 mm, 0.25 µm film, J & W Scientific, Folsom, CA, USA) was used. Water and oxygen traps (Supelco, Merk Life Science, Milano, Italy) were installed on the carrier gas lines, and ultra-high-purity helium was used as carrier gas at a flow rate of 1 mL min-1. The oven temperature was maintained at 40 ◦C for 5 min, then was

increased at a rate of 5 ◦C/min to 220 ◦C and 10 ◦C/min to 280 ◦C and held constant for 10 min. Molecular mass spectra were recorded using an ionization voltage of 70 eV and an ion source temperature of 220 ◦C [42]. The samples were analysed by the HS-SPME-GC-MS method using a PDMS-CAR-DVB fibre (Supelco, ITA). The extraction and purification process involved exposing the fibre to 3 g of treated blood orange slices in a 40 mL vial with a silica septum, held at 70 ◦C for 20 min. Following this, the fibre was manually inserted into a GC inlet port equipped with a specialized glass liner designed for SPME injection (0.75 mm id). Fiber was removed from the gas chromatograph inlet port after 3 min at 250 ◦C using the splitless injection mode. The compounds were confirmed by comparison of mass spectral data with those of authentic reference compounds. When standards were unavailable, the identification of components was conducted via mass spectrum matching utilizing the NIST 11 mass spectral library collection.

2.6 Sensory Evaluation

Ten semi-trained judges with extensive experience in sensory evaluation of food performed a liking test on previously prepared dried blood orange slices. During the preliminary session, 15 qualitative descriptors were chosen for sensory profiling [43,44], generated based on the frequency of citation (≥60%) and rated on a hedonic scale from 1- "absence of the descriptor" to 9- "highest descriptor intensity". Dried blood orange slices stored in active and passive MAP were evaluated at the end of the storage period (D100) by 99 consumers aged 18–60 years (37% women and 63% men) [45]. Consumers were given two slices from each MAP sample (passive and active) in coded and randomly distributed cups. They were trained to taste and score (1 to 9) the following descriptors: colour intensity, orange odour, honey odour, unpleasant odour, taste acceptance, orange taste, caramel taste, sweet, acid, bitter, unpleasant taste, gummy, masticability, fibrousness, overall acceptance. The test was carried out in individual booths under white light at room temperature in the Sensory Analysis Laboratory at the University of Palermo (Italy). The presentation order and the identification codes were randomized using Smart Sensory Box 2.11.4 software (Smart Sensory Solutions S.r.l., Sassari, Italy). Water was provided to cleanse the palate between the samples. After completing their tasting evaluations, consumers were asked how often they consume dried fruit by selecting one option from the following list: "almost every day", "several times a month", "very sporadically", or "never". They were then asked, using a dichotomous scale (yes/no) [46], if they "would buy this product if it were available in your usual supermarket".

3. Results and Discussion

3.1 Physicochemical Analysis

Fruits and vegetables have a highly porous and hygroscopic structure. One of the most important physical changes that food undergoes during drying is the reduction of its volume, typically defined as a shrinkage of the food product [48]. This phenomenon causes physicochemical changes and results in an increase in flesh texture and consumer preferences [49–52]. As shown in Table 1, there are no significant differences ($p < 0.05$) between fresh and dried orange slices in terms of LD and BI. While, after the drying process, the thickness decreases of 0.70 mm and the hardness increases of 8.51 N, due to the normal shrivelling of the flesh and the associated depolymerization of cell wall constituents, including pectin [53]. Similar results on hardness were obtained by Roppolo et al. [54], who observed an increase of 10 N on pescabivona fruits after the drying process.

Table 1. Longitudinal diameter (LD—mm), thickness (mm), hardness (N), browning index (BI), and soluble solids content (SSC—◦Brix) of fresh and dried orange slices. Results indicate the mean values ± standard deviation (S.D.) of the replicates (n = 3). Asterisks indicate significant differences between fresh and dried orange slices with * for $p < 0.05$, ** for $p < 0.01$; ns indicates no significant differences between the same.

Sample	Longitudinal Diameter (LD—mm)	Thickness (mm)	Hardness (N)	Browning Index (BI)
Fresh	47.70±2.02	5.48±0.68	5.67±1.72	30.22±2.58
Dried	47.67±2.49	4.78±0.71	14.18±1.25	31.21±2.85
T-Test	ns	*	**	ns

Therefore, the chosen time/temperature combination was effective in maintaining the same LD parameter after drying, according to Mayor and Sereno [55], who observed, under conditions of high temperature and reduced hot air exposure, different rates of water diffusion from the inside to the outside of the flesh. In this way, strong moisture gradients are formed in the material, which inhibit shrinkage during drying [55]. Instead, after the drying process LD decreased steadily ($p \geq 0.05$) up to D100 for both MAP treatments (Figure 1). On the last day of analysis (D100), the dried orange slices lost 7.80% in passive MAP and 8.32% in active MAP. However, although storage in active MAP showed no significant differences ($p \geq 0.05$) compared with passive MAP treatment during 100 days of storage, the presence of N_2 seems to have maintained the initial characteristics of the bags, according to Passafiume et al. [56].

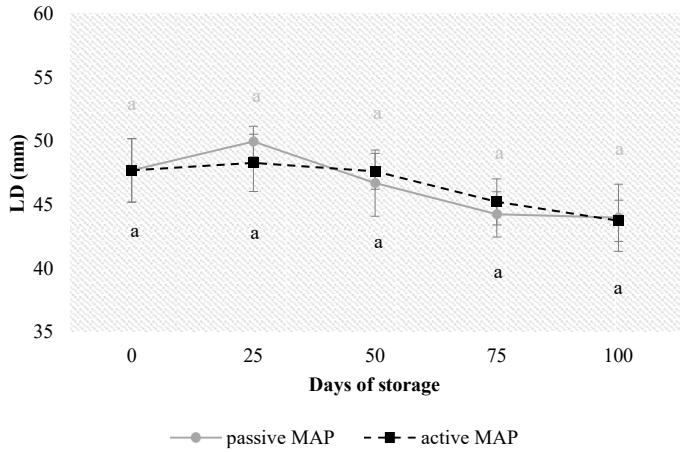


Figure 1. Longitudinal diameter expressed in mm for dried blood orange slices during 100 days of storage in active and passive MAP. Results indicate the mean values ± standard deviation (S.D.). Different letters and different colours of the letters represent significant differences ($p < 0.05$) on individual treatments for each day of storage; no asterisk indicates no significant differences between treatments.

Regarding the thickness of blood orange slices, Figure 2 shows the variation in slice thickness during the storage period. Statistically different behaviours were observed between passive MAP and active MAP at D25 ($p < 0.05$) and at D50 ($p < 0.01$). For passive MAP at D25, a higher value than the initial one is shown, probably due to some little difference in the cut of slices. The presence of N_2 in active MAP seems to have slowed down the thickness loss, showing an almost linear trend throughout the storage period and reaching (D100) a value of 3.46±0.88 mm. While in passive MAP, the thickness decreased much more rapidly, reaching a value of 2.87±1.06 at the last day of storage.

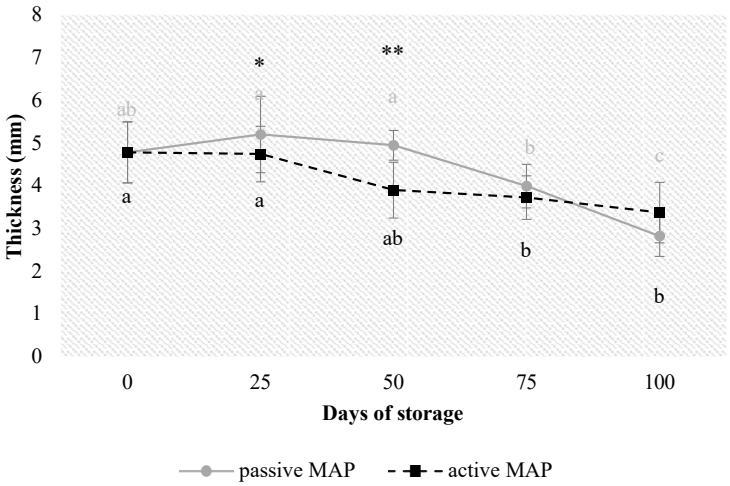


Figure 2. Thickness expressed in mm for dried blood orange slices during 100 days of storage in active and passive MAP. Results indicate the mean values ± standard deviation (S.D.). Different letters and different colours of the letters represent significant differences ($p < 0.05$) on individual treatments for each day of storage; asterisks indicate significant differences between MAP treatments with * for $p < 0.05$, ** for $p < 0.01$; no asterisk indicates no significant differences between treatments.

To obtain a moisture content of 14–16%, a loss of thickness occurred; however, our primary focus was microbiological stability. The decrease of thickness during storage may be due to the moisture loss that causing the pores of the slices to collapse [57]. Nevertheless, this change did not influence consumers' liking during the sensory evaluation, as confirmed by

the scores obtained on the texture descriptor. Therefore, the shrinkage of blood orange slices, as recorded through analytical tools, remains a negligible factor for positive consumer perception. Similar results were obtained by Llorca et al. [45] on the acceptability of dried persimmon slices. Zotarelli et al. [58] observed thickness losses of 17% and 23% on banana and mango slices dried at temperatures of 60 ◦C, respectively, to a residual moisture content of 12%. The short drying times likely preserved the cellular structure of the flesh, reducing the loss of thickness. This is because such processes could result in sample thickness reduction due to the moisture gradient passing outward in the form of water vapor. This can cause the collapse of capillaries responsible for transporting fluids inside, leading to irreversible structural changes [49]. Furthermore, during storage, the loss in thickness can be attributed to the characteristics of the packaging used. In fact, the 80% PA/20% PE blend maintained some water vapor permeability, leading to a reduction in thickness. In fact, the water vapor permeability of packaging plays a key role in food preservation, especially in maintaining the physical characteristics of food [50,51].

Regarding hardness, the data set (Figure 3) indicates that in both MAP treatments there was a significant loss of hardness from D0 to D25 (about 2.60 N). During the 100- day storage period, active MAP was more effective as it lost only 9.98% of hardness (at D100) compared to the passive MAP treatment, which decreases by 45.39%. Statistically significant differences were observed between the two treatments starting at D75. The loss of hardness represents a decline of quality for dried products. Varying hardness often affects the preservation of flavour [52], which may contribute to consumer non-acceptance, especially when consumed at the end of its shelf life.

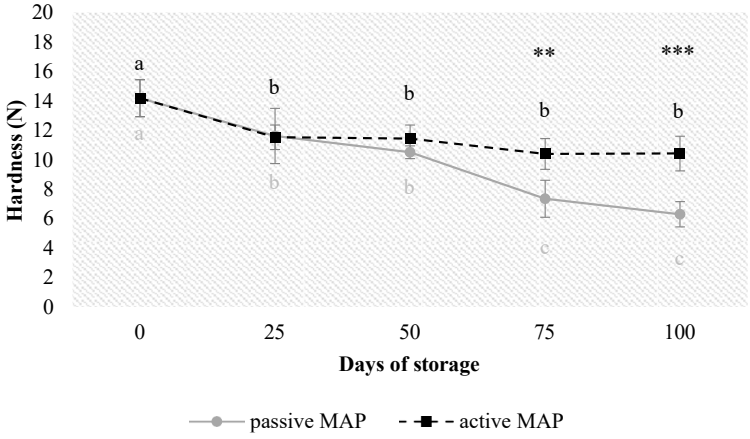


Figure 3. Hardness expressed in N for dried blood orange slices during 100 days of storage in active and passive MAP. Results indicate the mean values ± standard deviation (S.D.). Different letters and different colours of the letters represent significant differences ($p < 0.05$) on individual treatments for each day of storage; asterisks indicate significant differences between MAP treatments with ** for $p < 0.01$, *** for $p < 0.001$; no asterisk indicates no significant differences between treatments.

This behaviour could be associated with the presence of N_2 in the bags, which seems to maintain the hardness characteristics of the stored samples. Several researchers have shown that N_2 delays the senescence process in dried tomato [59], avocado [60], and loquat fruits [29], thereby keeping the cell structure intact for longer. Additionally, the removal of water from plant tissues causes the movement of water-soluble compounds within the cells, thereby increasing the hardness of the cell walls [61]. This phenomenon not only results in shrinkage of the product but also causes changes in mechanical properties, including texture, which define the quality of fruit products [12,62]. This property is mainly related to the microstructure and intracellular spaces of the dried product and the breaking of cellular bonds during the drying process [63,64]. In fact, dried products are characterized by a "glass-like" state that gives them a solid and crisp texture [65]. Colour is another critical parameter in the consumers' choice of product [66]. It is an important indicator of perceived quality because variations in it are often associated with non-enzymatic browning [67]. Figure 4 shows the browning index (BI) variation in dried blood orange slices during storage. Starting from a value of 33.31 (D0), BI increased for both treatments throughout the storage period. However, it was found that passive MAP remained significantly lower on the last day of analysis (BI = 41.97±2.53) than active MAP treatment (50.11±2.97).

In general, if fruit tissue cells are damaged, oxygen in the air reacts with enzymes on the fruit surface, generating melanin, which is mainly responsible for fruit browning, resulting in loss of economic value [68]. In addition, no flesh decay phenomena were detected, which was also confirmed by sensory analysis. According to a study conducted by Polat [12], the change in sample thickness and cell structure from fresh to dried affects light reflection on orange slices, altering the colour values of the samples. The tissues of dried products have been shown to undergo some degree of shrinkage, resulting in changes in chemical components. Consequently, there may have been a change in the chemical composition of the outer cell wall of the dried product. A similar study [69] explained that the reasons for the colour change are likely attributed to the decomposition of carotenoids and other pigments in the orange slices and the triggering of the Maillard reaction [70].

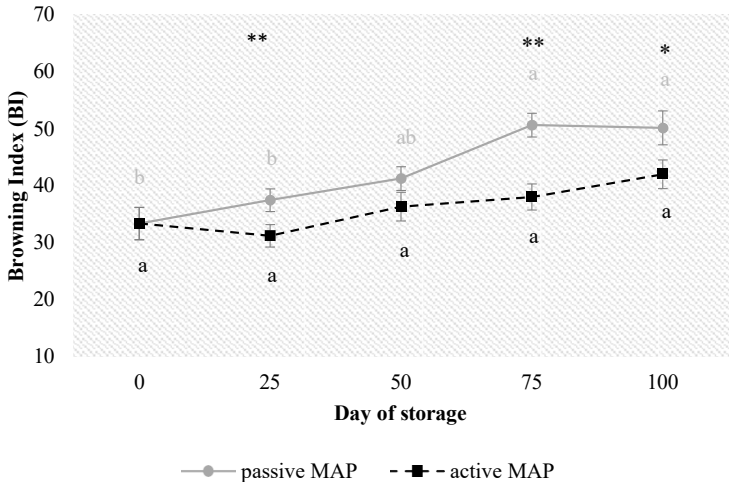


Figure 4. Browning index for dried blood orange slices during 100 days of storage in active and passive MAP. Results indicate the mean values ± standard deviation (S.D.). Different letters and different colours of the letters represent significant differences ($p < 0.05$) on individual treatments for each day of storage; asterisks indicate significant differences between MAP treatments with * for $p < 0.05$, ** for $p < 0.01$; no asterisk indicates no significant differences between treatments.

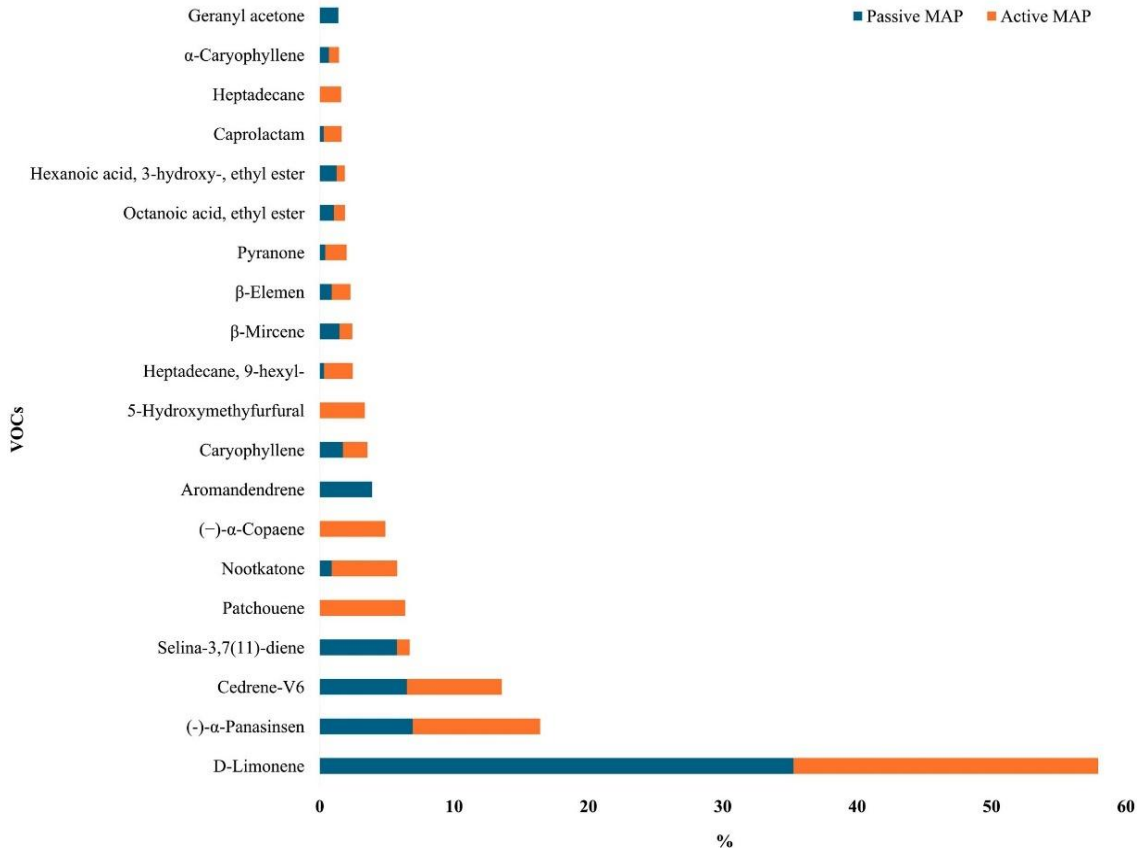
3.2 Microbiological Analysis

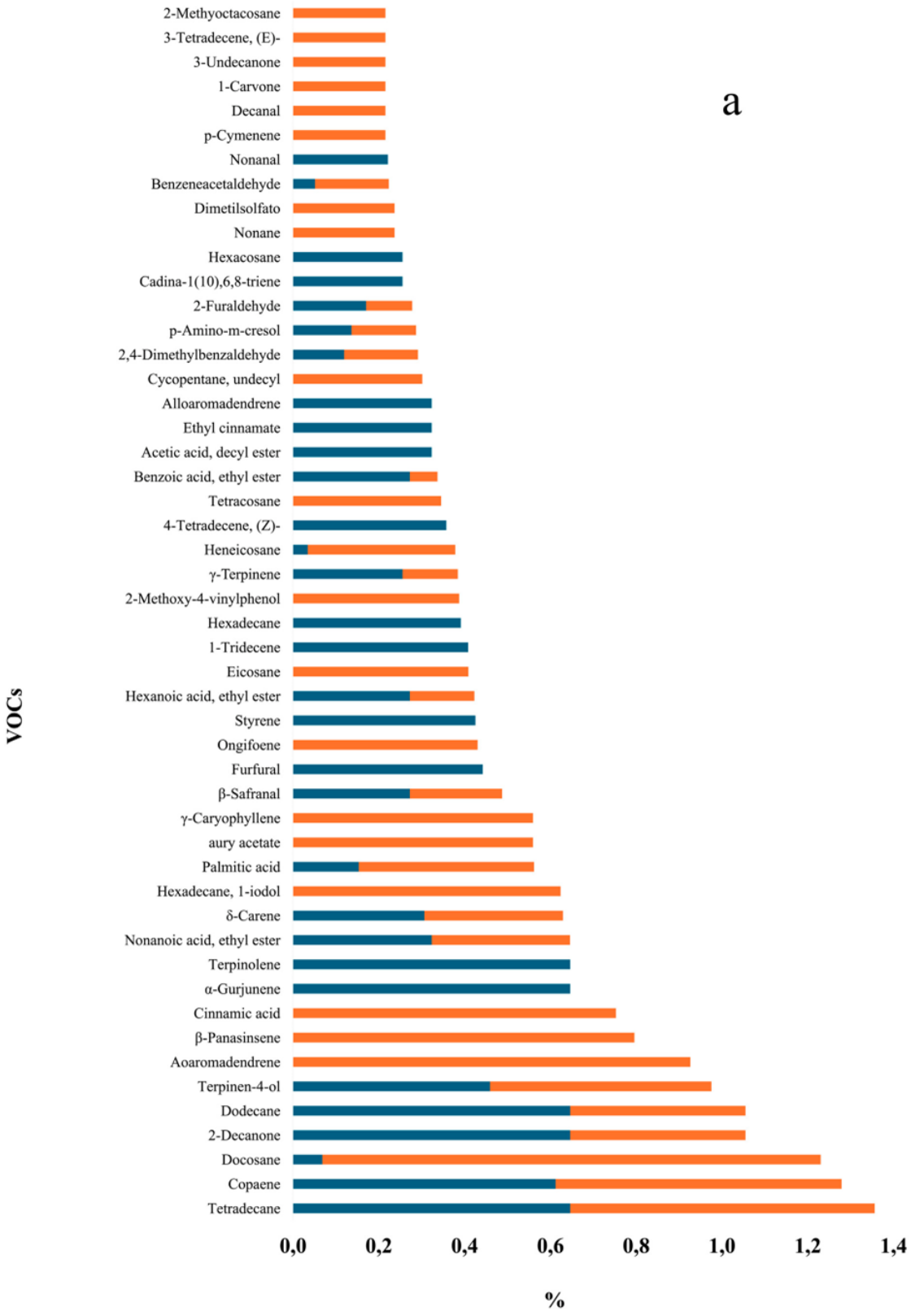
According to the report of the World Health Organization (WHO), despite having low water activity (a_w), dried fruits and vegetables can still serve as carriers for foodborne pathogens [71,72]. Many of these microorganisms can endure on dried fruits and vegetables for extended periods, potentially leading to foodborne illnesses due to their low infectious dose [73]. Therefore, it is crucial to evaluate the microbiological aspects of dried blood orange slices before their introduction to the market. In this study, the presence of the main spoilage and pathogenic microorganisms commonly associated with food of plant origin [74] was investigated by a culture-dependent approach. In all analysed samples, the levels of *Pseudomonas* spp., yeasts, and moulds, known to cause physicochemical alterations in plant-based food products [75], were below the detection limit of <2 Log CFU/g. Throughout the storage period (D0, D25, D50, D75, and D100), regardless of the MAP treatment used, no coagulase-positive *staphylococci*, *E. coli*, *L. monocytogenes*, and *Salmonella* spp. were detected. These findings comply with the microbiological criteria for foodstuff outlined in Commission Regulation 2073/2005 (EC, 2005). However, the absence of all investigated microbial groups on the dried blood orange slices can be attributed to both the drying process [76] and the strict hygiene standards of fruit sanitation and handling applied in this study [57].

3.3 VOCs Analysis

Dried orange slices treated with two different gas concentrations (active MAP and passive MAP) were analysed using the HS–SPME–GC–MS analytical method. The composition and relative amounts of volatile organic compounds (VOCs) are shown in Figure 5. A total of

120 VOCs were identified and quantified: 83 compounds in passive MAP- treated samples and 75 in active MAP-treated samples. Identification was based on chromatographic peak Linear Retention Index (LRI), Kovats' retention index (KI), similarity index over 80%, and reference standards [76]. Terpenes were the most abundant, constituting 68.2% and 64.7% of the total composition in passive and active MAP treatments, respectively. Hydrocarbons were 3.5% and 9%, and esters were 5.2% and 3%, respectively. Aromatic compounds were 2.5% in passive MAP and 3.4% in active MAP. Other compounds, including ketones (0.9% for both active and passive MAP), aldehydes (0.3% and 0.2%, respectively), and alkanes (0.1% and 0.5%, respectively), appeared in smaller proportions, representing 2.9% and 10% of total composition in passive and active MAP. D-Limonene had the highest relative peak area values, followed by (-)-α-Panasinsen and Cedrene-V6 in both treatments. Several studies have reported higher concentrations of terpenes in extracts from fresh orange, orange juice, and orange peel [77]. Terpenes are a large category of organic compounds commonly present in essential oils and resins and contributes to distinctive aroma and taste of various plants and fruits. In passive MAP treatment, D-Limonene made up 35.3%, (-)-α-Panasinsen accounted for 6.9%, and Cedrene- V6 was 6.5%. In active MAP treatment, D-Limonene constituted 22.7%, (-)-α-Panasinsen 9.5%, and Cedrene-V6 7%.




a
2-Methyoctacosane
3-Tetradecene, (E)-
3-Undecanone
1-Carvone
Decanal
p-Cymenene
Nonanal
Benzeneacetaldehyde
Dimetilsolfato
Nonane
Hexacosane
Cadina-1(10),6,8-triene
2-Furaldehyde
p-Amino-m-cresol
2,4-Dimethylbenzaldehyde
Cycopentane, undecyl
Alloaromadendrene
Ethyl cinnamate
Acetic acid, decyl ester
Benzoic acid, ethyl ester
Tetracosane
4-Tetradecene, (Z)-
Heneicosane
γ-Terpinene
2-Methoxy-4-vinylphenol
Hexadecane
1-Tridecene
Eicosane
Hexanoic acid, ethyl ester
Styrene
Ongifoene
Furfural
β-Safranal
γ-Caryophyllene
aury acetate
Palmitic acid
Hexadecane, 1-iodol
δ-Carene
Nonanoic acid, ethyl ester
Terpinolene
α-Gurjunene
Cinnamic acid
β-Panasinsene
Aoaromadendrene
Terpinen-4-ol
Dodecane
2-Decanone
Docosane
Copaene
Tetradecane
VOCs
0,0
0,2
0,4
0,6
0,8
1,0
1,2
1,4
%

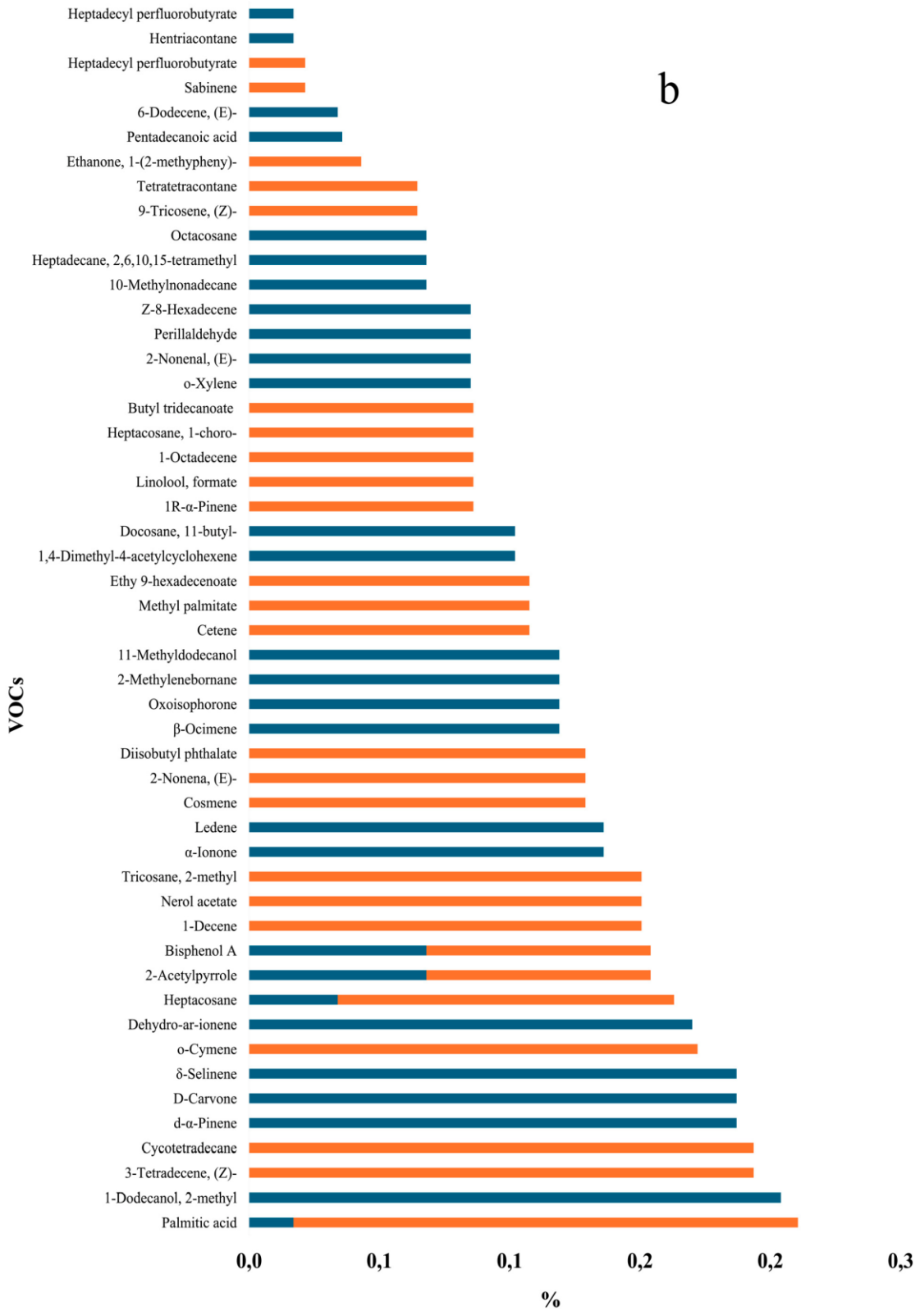


Figure 5. VOCs (%) detected in dried orange slices with Passive MAP and Active MAP treatments. (Panel (**a**) highlights VOC concentrations ranging from 0% to 1.4% while Panel (**b**) highlights those from 0% to 0.3%).

Significant differences in VOC concentrations were observed between the two treatments. For instance, Aromandendrene, Geranyl acetone, α-Gurjunene, and Terpinolene were exclusive to passive MAP-treated samples (3.9%, 1.4%, 4.6%, and 4.6%, respectively), while Patchouene (6.4%), (-)-α-Copaene (4.9%), 5-Hydroxymethylfurfural (3.3%), and

Heptadecane (1.6%) were uniquely found in active MAP-treated samples. Other compounds with lower concentration values, such as β-Myrcene (1.5% and 1%), Octanoic acid ethyl ester (1% and 0.8%), Terpinen-4-ol (0.4% and 0.5%), Hexanoic acid, 3-hydroxy-, ethyl ester (1.2% and 0.6%), and Caryophyllene (1.7% and 1.8%), were identified in both active and passive MAP treatments with different concentration values. The above-mentioned differences highlighted the potential presence of additional sources of VOCs by using active MAP atmosphere that increases the nutraceutical values of process production. Among the alkanes identified, the most abundant were Heptadecane 9-hexyl- with 0.3% and 2.1%, and Tetradecane with 0.6% and 0.7%, in passive and active MAP-treated samples, respectively. In dried blood orange slices, alkanes are originated from the waxy outer layer of the fruit's peel or from lipid-rich components within the orange fruit, and they were present primarily when the fruit reaches maturity [78]. Their presence can contribute to aroma, flavor, and other sensory attributes of dried fruit. Sixteen unsaturated and saturated fatty acid esters were additionally detected in dried blood orange slices. Among these, the most abundant were Octanoic acid-ethyl ester, with concentrations of 1% and 0.9%, Hexanoic acid, 3- hydroxy-, ethyl ester, with 1.2% and 0.6%, and Nonanoic acid-ethyl ester, which measured 0.3% in both passive and active MAP treatments. However, blood orange slices treated with passive MAP treatment exhibited a higher concentration value of esters compared to active MAP treatment, likely attributable to different compositions and types of gases within packaging. Different gas compositions within the passive MAP atmosphere influences esters formation compared to the active MAP atmosphere. Gas composition in passive MAP treatment promotes ester formation, whereas active MAP treatment determines the opposite effect, leading to a decrease in ester formation as described above and shown in Figure 5. Passive MAP treatment determined a higher production of ethylene, which influenced ripening and the formation of volatile compounds such as esters. Oxygen in passive MAP contributes to ester formation reducing the rate of fatty acid oxidation, which is a key step in ester synthesis. Furthermore, carbon dioxide concentration in passive MAP promotes ester formation, while in active MAP treatment, where carbon dioxide is absent, this process is not observed. Indeed, carbon dioxide influences acidity in food products, promoting the reaction between present acids and alcohols to form esters. This finding is supported by a study conducted on bitter oranges, which highlights that the presence of carbon dioxide in modified atmospheres, compared to untreated ones, improves quality and shelf life of food products. Finally, passive MAP atmosphere decreases humidity conditions inside the package, which contributes to ester formation through esterification reactions [79].

3.4 Sensory Evaluation

The sensory analysis results are presented in Figure 6, highlighting significant differ- ences in three attributes: colour intensity, orange aroma, and honey aroma. The active MAP sample exhibited significantly higher colour intensity ($p < 0.001$) compared to the passive MAP sample. Additionally, the orange and honey aromas were rated higher in the active MAP samples, indicating noteworthy differences ($p < 0.05$), as noted by the tasting panel. The two samples were perceived as similar in other attributes considered by the panel. Overall, attributes such as taste acceptance, sweetness, acidity, gumminess, masticability, and overall acceptance received higher scores for the active MAP sample, although these differences were not statistically significant. The panel reported lower ratings for any unpleasant tastes or odours, indicating that none were specifically identified. Based on responses from the affective test following the descriptive test, only 3% of participants reported consuming dried fruit almost daily, 14% consumed it several times a month, 50% consumed occasionally, and 33% did not consume dried fruit at all.

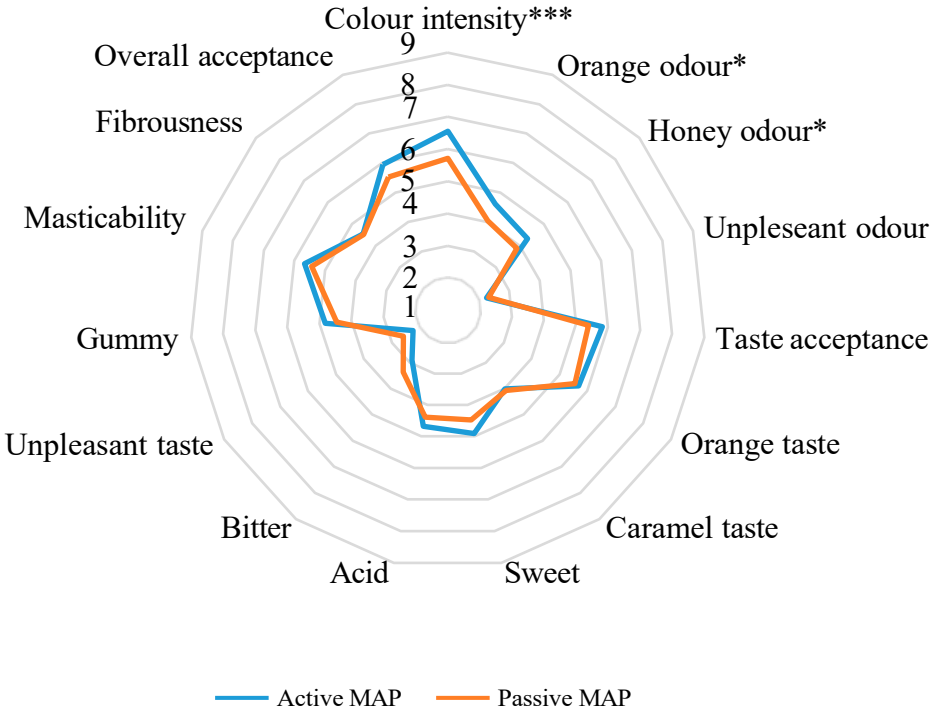


Figure 6. Sensory evaluation of dried blood orange slices at 100 days of storage in active and passive MAP. Symbols: * $p < 0.05$, *** $p < 0.001$.

Furthermore, 56% of participants expressed their willingness to purchase the active MAP sample if it were available in supermarkets (Figure 7).

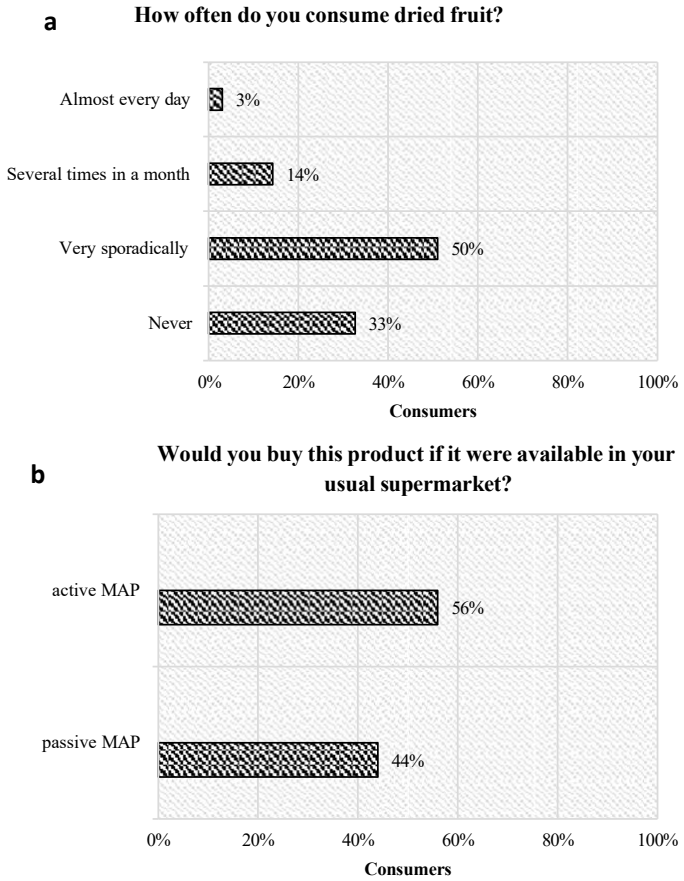


Figure 7. (**a**) percentage of consumers who have selected different dehydrated fruit consumption habits, (**b**) intention to purchase dried blood orange slices depending on the conservation treatment (active e passive MAP).

4. Conclusions

The appropriate choice of time and temperature for the drying process is essential to ensure microbiological stability and retain the characteristic taste of dried blood orange slices. Data confirms that hot air-drying at 70 ◦C for 12 h maintained shrinkage without altering the longitudinal-diameter slices. However, significant thickness loss was observed, especially in samples treated with passive MAP, even during storage. Additionally, an increase in hardness was noted due to water removal, enhancing masticability. Active MAP maintained high hardness values up to D100 and preserved the typical colour of blood oranges, as confirmed by sensory analysis. Sensory analysis showed that blood oranges dried and stored in active and passive MAP mainly differed in colour intensity and characteristic flavour. The use of HS-SPME chromatography facilitated the identification of chemical compounds responsible for aroma, flavour, and other sensory attributes. Costa et al. [80] also revealed the complex relationship between temperature, storage conditions, and VOCs production. This study highlighted significant enhancement in the food product's value. Enhancing organoleptic characteristics and nutritional integrity in blood orange slices can improve consumer

satisfaction and health outcomes by potentially enriching the nutraceutical profile through modified atmosphere processing.

In conclusion, combining the drying technique with MAP storage is a valid method for maintaining the initial quality of dried slices, making it an excellent approach to produce a healthy, tasty, and visually attractive snack.

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CHAPTER 4

Tray-drying is a new way to valorise white-fleshed peach fruit

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Abstract

Pescabivona is a highly appreciated fruit by consumers for its sweet flavour and juicy flesh; however, it has a short shelf life and is susceptible to postharvest damage, such as mechanical injury, loss of texture and alteration of organoleptic properties. Therefore, it's necessary to develop new methods of processing and conservation for this fruit. The aim of this study was to analyse the effects of tray-drying in white peach slices and cubes at 70 ℃ for 12 hours to extend their shelf-life and increase its commercial availability over a long period and to obtain a new food product. The physicochemical and sensory properties of dried fruits were assessed during 30 days of storage in polyamide/polyethene (PA/PE) bags containing two gas mixtures (treatments): MAP-N_2 (100% N_2) and MAP-P (78% N_2, 21% O_2 and 0.04% CO_2), at room temperature (20 ± 1 ℃). Both MAP treatments kept the fruit firmness, with MAP-P slightly more effective. Slicing produced fruit with a good appearance and firmness, while cubing produced sweet fruit with a caramel flavour and a chewier firmness. In addition, packing with MAP-N_2 reduced the phenomenon of fruit browning. Overall, this study provides significant information on the drying process (time-temperature treatments) and packaging techniques (MAP) of white-fleshed peach to obtain a novel food product.

Keywords: pescabivona; post-harvest; fruit quality; dried fruit; modified atmosphere packaging; sensory analysis

1. Introduction

The peach (Prunus persica (L.) Batsch) is the second most cultivated fruit in the world. Within the European Union (EU), Italy is the leader in peach production. In northern Italy, mainly in Emilia- Romagna, nectarines represent 50% of new orchards, while in southern Italy peaches are preferred. At the end of the 1970's the peach industry in Sicily was still based on clingstone white fleshed local types, called "montagnole" (fruits of the mountains), nectarine-type, white-fleshed, named "Sbergie" and flat-type named "Tabacchiere" [1]. In the '90s, peach and nectarine cultivars were introduced from the USA, consequently, a renewal of Sicilian orchards started [2]. In any case, white-fleshed peaches remain an important part of Sicilian production. Pescabivona, also known as "Pesca di Bivona," is an indigenous cultivar grown only in its original area, the Bivona area, located in the Middle-West Sicily [3]. Pescabivona includes four native varieties: Murtiddara (also known as Primizia Bianca), Bianca, Agostina and Settembrina. These varieties ripen at different times, from late June (Murtiddara) to late September (Settembrina) [4]. The Pescabivona cultivation area falls within the Sicani Mountains and includes the territories of Bivona, Alessandria della Rocca, Santo Stefano Quisquina and San Biagio Platani, in the province of Agrigento and Palazzo Adriano, in the province of Palermo [5]. Pescabivona obtained protected geographical indication (PGI) certification in 2014, and the product specification indicates peach fruits with a white non-melting flesh, characterized by a spheroidal shape and a creamy-yellow background skin colour with an extent of red over colouration (50%) for Murtiddara, followed by Agostina and Settembrina (40%) [6]. In addition, Settembrina has a peculiar red stripe along the suture line, due to a mutation involving the epidermal layer [7]. Its flavour is sweet and aromatic. Its odour stands out from the peaches produced in the rest of Sicily and Italy, due to its inebriating and persistent nature [5]. Furthermore, Pescabivona is highly valued for its organoleptic properties and high firmness. In fact, the fruit has been classified as non-melt, with sugar and phenolic content, finding considerable interest from the local market and large-scale organized distribution [8]. On the other hand, white fleshed peaches soften rapidly after commercial ripening, are very susceptible to chilling injuries and can be easily damaged during storage [9].

Peach is a climacteric fruit and, after harvest, numerous physiological and metabolic processes (respiration, transpiration, flesh softening, etc.) continue in it, drastically increasing ethylene production at a rate closely related to the storage temperature [10]. Being a white-fleshed peach, the main critical problems found on Pescabivona are

related to strong seasonality, reduced fruit shelf-life, enzymatic browning, susceptibility to cold and handling damage [5,11]. Ripening time influences dramatically fruit quality, and, for this reason, it is important to individuate the optimal period [12]. Recently, several postharvest treatments have been studied to reduce and limit cold damage, including the application of salicylic acid, jasmonic acid, 1-methylcyclopropene, controlled atmosphere, ozone and radiation [13–15]. In addition, techniques such as refrigeration are nonresolving and limit shelf- life due to the possible development of pathogens and damage caused by low temperatures, as well as the considerable environmental impact they cause, in some cases reducing consumer's purchasing intention [16–19]. Today, consumers also prefer to buy fruit close to physiological ripeness, when there is maximum expression of the sensory and organoleptic profile [20]. This creates technical problems related to reduced shelf-life due to senescence, increased waste due to microbial spoilage and altered visual appearance of fruit. Post-harvest waste of fruit and vegetables is alarming and represents a major challenge in the food supply chain due to its negative social, economic and environmental impact [21]. It is estimated that 20–60% of total production and many fruit and vegetables are lost and wasted along the food chain [22,23]. Among the possible techniques to prevent post-harvest losses of white-fleshed peaches, also given their seasonality and short shelf life, is tray-drying [24]. In addition, recent studies have reported a significant increase in the consumption of dried fruit, due to the many benefits of regular consumption of these products [25–27]. In recent years, there has been an increasing demand for these products in their many formats (mixes, bars, single portions, etc.), which are well suited to the different needs of consumers [28].

Drying is an old method based on the partial or total reduction of water content in the form of steam to a final concentration (10–14% RH) that ensures microbial stability and improves shelf life [29]. The drying treatment also leads to the breakdown of cell structures and the loss of membrane semi- permeability, resulting in a change in rheological properties. In fact, the outer layers of the dried matrix become rigid and protein aggregation takes place [30]. However, structural changes depend on the drying method and the residual moisture in the plant tissue. Drying processes influence the colour, flavour, texture and functional properties of fruit [24,31]. The oldest drying method is exposure to the sun, which allows a product with good quality characteristics but prone to environmental contamination [32], as well as being an extremely slow process. Using machines, however, heat invests the whole fruit or pieces/slices by

conduction, convection and radiation [33,34]. Hot-air drying is an alternative method that yields a qualitatively superior product using low temperatures and short processing times [35]. The basis of the drying process, along with the healthy element, the taste experience and the convenience of consumption, environmentally friendly preservation systems must be implemented that promote food shelf life and safety. Dried products must also be stored properly to maintain their quality and nutritional characteristics for a prolonged period. Among the different storage systems, MAP, maintains the qualitative characteristics of perishable products for a prolonged shelf life [36–38]. The principle behind MAP is to replace the atmosphere surrounding a product before sealing by mixing carbon dioxide, oxygen and nitrogen (the most used gases) at different concentrations. The modified atmosphere is useful because it reduces the oxidation of food products and, consequently, the production of compounds that cause the loss of colour, aroma and taste [39]. The use of inert gases, such as nitrogen, not only prevents package collapse but also helps prevent microbial growth, which is another factor contributing to product degradation [40]. Several studies [24,41] have shown that MAP treatments with 100% N_2 limit significant nonenzymatic browning, while maintaining weight and firmness characteristics over a long storage period.

This work aims to investigate the effects of hot-air drying technique and MAP during 30 days of storage on the quality of slices and cubes of white-fleshed peaches. In this way, it is intended to enhance a fruit that is highly susceptible to post-harvest damage, prolonging its shelf life and allowing a wider commercial distribution. At the same time, it is also aimed at using fruit that does not conform to commercial standards to reduce waste.

2. Materials and methods

2.1 Plant material

Peach fruits (Prunus persica Batsch) cv. Pescabivona var. Settembrina, ready to consumption with typical taste (sweetness ≥ 10 °Brix), colour (overcolour < 50%) and texture (≥34.3 N) were used [42]. The fruits were harvested at the "Soletta" Farm, located in the city of Castronovo di Sicilia (PA) (37°40'27.98"N; 13°39'12.55"E, 371m a.s.l.). After harvest, the fruits were stored at 5±1°C and after 24 h were subjected to analysis. Pomological characterization was carried out on a sample of 20 fresh fruits.

2.2 Experimental design

Due to the lack of literature data, several preliminary tests on drying peach fruit were conducted to find the right time/temperature combination and whether to dry the fruit with or without the peel. For this reason, the initial preliminary test involved unpeeled fruit, dried for 12 h at 70 °C. However, although the time/temperature combination proved to be effective in terms of maintaining organoleptic and sensory characteristics, it was found that the presence of the epicarp, after drying, causes excessive wrinkling, resulting in an uneven and aesthetically unappealing product (data not shown). Hence, the definitive experimental protocol involved the use of fruit peeled and dried for 12 h at 70°C. The whole fruits were washed and sanitized with distilled water (20±5 °C) and 200 µL-L NaClO (5–6.5% NaClO solution, CHEMLAB) for 10 min. Then, the fruits were peeled, de-stoned and cut with a curved knife to a thickness of 5±0.5 mm as follows:

- Slices (S)
- Cubes (C)

To prevent browning, the fruit were dipped in natural anti-browning agents [43], for 2 min. The antioxidant solution was obtained by dissolving citric acid (99.5–101% C6H8O7.1H2O, CHEMLAB) and ascorbic acid (99+% C6H8O6, CHEMLAB) in distilled water at concentrations of 0.5 g/0.5 L. drying process was conducted by means of a convective hot-air dryer operating at constant temperature. The dryer consists of a stainless-steel chamber (86 cm x 86 cm x 76 cm) provided with an electric heater to heat the air and a centrifugal fan to feed the airflow and recirculate it. The samples were put on the grids of mesh of size 0.01 m x 0.01 m in the dryer and, after that, they are dried at 70°C for 12 h at a centrifugal fixed air velocity of 2.3 m/s until the relative humidity was constant(14–16%R.H.), according to Diaz [44] and Kesbi [45]. Dried fruits were stored in MAP and specifically, two treatments were applied to understand the effect of modified atmosphere packaging:

- MAP-N2 (100% N_2);
- MAP-P (78% N_2, 21% O_2 and 0.04% CO_2).

The samples were packed in polyamide/polyethylene bags composed of 80% polyamide and 20% polyethylene, with a thickness of 90 µm and a volume of 500 cm^3, with an oxygen permeability of 47.6 cm^2/ (m^2 day atm) and a water vapor transmission rate of 3.9 / ((m^2 day atm) [46]. A modified atmosphere was obtained inside the sealed bags, using a digitally controlled packaging machine (VM 16 Orved S.p.A, Musile di Piave, Venezia, Italy). Each bag contained 100 g of dried fruit divided according to the type of cut and treatment:

- MAP-packed cubes (MAP-N_2 C);
- MAP-packed slices (MAP-N_2 S);
- MAP-P packed cubes (MAP-P C);
- MAP-P packed slices (MAP-P S).

Samples were stored at room temperature (20±1°C) for 30 days and analyses were performed every 10 days (D10; D20; D30).

2.3 Physico-chemical analysis

Fresh weight (FW-g) was measured with a precision electronic scale (Gibertini EU-C 2002 RS, Novate Milanese, Italy), while fruit longitudinal diameter (LD-mm) and fruit transverse diameter (TD-mm) were measured with a digital calliper (Turoni TR53307, Forli, Italy). The colour of the fruit was determined based on the CIE L*a*b* colour system [brightness (L*); red/green (a*); yellow/blue (b*)] by means of digital colourimeter (CR-400 Chroma Meter, Minolta, Japan). Colourimeter calibration was performed against a white plate (illuminants C: Y ¼ 89.53, x ¼ 0.3247, y ¼ 0.3198) before each measurement. For each sample, the parameters L*, a* and b* were measured and the averages of all measurements were determined for each package. Chroma (C*) values, which indicate the quantitative attribute of colour intensity, have been calculated using Equations (1), to characterize fresh sample: [47]:

$$C^* = \sqrt{a^2 + b^2} \quad (1)$$

Total colour difference (ΔE), between the colour of fresh fruit and that of the fruit after drying for both types of cuts were measured at D0, following the Equation (2) [47]:

$$\Delta E = \sqrt{(L^* - L_0^*)^2 + (a^* - a_0^*)^2 + (b^* - b_0^*)^2} \quad (2)$$

where L*0, a*0 and b*0 represent lightness (L*), redness (a*) and yellowness (b*) of the samples after drying, respectively. In addition, the browning index (BI) at each observation during the 30 days of storage was measured with the following Equation (3) [48]:

$$BI = \left(\frac{x - 1{,}75L^*}{0{,}17}\right) \times 100 \quad (3)$$

where $x = \left(\frac{a^* + 1{,}75L^*}{5{,}645L^* + a^* - 0{,}3012b^*}\right)$

The firmness (FF) of the slices and cubes was measured using a durometer (Durofel Agrosta 100 Field, Serqueux, France) and converted to Newtons using Equation (4) [49]:

$$N = 9{,}8\, e^{\left(\frac{Durofel - 59{,}32}{14{,}89}\right)} \quad (4)$$

Total soluble solids content (TSSC) was measured by means of digital refractometer (Atago, Tokyo, Japan) and the values were expressed as °Brix; while titratable acidity (TA g/L−1 malic acid) was measured with a pH meter-titrator (Titromatic 1S, Crison, Barcelona, Spain) and expressed as grams of malic acid per litre of fruit juice (g L−1 malic acid) using 5 mL of juice diluted in 45 mL of distilled water. The dried fruit was checked for drying efficiency (DR%), which was calculated using Equation (5) [50]:

$$\%DR = \left(\frac{c-a}{b-a}\right) \times 100 \quad (5)$$

where:

a = weight of the empty tray

b = weight of the tray with the product before drying

c = weight of the tray with the product after drying

2.4 Sensory evaluation

Sensory evaluation was carried out by a team of 30 judges with good training and knowledge in this type of food evaluation [51], following the guidelines of UNI 10957:2003 [52]. During the preliminary test, 15 qualitative descriptors were chosen for sensory profiling, generated on the basis of citation frequency (≥60%) and listed below: visual appearance (VA); firmness (FR); peach odour (PO); honey odour (HO); off-odour (OO); acidity (AC); sweetness (SW); bitterness (BT); juiciness (J); crispness (CR); rubberness (RU); peach flavour (PF); caramel flavour (CF); off-flavour (OF); degree of browning (B). The evaluation was conducted from 10 to 12.00 in a room with white lights. Each judge received, in random order, a sample of three pieces of fruit, by cut and treatment. Between each sample, water was provided to rinse the mouth. The judges rated the intensity of each descriptor on a hedonic scale by assigning a score between 1 and 9, where: 1 - no sensation, 2 - scarcely discernible, 3 - very low, 4 - low, 5 - slight, 6 - moderate, 7 - intense, 8 – very intense and 9 - extremely intense [53].

2.5 Statistical analysis

Using the XLSTAT software (Addinsoft, Paris, France), repeated measures ANOVAs and Tukey's honestly significant difference (HSD) tests ($p<0.05$) were performed for all parameters studied to assess significant differences between the treatments and days of storage.

3. Results and discussion

3.1 Fresh fruit

A preliminary physicochemical characterization was carried out on a sample of 20 fresh fruit before drying (Table 1).

Table 1. Qualitative characteristics of fresh Pescabivona var. Settembrina fruit. TSSC values (total soluble solids content, Brix); TA (titratable acidity, g/L^{-1} malic acid); FF (firmness; N); TSSC/TA ratio. Values are represented as mean ± SD.

	TSSC (°Brix)	TA (g malic a./L)	FF (N)	C* (Chroma)	TSSC/TA
Pescabivona var. Settembrina	12.93±0.40	0.18±0.07	0.20±0.01	27.60±0.53	71.83

From the obtained data, it can be said that peach fruits are sweet and low in acidity, with an intense colouration tending toward yellow/orange, according to Montevecchi et al. [54]. Crisosto et al. [55] demonstrated a higher acceptance of grapes with a high TSSC/TA (greater than 25 .0) among U.S. consumers. From the obtained data, the TSSC/TA ratio reached 73.83 confirming a degree of ripeness close to consumption and therefore closer to consumer preferences. These data have been considered important to determine the initial characteristics of the fruit and the differences between fresh and dried products.

3.2 Longitudinal and transverse diameter and dry residue

During the drying process, longitudinal (LD) and transverse (TD) diameter values decreased in both types of cuts (slices and cubes). The porous and hygroscopic nature of fruits and vegetables makes them highly shrinkable during drying [56].

Table 2. Temporal trend of longitudinal (LD) and transverse (TD) (mm) diameters of slices (a) and cubes (b) of Pescabivona var. Settembrina. Diameter data are expressed as mean values ± SD. Values in the same column followed by a different letter are significantly different ($p < 0.05$); ns = non-significant; asterisks indicate statistically significant differences between MAP treatments at the same storage day with * $p < 0.05$; no asterisks indicate the non-significant difference between treatment means.

(a)	**Slice (S)**	
	LD	**TD**
Fresh	57.57±1.71	25.20±1.81
Post drying	49.80±5.47^{ns}	19.28±3.23^{ab}
D10 MAP-P	48.46±4.61	20.92±1.40^{a}
D20 MAP-P	49.60±6.02	19.75±2.43^{a}
D30 MAP-P	46.00±2.49*	17.30±2.94^{b}
Post drying	49.80±5.47^{ab}	19.28±3.23^{a}
D10 MAP-N_2	50.87±5.89^{a}	19.75±2.01^{a}
D20 MAP-N_2	45.45±7.49^{bc}	18.60±2.35^{a}
D30 MAP-N_2	41.80±4.84^{c,*}	16.15±3.86^{b}
(b)	**Cubes (C)**	

	LD	TD
Fresh	36.07±1.36	24.56±1.13
Post drying	27.31±1.83^{a}	18.53±1.43^{ab}
D10 MAP-P	25.33±4.06^{ab}	19.95±3.74^{a}
D20 MAP-P	23.60±2.60^{b}	16.90±1.97^{bc}
D30 MAP-P	22.65±6.29^{bc}	15.45±3.09^{c}
Post drying	27.31±1.83^{a}	18.53±1.43^{a}
D10 MAP-N_2	24.81±4.81^{ab}	19.59±3.29^{a}
D20 MAP-N_2	21.75±4.95^{b}	15.20±2.38^{b}
D30 MAP-N_2	23.40±4.62^{bc}	15.15±1.73^{b}

As shown in Table 2, before drying, the peach slices had a length dimension (LD) of 57.57 mm and a width dimension (TD) of 25.20 mm. After drying, these values decreased to 49.80 mm and 19.28 mm, respectively, representing a significant reduction in LD of 13.49% ($p < 0.01$) and TD of 23.49% ($p < 0.001$). Similarly, in cubes the value also decreased, with a reduction of 24.29 % ($p < 0.001$) in LD and 24.55 % ($p < 0.001$) in TD. Although no significant variation was observed for TD between the two cuts, LD was reduced (10.8% more) in the cubes. This was due to the shape and physical structure of the samples. The slices probably retained their LD due to the even distribution of heat stress they were subjected to. By comparing these data with %DR, a loss of water content of 84.57% and 83.64% was observed for S and C, respectively. For the same thickness, a higher dry efficiency was observed for slices than for cubes, probably due to the greater surface area exposed during drying.

During the 30-day storage period, the LD and TD diameters further decreased. Storage of the samples in MAP showed a significant decrease in LD in both cuts and only for MAP-P S was not significant ($p \geq 0.05$). In addition, a significant difference was observed between MAP-P and MAP- N_2 treatments at the end of the storage period (D30). Specifically, in the slice, the LD decreased by 3.80 mm in samples stored in MAP-P and by 8 mm in those stored in MAP-N_2, showing that, for the type of cut, storage in MAP-P is more suitable ($p = 0.01$). In the cubes, no significant changes were observed between the two MAPs treatments to D30. In fact, the LD decreased by 4.66 mm in samples stored in MAP-P and 3.91 mm in those stored in MAP-N_2. Similarly, changes were observed for TD during the 30-day storage period. In the slices, a significant decrease of 1.98 mm was observed in the samples stored in MAP-P and 3.13 mm in the samples stored in MAP-N_2. Whereas, in the cubes, the reduction was 3.08 mm in MAP-P and 3.38 mm in MAP-N_2. There was no significant effect of MAP treatment on transverse diameter reduction in any of the cuts throughout the storage

period ($p \geq 0.05$). For slices, treatment with MAP-P seems to have been more appropriate, as it caused a smaller reduction in LD than treatment with MAP-N_2. In cubes, on the other hand, both MAP-P and MAP-N_2 appear to be equally effective because no significant changes were found in the reduction of transverse diameter.

The results indicate that the drying process influences LD and TD reduction while storage in modified atmosphere had no significant influence.

3.3 Firmness

One of the main changes affecting the structure of plant tissues during drying is the change in firmness [57]. The removal of water content from the fruit during the drying process results in a loss of volume and weight, as well as a change in mechanical properties, such as hardness and crispness [58]. Fruit firmness is one of the most important quality properties, as it influences the acceptability of the fruit to consumers [59]. Manrique et al. and Mafra et al. [60,61] report that changes in fruit texture are largely determined by changes in the structure of the fruit cell wall and polysaccharides in the middle lamella. After the drying process, there was the same significant increase of 0.1 N firmness ($p < 0.001$) in both cuts (0.30 N) compared to the fresh fruit (0.20 N). This could be due to drying, through the removal of water from the fruit tissues, increasing the concentration of solutes within the cells, causing them to become more compact and less susceptible to deformation [62]. However, it is important to consider that firmness values may also depend on other factors, such as the type of fruit, degree of ripeness, temperature and drying time used during the process [63]. Considering ripe peach for consumption, the increase of 0.1 N can be perceived as a positive result for handling and transport because it indicates a greater resistance to pressure and a greater ability of the fruit to maintain its shape and integrity [64]. However, it is also important to consider consumer preferences to determine if this result is also desirable for the market. For example, a study by Wong et al [65] showed that Korean consumers prefer apple-based dried fruit produced in South Korea with a crunchy firmness, while they prefer pear-based dried fruit produced in the United States with a soft, chewy firmness. Therefore, dried fruit companies should take these preferences into account when developing new products for different markets.

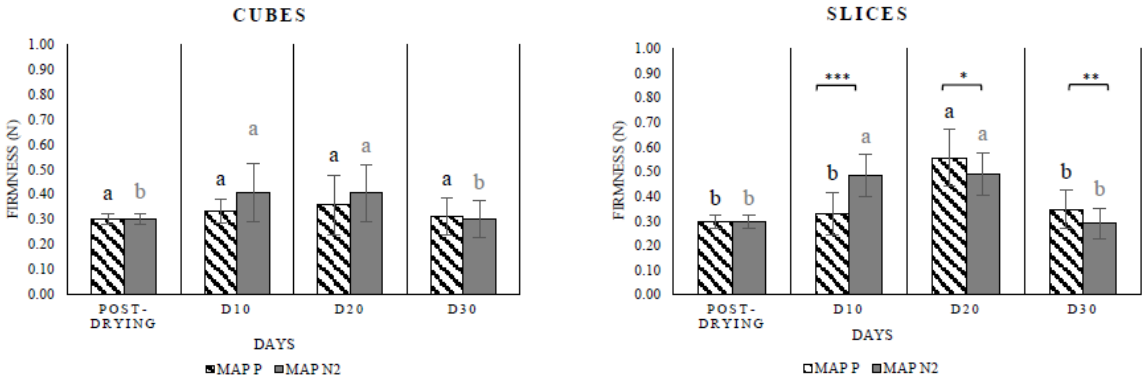

Figure 1. Firmness evolution of fruits Pescabivona var. Settembrina dried and stored for 30 days at room temperature (20±1 °C). Values in the same set of histograms followed by a different letter are significantly different ($p < 0.05$); asterisks indicate statistically significant differences between MAP treatments with * $p < 0.05$, ** $p < 0.01$, *** $p < 0.001$; no asterisks indicate a non-significant difference between the means of the treatments.

During the 30-day storage period, as can be seen from the results (Figure 1), the firmness (FF) of peach fruits varied significantly for MAP-N2 C, MAP-P S and MAP-N_2 S treatments, although the S values were slightly higher than C values. However, no significant loss of firmness was found for S and C at the end of the 30-day storage period. Moreover, when comparing the two MAP treatments during the observations (D10, D20 and D30), significance was only found for the cut. The data show that although at D10 the MAP-N_2 packing positively influenced the maintenance of fruit firmness, the same behaviour was observed with the MAP-P treatment from D20 and D30. For this reason, we can state that storage in MAP-N_2 is not cost-effective for the Pescabivona supply chain.

The obtained data through the evaluation of firmness evolution can be compared with what emerged from the sensory analysis. As can be seen from Figure 4, firmness, as perceived through the FR descriptor, is evaluated positively for the entire storage period. In particular, the MAP-P S treatment maintains the values up to D30.

Similar results were reported by Tinebra et al. on behaviour of dried loquats [24]. Wu et al. [66] studied the firmness maintenance in dried fruit seeds, showing that N_2 plays a key role in maintaining organoleptic qualities. A study on the importance of N_2 was conducted on pomegranate arils [36], according to which, MAP was used to maintain the quality of the processed fruit and showed optimal results in terms of weight retention, which allowed the pulp structure to remain intact. A slight decrease in fruit firmness was observed during the storage period, attributable to the loss of turgor [35] and compensated by hardening due to drying. The results obtained are satisfactory when compared to studies conducted on peaches, pears, and apricot [24], which lose up to 99% of their firmness values following drying.

3.4 Colour

The drying of fruit results not only in changes in firmness but also in the characteristic flesh colour [65]. This occurs due to the oxidation of proteins and lipids and non-enzymatic reactions [68].

Table 3. Post-drying values of CIE L*a*b* coordinates that measure chroma and colour differences in both types of cuts (lightness (L*); redness (a*); yellow (b*); chroma (C*); colour differences (ΔE); dry residue (%DR). The data corresponds to the means ± SD.

	L*	a*	b*	C*	ΔE	%DR
Slices	72.38±4.98	3.51±2.17	38.90±3.66	39.10±3.80	16.12±4.47	15.43
Cubes	73.25±4.98	4.20±2.17	40.22±3.66	40.58±3.80	17.91±4.58	16.35

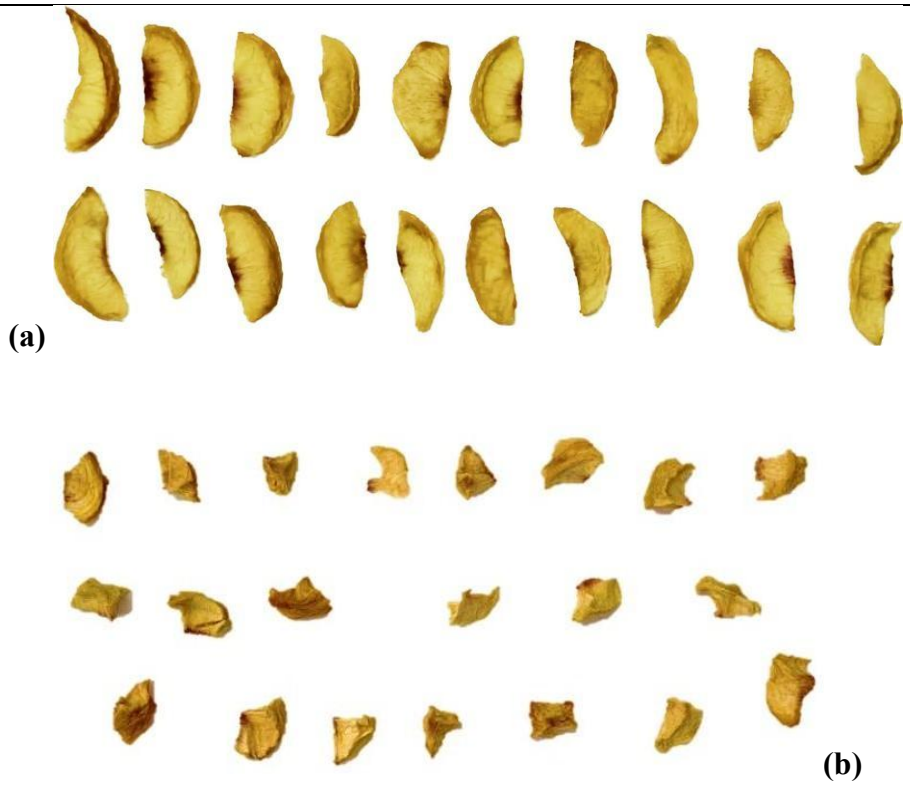

Figure 2. A representative sample of slices (a) and cubes (b) of Pescabivona var. Settembrina fruits after drying process.

Total colour differences (ΔE) after drying process showed similar browning between slices (S) and cubes (C) compared to the fresh unprocessed form (Table 3). These values are also confirmed by comparing L*, a* and b* values with Chroma. Therefore, temperatures and processing times did not cause significant variations between the two cuts (Figure 2). One possible reason could be that both cuts had similar amounts of initial moisture before drying process. In fact, when comparing the initial weights before drying, the two lots showed very similar values. This is also confirmed by the results of %DR (Table 3), which may have resulted to a similar drying process for both cuts.

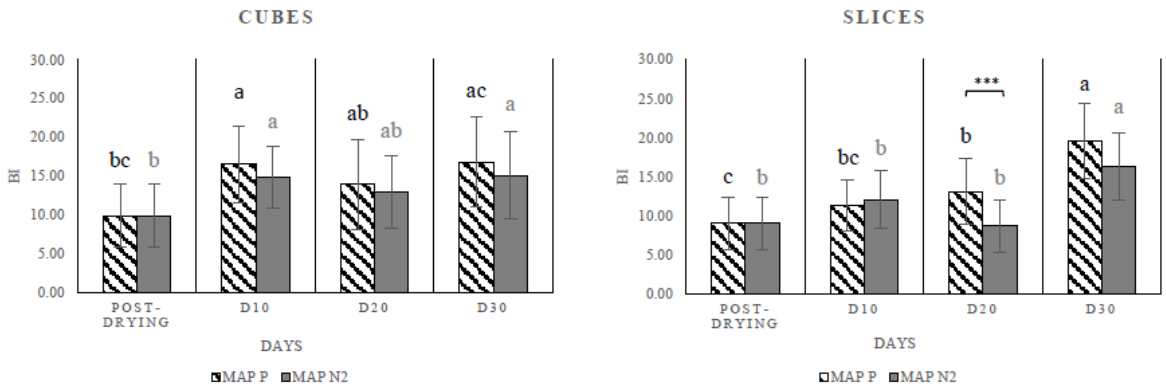

Figure 3. Browning Index (BI) values of dried Pescabivona samples were analysed during the experiment. Values in the same set of histograms followed by a different letter are significantly different ($p < 0.05$); asterisks indicate statistically significant differences between MAP treatments with *** $p < 0.001$; no asterisks indicate a non-significant difference between means of treatments.

However, different behaviour was observed during the 30-day storage in MAP (Figure 3). The measured data shows, in both types of cuts, a significant increase ($p < 0.05$) in the browning index (BI) although, after 30 days of storage, the cut that maintained the lowest BI values was the cubed cut. In S, there was an increase in BI of 10.56 in MAP-P and 7.26 in MAP-N_2 from the day after drying to D30. In cube cut, on the other hand, BI increased by 6.91 in MAP-P and 5.21 in MAP-N_2. In the presence of nitrogen, BI appeared to show an almost constant trend up to the last day of storage (D30) and, in all cases, maintained values closer to the first day of storage in MAP-P. Although variations in BI were observed in the two MAP treatments during the 30-days period, statistical analysis showed significance only for the slice at D20. As can be seen from Figure 3, the BI values during storage do not differ much from those after drying. This shows that the pulp did not undergo significant changes in colouration. This phenomenon could be due to enzymatic processes mainly involving phenolic compounds [38]. Furthermore, there seems to be a positive correlation with both the time-temperature combination used and the immersion in antioxidant solutions carried out in the pre-drying phase. In addition, the presence of N_2, used to reduce the oxygen level within the package, inhibits the growth of aerobic microorganisms and prevents the oxidation of the nutrients present, thus preserving the quality of the fruit [69]. According to Wu et al. [66], in addition to maintaining firmness, N_2 is also responsible for less browning during storage. The results of this study showed that fresh peanut kernels treated with MAP N_2 maintained lower browning values than the control group during storage.

3.5 Sensory analysis

The sensory analysis of the dried products was conducted throughout the storage period, as shown in Figure 4. From the analysis of the results, the fruit was most appreciated for peach flavour (PF), sweetness (SW), juiciness (J) and crunchiness (CR). After 10 days of storage,

the slices (S) stored in MAP-P were most appreciated, receiving scores of 7 in the positive descriptors of visual appearance (VA) and firmness (FR). In contrast, the cubes (C) stored in MAP-N_2 were found to be the sweetest but with a pronounced caramel flavour (CF) and more rubberiness (RU) than the other treatments. After 20 days of storage, fruit from all treatments was highly appreciated for sweetness (SW), rubberiness (RU) and visual appearance (VA). Finally, after 30 days of storage the peach cubes (C), stored in both MAP packaged, scored high for the negative descriptor browning (B), while the S fruit continued to have a very low browning value (B) and scored high for the positive descriptors sweetness (SW) and visual appearance (VA).

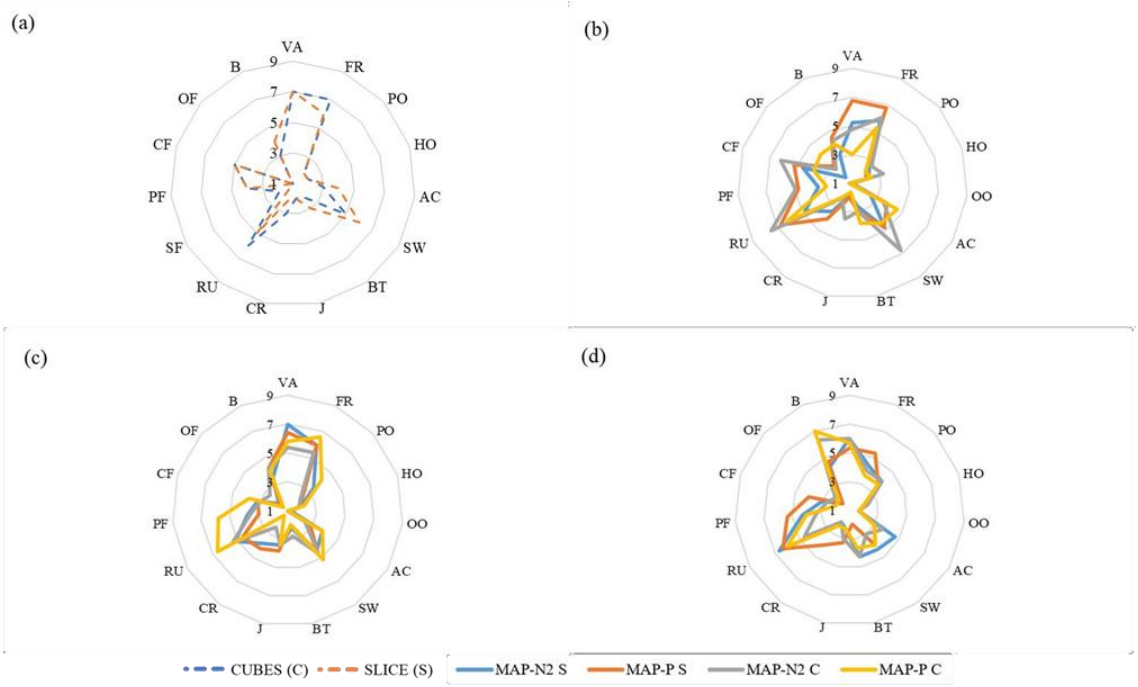

Figure 4. Results of sensory analysis conducted on Pescabivona var. Settembrina fruits dried at 0 (a), 10 (b), 20 (c), and 30 (d) storage days. Legend: visual appearance (VA); firmness (FR); peach odour (PO); honey odour (HO); off-odour (OO); acidity (AC); sweetness (SW); bitterness (BT); juiciness (J); crispness (CR); rubberness (RU); peach flavour (PF); caramel flavour (CF); off-flavour (OF); degree of browning (B).

4. Conclusions

In this study, the effect of MAP drying and storage on the physicochemical and sensory quality of Pescabivona var. Settembrina fruit was evaluated, with interesting results. The study concluded that it is possible to apply the drying process to white-fleshed peaches by identifying an appropriate time- temperature combination, which helps to maintain the quality characteristics of the fruit. Samples subjected to MAP maintained an attractive colour after drying and a good firmness and especially MAP-N_2 C treatments limited non-enzymatic browning during the storage. Sensory analysis showed that the slicing technique was the best choice, producing fruits with a good visual appearance and firmness, while the cube technique produced sweet fruits with a caramel flavour and more chewy firmness.

Moreover, the tray-drying method could help recover Pescabivona var. settembrina fruit waste from the supply chain and create a new value-added- product. Finally, there are no studies in the literature concerning tray drying of this fruit species. Therefore, this study is preliminary to future investigations concerning drying kinetics to more accurately assess the time interval in which a constant moisture value is obtained to preserve the organoleptic quality of the product in question. Furthermore, this approach could bring to light differences, in terms of time, between cubes and slices.

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CHAPTER 5

Effects of Tray-Drying and modified atmosphere on quality of mango fruit

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Abstract

Mango cultivation in Sicily has emerged in recent decades due to the growing demand for tropical fruit from the European market and accelerated by climate change in the region. The Sicilian mango, unlike its imported tropical counterpart, has the advantage of being harvested at a degree of ripeness close to that of consumption (tree-ripe), which results in increased colour, aroma, and sweetness. However, growing tropical species in Mediterranean environments, in the last decade can present challenges due to climatic variations and new biotic threats, resulting in an increased likelihood of obtaining fruit that does not meet commercial standards or is damaged. Dehydration is one of the food processing techniques that could mitigate the economic and environmental impact of waste in the Sicilian mango supply chain. The aim of this study was to assess the impact of drying on mango fruits (cv. Tommy Atkins) harvested at two different pre-harvest ripening stages, with a focus on their physicochemical, microbiological, nutraceutical, and sensory properties. Additionally, the study aimed to evaluate the effectiveness of modified atmosphere packaging (MAP) in preserving the quality of the dehydrated products during storage. The fruits, at two different stages of ripening (Green and Mature), were dehydrated in a convective tray drying at 70°C for 12 hours. Then, they were stored in polyamide/polyethylene (PA/PE) bags with two different MAP treatments (100% N_2 and passive) at room temperature for 30 days. Physicochemical analysis showed significant changes in terms of C* (Chroma) only in Green MAP N_2 samples, whereas h* (hue angle) was maintained only in Mature MAP P. Firmness (N) changed significantly, indicating that Green MAP N_2 samples maintained a higher consistency. Green mango cubes showed the least shrinkage in terms of longitudinal (19%) and transverse (16%) diameter during the transition from fresh to dehydrated, without significant changes during storage. Mature

dehydrated fruits maintained a high content of polyphenols, minerals, and vitamins (A, B1, B3, C) throughout the storage period and were microbiologically safe. Consumers evaluated Mature MAP N_2 dehydrated samples positively, showing a high propensity to purchase (40%). In conclusion, convective tray drying proved to be an effective method to utilize mango fruits otherwise excluded from the market, positioning them as processed products, and enhancing the value of supply chain waste.

Keywords: *Mangifera indica*; post-harvest quality; dried fruit; MAP; sensory analysis; waste recycling

1. Introduction

In recent decades, the cultivation of many exotic species such as avocado, papaya, litchi, macadamia, annona and mango has increased significantly in Sicily [1,2], driven by the growing demand for tropical fruit on the European market and facilitated by climate change in the region [3–5]. Mango (Mangifera indica L.) is a fruit of tropical origin that is highly valued for its nutritional richness in vitamins, proteins, sugars, fats and minerals, including calcium, potassium and magnesium [6]. Its quality and mineral content vary significantly between varieties and ripening stages [7]. Different from its imported tropical counterparts, Sicilian mangoes have the advantage of being harvested at a degree of ripeness closely conformed to optimal consumption, resulting in improved colour, aroma, and sweetness directly from the tree. However, growing tropical species in Mediterranean environments presents challenges related to the climatic changing, and the development of new biotic threats, increasing the likelihood of obtaining fruit that may not meet commercial standards or suffer damage [8–10]. Therefore, there is a need to limit the loss of commercially unsuitable fruit by doing their transformation in by-products. One of the possible strategies is to apply the tray drying to obtain a new product. Moreover, this process extends the storability through the reduction of water activity [11]. Water activity is crucial for the preservation of produce; when it is below 0.65, the growth of bacteria, yeasts and moulds is suppressed [12]. To address these challenges, drying has emerged as a food processing technique that could mitigate economic and environmental impacts in the Sicilian mango supply chain. Many studies have been conducted on mango processing techniques, particularly drying [13–16]. However, there is a gap in research on the effects of drying on Sicilian mango cubes at different stages of ripening. Drying has been identified as an established solution to address post-harvest losses. Studies conducted by Abe-Inge *et al*. (2018) [17], Farhana *et al*. (2018) [18], Omolola *et al*. (2015) [19], Rwubatse *et al*. (2014) [20] e Surendar *et al*. (2018) [21] have contributed significantly to the understanding of

drying process and their impacts. Drying is one of the most common operations in the agri-food industry, characterised by its ability to reduce product weight, slow microbial growth and mitigate undesirable biochemical reactions [22], as well as the possibility to recover supply chain waste and contribute to value-added food. However, different drying methods can alter the physico-chemical and nutritional properties of dried products, despite increasing their shelf life. Economical drying techniques and improvements in processing methods are needed to mitigate the negative impact of high temperatures on food [23]. Physical quality, particularly colour, is essential for consumer choice. Colour changes in dried fruit are associated with high sugar levels and may result from the degradation of carotenoids, oxidation of vitamin C and the formation of browning pigments [24]. Therefore, preliminary treatments, such as *dipping* in solutions with natural antioxidants, are often performed to reduce adverse effects during drying and subsequent storage [25,26]. Another crucial factor for the shelf life of dried products is the storage method. In fact, Modified Atmosphere Storage (MAP) is considered a supplement to maintaining optimal conditions in terms of reducing the rate of fruit respiration [27,28]. The main gases used in MAP are CO_2, O_2 and N_2, which influence the density of bacterial and viral populations, reduce the possibility of fruit sticking [29,30] and keep the packaging volume from collapsing on product, without altering the sensory characteristics of the food [31]. However, the choice of each or their combination depends on the product to be stored.

The aim of this study was to assess the impact of drying on mango fruits (cv. Tommy Atkins) harvested at two different pre-harvest ripening stages, with a focus on their physicochemical, microbiological, nutraceutical, and sensory properties. Additionally, the study aimed to evaluate the effectiveness of modified atmosphere packaging (MAP) in preserving the quality of the dehydrated products during storage.

2. Materials and methods

2.1 Plant material

The plant material used in this study consisted of mango (*Mangifera indica* L.) fruits of the Tommy Atkins cultivar (cv.). Fruits were selected among waste based on two distinct ripeness grades: Green Ripe (11±1°Brix) and Mature Ripe (16±1°Brix), corresponding to the third commercial grade as defined by the BBCH scale [32]. The fruits were harvested in Sant'Agata di Militello, Messina, Sicily, Italy. A total of 100 fruits were utilized for the analysis, with an average weight of 500 g per fruit across the different ripeness stages.

2.2 Experimental design

Whole fruits were washed and disinfected with a 2% chlorine-water solution for 20 minutes. Then, the fruits were peeled and cut lengthwise with a curved knife into two halves, from which regular cubes approximately 3 ± 1 cm thick were obtained. These were immersed in an antioxidant solution containing ascorbic acid and citric acid at concentrations of 0.5g/0.5L for 2 minutes. Several preliminary tests were conducted to determine the appropriate drying time/temperature combination (data not shown). For reasons of time efficiency, the experimental protocol adopted involved drying the fruits using a convective hot-air dryer (Ausla, Model Ausla o4m0ggwtfb-13, Italy) at a temperature of 70 °C for 12 hours (aw = 0.66).Dried cubes were stored in PA/PE (polyamide/polyethylene) bags in active MAP (modified atmosphere packaging) with 100% N_2 (MAP N_2) and in passive MAP (MAP P), with a digitally controlled packaging machine (VM 16 Orved S.p.A, Musile di Piave, Venice, Italy) and then stored at room temperature (20±1°C) for a period of 30 days. The bags had the following characteristics: 80% PA/ 20% PE, thickness 90 µm, volume 500 cm^3, oxygen permeability 47.6 cm^2/ (m^2 day atm) and water vapour transmission rate 3.9 g/ (m^2 day atm) [32]. The dried cubes were divided into lots as follows: 50 g sample per bag × 4 treatments (GP, GN_2, MP and MN_2) × 3 replicates × 30 days of analysis (D0 - immediately after drying, D10, D20, D30). Ultimately, the treatments were:

- Green MAP P (GP);
- Mature MAP P (MP);
- Green MAP N_2 (GN_2);
- Mature MAP N_2 (MN_2).

Dried cubes were analysed at ten-day intervals (D0, D10, D20 and D30).

2.3 Physical-chemical analysis

After drying and before packing in MAP, the following parameters were evaluated on a representative sample of ten cubes:

- Colour (CIELab*);
- Firmness (N);
- Longitudinal (LD) and Transverse Diameter (DT) (mm).

The colour of the samples was determined based on the CIE L*a*b* colour system [brightness (L*); red/green (a*); yellow/blue (b*)] using of digital colourimeter (CR-400 Chroma Meter, Minolta, Japan). Colourimeter calibration was performed against a white plate (illuminants C: Y ¼ 89.53, x ¼ 0.3247, y ¼ 0.3198) before each measurement. For

each sample, the parameters L*, a* and b* were measured and the averages of all measurements were determined for each package. Values of L*, a* and b* read by the Chroma meter were then converted into Chroma (C*) and hue angle (h°) via Equations (1), [33]

$$C* = \sqrt{(a^{*2} + b^{*2})} \quad (1)$$

and (2) [5]

$$h° = \begin{cases} arctan\left(\frac{b*}{a*}\right) \; if \; a* \geq 0 \; and \; b* \geq 0 \\ 180 + arctan\left(\frac{b*}{a*}\right) \; if \; a* < 0 \\ 360 + arctan\left(\frac{b*}{a*}\right) \; if \; a* \geq 0 \; and \; b* < 0 \end{cases} \quad (2)$$

respectively.

Firmness was measured using an analogue penetrometer (3mm) (Turoni TR5325, Forlì, Italy) and expressed in Newtons (N). Transverse (DT) and longitudinal (DL) diameter was determined using a digital caliper (Turoni TR53307, Forlì, Italy) and expressed in mm. The dimensional changes of mango fruits during storage were expressed as the percentage of shrinkage in longitudinal and transverse diameter. These attributes are calculated as shown in Eq (3) and (4) [34]:

$$Shrinkage \; in \; longitudinal \; diameter \; (\%) = \left(\frac{D_0 - D}{D_0}\right) \quad (3)$$

$$Shrinkage \; in \; transverse \; diameter \; (\%) = \left(\frac{D_0 - D}{D_0}\right) \quad (4)$$

where D_0 and D represent the initial diameter (D0-mm) and the diameter of the samples at each observation day (D10, D20 and D30) respectively.

2.4 Nutritional analysis

The ash content was determined following the procedure outlined by the Association of Official Analytical Chemists (AOAC) [35]. Protein content was measured using the Kjeldahl method [36]. Fat content (S.S.G.) was extracted via acid hydrolysis using an HCl (1:4) solution, followed by filtration and dehydration. After solvent evaporation, residual fat was determined gravimetrically using Soxhlet extraction with petroleum ether [37]. Riboflavin (vitamin B2) was extracted in an autoclave with diluted H_2SO_4, subjected to enzymatic treatment, and quantified via high-performance liquid chromatography (HPLC) with fluorescence detection, following AOAC official methods. Ascorbic acid (vitamin C) was extracted by drying a methanolic extract (100 mg) and dissolving it in 10 mL of 1% metaphosphoric acid. The extract was incubated for 45 minutes at room temperature, filtered

through Whatman No. 4 paper, and 1 mL of filtrate was mixed with 9 mL of 2,6-dichlorophenolindophenol. Absorbance was measured at 515 nm within 30 minutes against a blank, with ascorbic acid content calculated using an L-ascorbic acid calibration curve (0.02–0.12 mg mL^{-1}) [38,39]. Total polyphenols were analysed following Singleton and Rossi's method [40]. Phenolic content was expressed as milligrams of gallic acid equivalents (GAE) per 100 g of dry matter. All analyses were performed in triplicate to ensure accuracy.

2.5 Water activity detection

The water activity was measured using the AquaLab series TE, 4TEV, DUO instrument. All analysis was performed in triplicate. For each sample, the M (absolute humidity) value, aw and the moisture absorption isotherms were determined using an AquaLab analyser. The interpolation of the data was evaluated from the mean error value (E%) defined by equation (5):

$$E\% = \frac{100}{N}\sum_{i=1}^{N}\left|\frac{X_{spe,i}-X_{prev,i}}{X_{spe,i}}\right| \quad (5)$$

in the equation N represents the number of interpolated points, X_{spe} the test point and X_{prev} the point obtained using the mathematical model. To determine the temperature dependence of a_w, Arrhenius's law was applied using equation (6):

$$ln\ a_w = ln\ a_{w,0} - \frac{\Delta H_a}{RT} \quad (6)$$

where, a_w is the activity of water, R is the universal constant of the gas and T is the temperature. Through Arrhenius's law, the value of the desorption energy (ΔHa) was traced. It was determined that ΔHa was a function of absolute humidity (M), the value of M decreases as the value of ΔHa increases. It calculated the drying kinetics under different experimental conditions down to a minimum value of 15% of the residual water content.

2.6 Total Polyphenols Content

Total polyphenols analysis was carried out according to Singleton and Rossi. Briefly, 1 mL of the diluted and filtered sample obtained as above with distilled water was mixed with 1 mL Folin–Ciocâlteu reagent, and after 3 min, 2.5 mL of sodium carbonate (Na_2CO_3) was added. The solution was brought to a final volume of 25 mL and incubated in the dark for 1 h. The absorbance was measured with an UV-VIS spectrophotometer at 725 nm. The equation of the calibration curves is obtained with the gallic acid (3,4,5-trihydroxybenzoic acid) standard. The phenolic content was expressed as mg of gallic acid equivalents (GAE) per 100 g of dry matter. All the analyses were made in triplicate.

2.7 Microbiological analysis

Mango slices obtained immediately after dehydration at 70 °C for 12 h (D0) and at subsequent storage times (D10, D20, and D30) were subjected to decimal serial dilutions. Microbial populations were enumerated on the following media: Plate Count Agar (PCA) for total mesophilic microorganisms (2 d, 30 °C) and total psychrotrophic microorganisms (7 d, 7 °C); Pseudomonas Agar Base (PAB) supplemented with CFC for Pseudomonadaceae (2 d, 25 °C); Violet Red Bile Glucose Agar (VRBGA) for Enterobacteriaceae (1 d, 37 °C); Baird-Parker (BP) agar for coagulase-positive staphylococci (2 d, 37 °C); Hektoen Enteric Agar (HEA) for presumptive *Escherichia coli* and *Salmonella* spp. (1 d, 37 °C); Listeria Selective Agar Base (LSAB) for *Listeria monocytogenes* (1 d, 37 °C); and Yeast Extract Peptone Dextrose (YPD) agar for yeasts (2 d, 25 °C) and filamentous fungi (7 d, 25 °C). Detection of *L. monocytogenes* and *Salmonella* spp. was performed on 25 g of dried-mango sample according to ISO 11290-1 and ISO 6579-1, respectively. All analyses were carried out in triplicate using media purchased from Oxoid (Basingstoke, UK).

2.8 Sensory analysis

Sensory evaluation of the samples consisted into two approaches. Determining consumers' acceptance of the samples by evaluating the global liking and purchase intention and obtaining a description of the main samples attributes by means of the CATA-test in combination with the *Ideal Product*. As previously explained this combined approach allow to obtain relevant information not only of our specific samples but also of the characteristics that a dehydrated mango should have to satisfy consumers [41,42].

In total, 100 persons participated in the study. Participants were aged between 18 and 59 years, and the male/female ratio (%) was 40/60. The evaluations were carried out in a standardized test room (ISO 8589, 2018). Samples consisted of two portions of dried mango: Green MAP P (GP), Mature MAP P (MP), Green MAP N_2 (GN2), Mature MAP N_2 (MN2), and commercial mango samples, represented by commonly marketed dried mango products typically produced through industrial dehydration and often formulated with added ingredients such as preservatives, acidulants, sweeteners, and other additives to enhance shelf life, color stability, and flavor consistency. Samples were coded with three-digit random numbers and were presented monadically accordingto a Williams' Latin-square design (Meyners et al., 2016). Consumers were provided with water to cleanse palate between samples. Consumers were instructed to firstly evaluate the sample visually and indicate how much they liked the aspect using a 9-point hedonic scale anchored at 1 – "*dislike very much*" and 9 – "*like very much*" [43]. Then, they were asked to taste the sample

and indicate their global liking (9-point hedonic scale) and their purchase intention on a 5-point scale ranging from 1 – "*I definitely would not buy*" to 5 – "*I definitely would buy*" [44]. Next, they were asked to answer the CATA question with 23 terms related to sensory characteristics of dried mango. After testing each of the five samples, consumers were asked to complete the CATA question to describe their Ideal dried mango. The descriptors included on the CATA list were obtaining in a pre-test performed by a group of six semi-trained panellists who evaluated the samples to identify those specific characteristics that allow better differentiating them [42]. On the final list, attributes were ordered in such a way that they were likely to be perceived. The selected terms were: *natural colour that remember to mango, aromatic, easy to chew, hard to chew, rubbery, floury, too dry, sticks to teeth, tasteless, non-acid, medium acidity, very acid, non-sweet, slightly sweet, very sweet, refreshing flavour, slightly mango flavour, intense mango flavour, overripe fruit flavour, immature fruit flavour, caramel flavour, off-flavour, tropical fruit flavour*. Finally, when consumers had finished their mango assessments, they answered demographic questions about the gender, age, and frequency of dried fruit consumption. The response options for fruit consumption were as follows: "*Almost every day*", "*At least twice a month*", "*Sporadically*" or "*Never*". The experimental procedure was explained and a written consent indicating voluntary participation was obtained from each participant prior the beginning of the study [45].

2.9 Statistical analysis

The results of the chemical-physical, microbiological, and nutritional analyses were analysed using the XLStat software version 2020.3.1 for Excel (Addinsoft, New York, USA). Averages were evaluated on the data, and one way variance analysis (ANOVA) was performed, and pair comparisons were evaluated with the Tukey's test. The differences were considered significant for $p<0.05$. The Kruskall Wallis test followed by Dunn´s multiple comparisons test was applied to evaluate difference in acceptance scores among samples ($p<0.05$). The frequencies of citation of each attribute in the CATA question were determined for each sample. The non-parametric Cochran's Q test was performed on the raw binary CATA data to determine significance among samples for each sensory attribute ($p<0.05$). Only those attributes that contributed significantly to differentiate samples were included in the correspondence analysis (CA) performed to obtain a plot of the sample's distribution based on consumers' description. Penalty analyses of the CATA data including the Ideal was used to determine the attributes with impact on acceptability. For each attribute, for those consumers who selected it in the Ideal, the difference in average liking of those mango

samples having the attribute and those mango samples not having the attribute was calculated. Those attributes showing a significantly ($p<0.05$) higher acceptability when the attribute was present were considered "must have" attributes. For each attribute, for those consumers who did not select it in the Ideal, the difference in average liking of those mango samples having the attribute and those mango samples not having the attribute was calculated. Those attributes showing a significantly ($p<0.05$) lower acceptability when the attribute was present were considered *"must NOT have"* attributes.

3. Results and Discussion

3.1 Raw material

Ripening is a complex process characterised by significant changes in the physiological, biochemical and organoleptic properties of a given product, culminating in the achievement of an acceptable quality [46].

Table 1. Quality characteristics of fresh mango fruit (n=20). L* (luminosity); a* (green/red); b* (blue/yellow); C* (chroma); h° (hue angle); FIRMNESS (N); TSSC (content of soluble solids, solid brix); TA (titratable acidity, g/L^{-1} citric acid). Values are represented as mean ± standard deviation of the obtained data.

Parameter	Green	Mature
L*	56.01±4.92	61.48±4.37
a*	-7.34±2.72	-3.11±7.21
b*	26.65±6.98	43.22±7.13
C*	27.88±6.37	43.99±6.57
h°	179.46±1.34	178.58±0.01
FIRMNESS (N)	81.75±8.00	68.50±7.50
TSSC (°Brix)	11.30±0.43	16.6±0.45
TA	0.29±0.01	0.11±0.01
TSSC/TA	3.44±0.13	6.22±0.01

Table 1 shows the results of the preliminary physicochemical analysis conducted on a sample of 20 fresh fruits. The mango BBCH scale [47] was used to determine the ripening stage of mango fruits. The fruits considered fell within the ripening stage range defined by the BBCH scale (800–801). In particular, the Green samples were classified as stage (800), which corresponds to the onset of ripening: fruit growth is complete, but peel colour remains green, and internal physiological changes such as sugar accumulation and softening are beginning. These fruits were fully developed and exhibited creamy green flesh, confirming their classification. The Mature samples, instead, belonged to stage (801), which indicates full

ripeness: the fruit shows typical colour, flavour, and texture, and is considered ready for harvest [47], indicating that they were at a more advanced stage of ripening. These fruits were in the beginning stage of skin colour change with texture typical of this stage. As the degree of ripeness increases, both brightness and hue angle decrease. Furthermore, a negative correlation emerged between total soluble solids content (TSSC) with titratable acidity (TA) and firmness (N), meaning that as TSSC increased, both TA and N values decreased. These relationships but also the difference in the TSSC/TA ratio underline the significant differences in the ripening stages between the samples, confirming that the Green samples were at an earlier stage of ripening than the Mature samples. Similar results were obtained by Dissa *et al*. [48] both in terms of absolute values of the maturation indices (TSSC and TA) and the degree of maturation represented by the TSSC/TA ratio. Fruit ripening is accompanied by significant biochemical and structural changes, including alterations in carbohydrate, organic acid, lipid, and phenol levels, as well as modifications to volatile compounds and polysaccharide structure [49]. The observed decline in brightness and hue angle during ripening highlights the visual transition from a greenish yellow to an orangey-red colour, a hallmark of ripening progression [50]. These findings provide important insights into the physicochemical changes occurring during mango ripening and their implications for both sensory quality and potential processing applications.

3.2 Physical-chemical analysis

3.2.1 Colour

An important quality parameter for consumers is visual appearance. Colour is the attribute that most influences consumers when purchasing fruit or other foods. In fact, it is considered the most important visual attribute in the perception of quality [51]. Drying can lead not only to changes in the firmness, flavour and taste, but also in the characteristic colour of the fruit [19,52]. Table 2 shows the results of the colourimetric analysis represented by the average values of L*, a*, b*, C* and h° conducted during the 30-day storage period for the dried mango samples.

Table 2. Values of L*, a*, b*, C* and h° of the dried mango samples analysed during the experiment. Values are represented as mean ± standard deviation. Values followed by different letters indicate statistically significant differences in the same treatment during storage; *ns* indicates no difference. Presence of an asterisk indicates statistically significant differences between the two MAP treatments with *-*p<0.05*, **-*p<0.01* and ***-*p<0.001* during storage; no asterisk indicates no difference.

	Day	***L****	***a****	***b****	***C****	***h°***
FRESH		81.20±2.91	-6.31±0.25	41.29±2.77	27.88±6.37	178.58±0.01
GP	0	75.54±3.42^{a}	4.45±1.21^{ab}	66.48±4.53^{ns}	66.67±4.52^{ns}	86.20 ±1.03^{a}

	10	68.55±5.79$^{ab, **}$	4.72±1.82^{b}	65.29±6.37$^{ns, *}$	65.49±6.39$^{ns, *}$	85.93±1.60^{a}
	20	68.69±4.79$^{b, ***}$	3.61±1.37^{b}	64.82±4.58^{ns}	64.98±4.60^{ns}	86.86±1.54^{a}
	30	72.45±5.65^{ab}	7.06±3.95^{a}	60.34±5.69^{ns}	60.85±5.89^{ns}	83.50±3.47^{b}
GN_2	0	75.54±3.42^{ns}	4.45±1.21^{ab}	66.48±4.53^{a}	66.67±4.52^{a}	86.20 ±1.03^{a}
	10	74.81±6.34$^{ns, **}$	3.04 ±1.20^{b}	59.00±5.85$^{b, *}$	59.03±5.85$^{b, *}$	87.02±1.31^{a}
	20	76.89±3.78$^{ns, ***}$	2.19 ±0.57^{b}	59.56±6.57^{ab}	59.60±6.55^{ab}	87.87±0.6^{a}
	30	72.25±6.08^{ns}	6.89±3.44^{a}	61.94±4.66^{ab}	62.39±4.86^{ab}	83.77±2.87^{b}
FRESH		66.10±1.76	4.88±0.75	69.79±1.99	43.99±6.57	179.46±1.34
MP	0	70.53±6.16^{ab}	5.07±1.64^{b}	64.82±2.46^{ns}	65.09±2.38^{ns}	84.68±1.84^{ns}
	10	66.02±7.38^{bc}	6.95±4.14$^{ab, ***}$	63.34±8.98^{ns}	63.87±8.89^{ns}	83.68 ±4.07$^{ns, ***}$
	20	62.93±6.16^{c}	9.15±3.00^{a}	63.12±7.31^{ns}	63.86±7.20^{ns}	81.65±2.97^{ns}
	30	73.60±5.93^{a}	5.42±2.46^{b}	64.97±6.11^{ns}	65.24±5.99^{ns}	85.15±2.45^{ns}
MN_2	0	70.53±6.16^{a}	5.07±1.64^{b}	64.82±2.46^{ab}	65.09±2.38^{ns}	84.68±1.84^{a}
	10	60.79±4.89^{b}	11.28±2.27$^{a, ***}$	60.88±5.96^{ab}	61.96±5.86^{ns}	79.45 ±2.35$^{b, ***}$
	20	62.98±5.96^{b}	10.52±1.51^{a}	60.05±7.14^{b}	61.00±6.93^{ns}	79.91 ±2.19^{b}
	30	71.15±6.52^{a}	6.88±2.90^{b}	66.33±5.72^{a}	66.75±5.60^{ns}	84.05±2.74^{a}

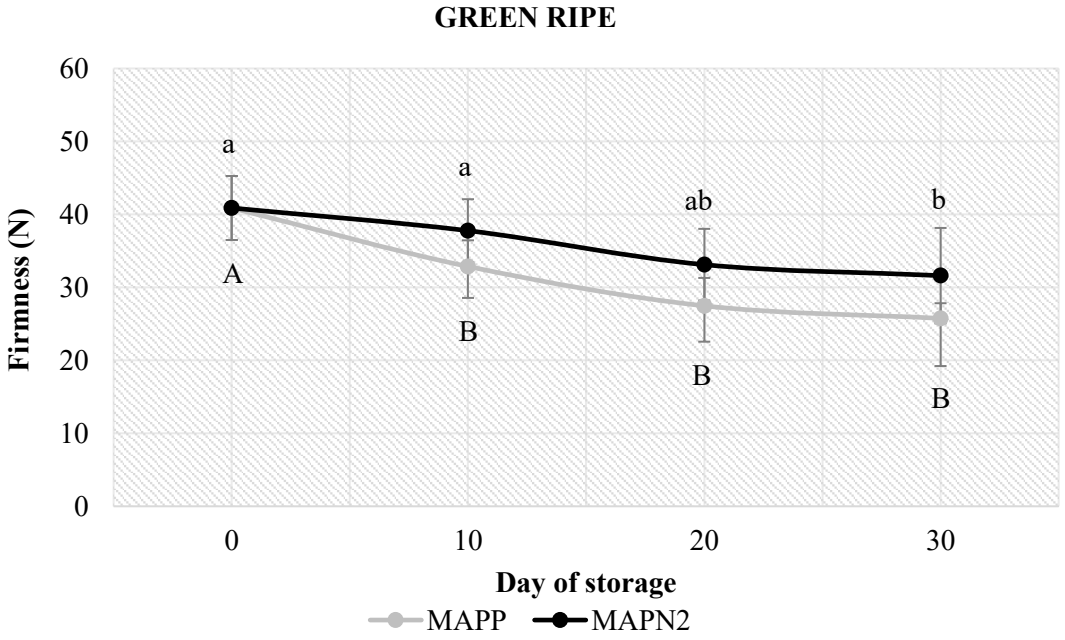
The colourimetric analysis of mango samples subjected to different storage conditions reveals important information about variations in colour parameters during the 30-day observation period. In the less mature mango samples (Green), fluctuations in brightness values (L*), particularly observed at D10 and D20, suggest a certain instability that, however, resolves without significant variations compared to the initial value at D30. This may indicate an initial adaptation phase to the storage process. The use of nitrogen (GN_2) appears to help maintain a stable brightness value, suggesting a positive impact on colour preservation. On the other hand, in the more mature samples (Mature), a significant loss in brightness (L*) is observed at D20, indicating greater sensitivity to the storage process. However, the value nearly returns to the initial levels at D30, suggesting a potential subsequent stabilization. The increase in a* values in the Mature samples at D20 may indicate a greater intensity of red colour during the early stages of storage, with both treatments (MAP P and MAP N_2) showing similar results. The variations in b* and C* parameters are negligible. The analysis of samples subjected to modified atmosphere packaging (MAP) highlights substantial differences. In the case of the Green samples, MAP P seems to preserve brightness (L*) and colour saturation (C*) compared to nitrogen (MAP N_2). Conversely, in the Mature samples, nitrogen shows a more significant effect on hue parameters (a*) and hue angle (h°). The response of the colourimetric parameters of mango to storage is influenced by the degree of ripeness and storage conditions. While passive

atmosphere storage appears to be more effective in preserving colourimetric characteristics in less mature samples (Green), nitrogen may have a positive impact on specific parameters in more mature samples.

The colour change in dried fruit is mainly attributed to the degradation of carotenoids during exposure to heat, oxygen, and light. Studies have reported that drying has a significant impact on changes in colour, saturation, hue angle, and browning index. Tadlo *et al*. [53] has been reported that these significantly increased due to the Maillard reaction when samples were subjected to solar drying and integrated oven drying. In our case, no significant variation was observed, confirming the appropriate time/temperature balance. Similar results were obtained from the hue angle, which decreased. Modified atmosphere storage helped maintain colour parameters during storage.

3.2.2 Firmness

One of the main changes involving the structure of plant tissue during drying is the change in firmness [54]. The removal of water content from the fruit during the drying process leads to a loss of volume and weight, as well as a change in mechanical properties, such as hardness and crispness [55]. Fruit firmness is one of the most important quality properties, as it influences the acceptability of the fruit to consumers [56]. Manrique *et al*. (2004) [57] and Mafra *et al*. (2001) [58] report that changes in fruit firmness are largely determined by changes in the structure of the fruit cell wall and polysaccharides in the central lamella.



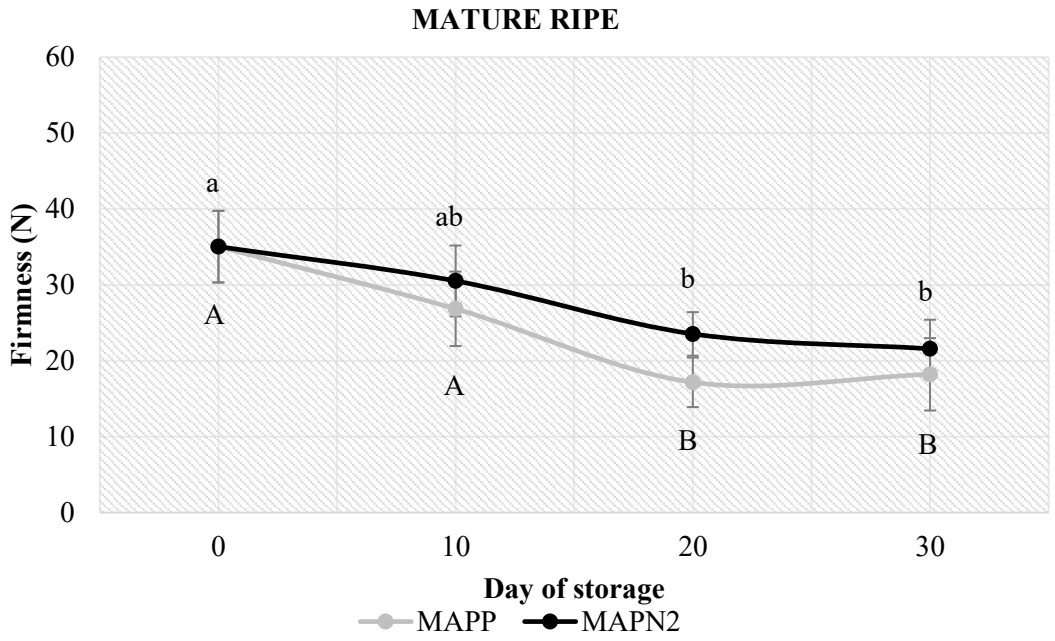


Figure 1. Variation in firmness (N) of Green and Mature mango samples. Values are represented as mean ± standard deviation. Different letters indicate statistically significant differences ($p<0.05$) in the same treatment during storage. Asterisks indicate statistically significant differences between the two MAP treatments with *-*$p<0.05$*, **-*$p<0.01$* and ***-*$p<0.001$* during storage; no asterisk indicates no difference.

The analysis of mango cube firmness, divided between Green and Mature samples during the storage period, provides useful insights into the structural stability of the product (Figure 1). In Green samples, a significant decrease in firmness was observed in those stored with passive atmosphere packaging (MAP P), decreasing from 40.87 N to 25.75 N after 30 days. In contrast, samples stored in MAP N_2 maintained greater firmness stability, reaching a value of 31.61 N at the end of the period. This suggests that nitrogen packaging contributes positively to maintaining the firmness of less ripe mango cubes during storage. In the Mature samples, firmness decreased steadily for both MAP treatments during the 30-day storage period, with no statistically significant differences between MAP P and MAP N_2. This result indicates that both packaging methods similarly affect the firmness of the riper mango cubes. Given the lower costs of MAP P compared to nitrogen, this method may represent a more advantageous option for Mature samples. When examining the percentage of firmness loss between Green and Mature samples, the Mature samples exhibited greater structural sensitivity, with a loss of 43.20% compared to 27.37% for the Green samples after 30 days. This highlights the importance of considering the ripeness level when designing storage strategies, as less ripe fruits tend to better maintain their structure during storage. Firmness loss during storage is also influenced by the characteristics of the packaging material used [59]. In The 80% PA/20% PE compound demonstrated a certain degree of water vapor permeability [60], leading to a reduction in firmness at the end of the storage period. Pectins

and hemicelluloses in the cell walls of dried foods are the material that provides structural integrity and mechanical strength [61,62]. The constant flow of water vapour to the inside of the bags probably resulted in a rehydrating effect on both samples, imparting flexibility, and loss of texture. Thus, the water vapour permeability of packaging plays a crucial role in food preservation, particularly in maintaining the physical characteristics of food.

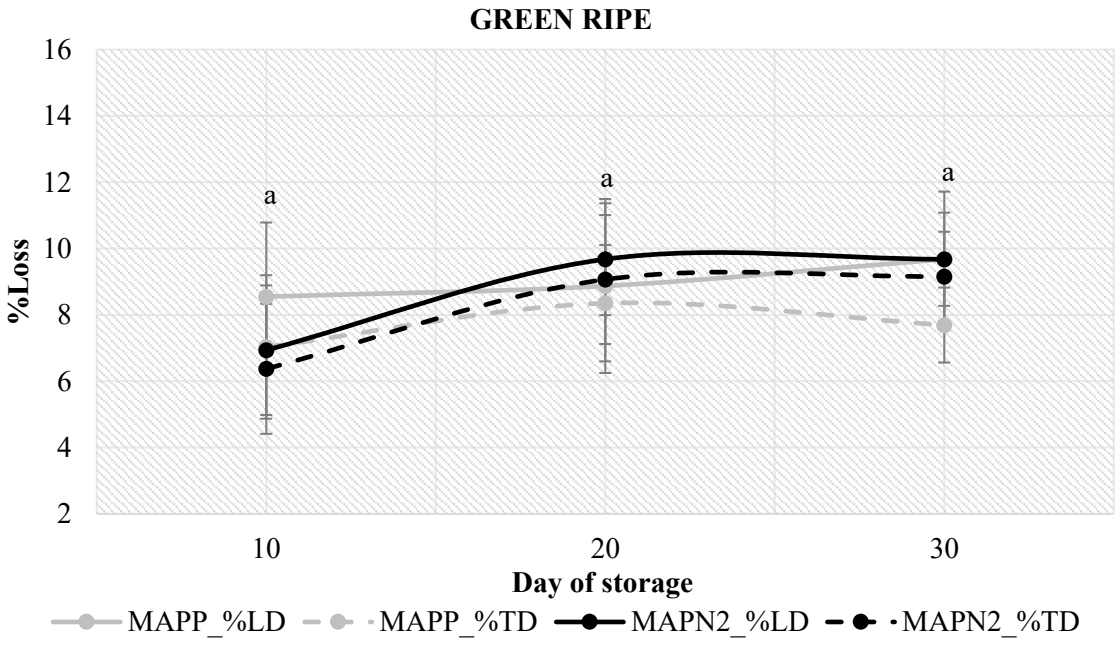
3.2.3 Longitudinal and transverse diameter

During the drying process, the longitudinal (LD) and transverse (TD) diameter values decreased in both samples. In fact, the porous and hygroscopic nature of fruit and vegetables makes them highly shrinkable during drying [63].

Table 3. Longitudinal (LD) and Transverse (TD) diameter of fresh and dried mango. Values are represented as mean ± standard deviation. Values followed by different letters indicate statistically significant differences ($p<0.05$).

	SAMPLE	*GREEN*	*MATURE*
LD	Fresh	38.20±1.64[a]	38.20±1.64[a]
	Dried	31.00±3.46[b]	29.42±3.99[b]
TD	Fresh	26.65±2.13[a]	26.65±2.13[a]
	Dried	22.75±3.46[a]	21.50±2.46[b]

The effect of drying on the reduction of longitudinal diameter (LD) and transverse diameter (TD) of the Green and Mature samples is presented in Table 3. Drying had a significant effect ($p<0.05$) on the shrinkage of the cubes. Specifically, LD decreased significantly in the Green by about 7 mm and in the Mature 9 mm. TD was significantly reduced only in the Mature samples with a reduction of about 5 mm. Comparing the stage of maturity, the Mature samples had the highest shrinkage.



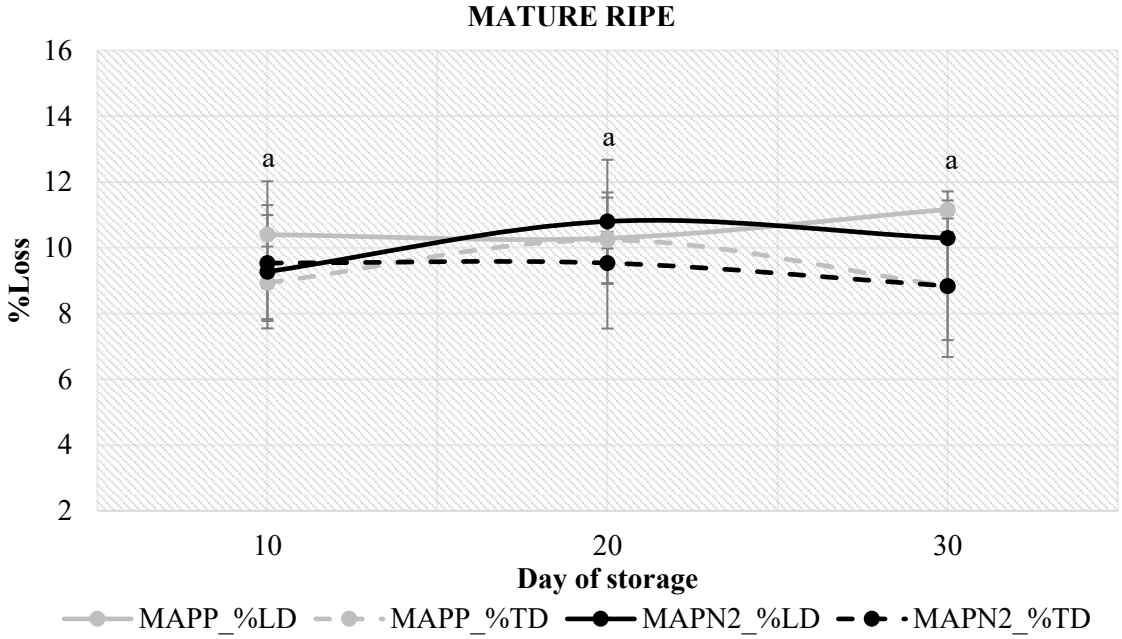


Figure 2. Variation in % loss of LD and TD parameters of the Green and Mature mango samples. Values are represented as mean ± standard deviation. Different letters indicate statistically significant differences ($p<0.05$) in the same treatment during storage. Asterisks indicate statistically significant differences between the two MAP treatments with *-$p<0.05$, **-$p<0.01$ and ***-$p<0.001$ during storage; no asterisk indicates no difference.

During storage, the percentage loss of LD and TD compared to D0 (day post drying) was evaluated for Green and Mature samples stored with the two MAP techniques. The results are presented in Figure 2. From the data obtained, a higher percentage loss was observed for all days of observation in the Mature samples. During the 30-day storage period, the highest loss was observed for the LD parameter stored in MAP P (11.16%). However, no significant difference was detected either in the days of storage or in the comparison with MAP N_2. For the TD parameter, the greatest loss (10.23%) was observed at D20 in the samples stored in MAP N_2. However, the two parameters did not show significant losses either during the entire observation period or when comparing the two storage techniques. In the Green samples, the losses were smaller. The highest loss of LD (9.67%) was observed at D20 in the samples stored in MAP N_2 while at D30 the TD parameter reached the highest loss value (9.15%) in the samples stored in MAP N_2. However, statistical analysis showed no significant differences between days and between storage treatments. Water removal and high temperatures during food drying produce significant stress that can lead to the collapse of cells and pores, change of shape and decrease in size [64,65]. Shrinkage will have an impact on the size, structure, colour, taste and aroma of the product [65,66]. Shrinkage can be influenced by drying conditions, food structure, cell structure, sample shape and porosity [66–68]. Ideal shrinkage assumes that the volume reduction of food during drying follows

the amount of water removed during the entire process [62]. However, a study conducted on banana, pineapple and mango drying [69] revealed that there is no linear relationship between water content and shrinkage of the samples. Although our samples did have shrinkage, the quality of the product was not affected, as was also confirmed by sensory analysis. Probably the combination of time/temperature and the high presence of fibrous structures (pectin, hemicellulose and cellulose) in the mangoes contributed to keeping the cell wall structure of the samples as unaltered as possible during dehydration.

3.2.4 Nutritional analysis

Table 4 shows a series of significant trends regarding the effects of drying and storage on the nutritional properties of dehydrated mango. The most significant effects were observed for crude protein, soluble solids (S.S.G), total sugars, and vitamins (B3, B1, C, A).

Table 4. Evolution of the nutritional values of dehydrated mango stored under MAP-P and MAP-N_2. Values are expressed as mean ± standard deviation. Different letters indicate statistically significant differences ($p < 0.05$) within the same treatment during storage. Asterisks denote statistically significant differences between the two MAP treatments during storage: * $p < 0.05$, ** $p < 0.01$, *** $p < 0.001$; absence of an asterisk indicates no significant difference.

		Pre-drying		Post-drying			
	D	**FRESH G**	**FRESH M**	**GP**	**GN_2**	**MP**	**MN_2**
		0.34±0.02	0.38±0.01				
CRUDE PROTEIN	0			$0.33\pm0.03^{ns,\ AB}$		$0.36\pm0.02^{ns,\ AB}$	
	10			0.33 ± 0.03^{ns}	0.36 ± 0.02^{A}	0.38 ± 0.01^{ns}	0.36 ± 0.03^{A}
	20			0.32 ± 0.03^{ns}	0.29 ± 0.03^{B}	0.36 ± 0.02^{ns}	0.31 ± 0.02^{AB}
	30			0.35 ± 0.01^{ns}	0.31 ± 0.01^{AB}	0.33 ± 0.03^{ns}	0.31 ± 0.01^{B}
		0.42±0.02	0.32±0.01				
S.S.G	0			0.40 ± 0.01^{aA}		$0.33\pm0.01^{ns,\ B}$	
	10			$0.42\pm0.01^{a***}$	$0.32\pm0.01^{B***}$	0.32 ± 0.01^{ns}	0.37 ± 0.02^{AB}
	20			$0.42\pm0.02^{a***}$	$0.32\pm0.02^{B***}$	0.32 ± 0.01^{ns}	0.38 ± 0.12^{AB}
	30			0.33 ± 0.00^{b}	0.34 ± 0.02^{B}	$0.27\pm0.02^{ns***}$	$0.45\pm0.00^{A***}$
		0.82±0.04	0.73±0.02				
GREEN FIBER	0			$0.77\pm0.05^{b,\ ns}$		0.93 ± 0.02^{aAB}	
	10			0.76 ± 0.05^{b}	0.85 ± 015^{ns}	0.73 ± 0.02^{b}	0.85 ± 015^{AB}
	20			0.76 ± 0.01^{b}	0.70 ± 0.02^{ns}	0.75 ± 0.07^{ab}	0.76 ± 0.05^{B}
	30			$0.99\pm0.01^{a**}$	$0.76\pm0.05^{ns**}$	$0.68\pm0.02^{b***}$	$0.99\pm0.01^{A***}$
		8.79±0.08	13.36±0.03				
TOTAL SUGARS	0			12.35 ± 0.11^{aA}		11.21 ± 0.03^{bB}	
	10			$12.01\pm0.28^{a***}$	$10.55\pm0.49^{B***}$	13.36 ± 0.03^{a}	14.17 ± 0.13^{A}

	20			8.79±0.08^{b***}	10.79±0.07^{B***}	12.18±0.06^{b***}	10.62±0.54^{B***}
	30			13.17±0.05^{a***}	10.99±1.08^{B***}	11.13±011^{b}	11.33±0.94^{B}
		0.50±0.02	0.33±0.01				
ASHES	0			0.46±0.02^{abA}		0.39±0.01$^{a, ns}$	
	10			0.38±0.01^{b}	0.32±0.07^{B}	0.33±0.01^{ab*}	0.43±0.02^{ns*}
	20			0.50±0.02^{a***}	0.32±0.07^{B***}	0.32±0.07^{ab}	0.38±0.04^{ns}
	30			0.38±0.02^{b}	0.38±0.05^{AB}	0.27±0.01^{b}	0.38±0.05^{ns}
		0.39±0.01	0.54±0.04				
VIT. B3 (Niacin)	0			0.46±0.02^{bA}		0.33±0.01^{bcC}	
	10			0.53±0.03^{a***}	0.39±0.01^{B***}	0.53±0.01^{a}	0.53±0.01^{A}
	20			0.43±0.00^{b***}	0.35±0.01^{B***}	0.36±0.00^{b***}	0.45±0.01^{B***}
	30			0.26±0.04^{c}	0.26±0.02^{C}	0.29±0.05^{c***}	0.46±0.02^{B***}
		0.01±0.02	0.06±0.00				
VIT. B1 (Thiamin)	0			0.05±0.00$^{a, ns}$		0.05±0.01$^{ab, ns}$	
	10			0.06±0.01^{a}	0.06±0.00^{ns}	0.04±0.00^{b}	0.06±0.00^{ns}
	20			0.01±0.02^{b***}	0.05±0.00^{ns***}	0.07±0.00^{a}	0.06±0.01^{ns}
	30			0.05±0.00^{a}	0.04±0.00^{ns}	0.01±0.02^{c}	0.05±0.00^{ns}
		85.24±0.63	111.92±2.99				
VIT. C (Ascorbic acid)	0			120.90±6.69^{aB}		127.08±4.55^{aA}	
	10			107.89±3.71^{b***}	130.15±2.25^{A***}	113.83±1.37^{b***}	138.28±2.54^{B***}
	20			85.16±0.56^{c***}	113.83±1.37^{B***}	127.24±1.19^{a***}	103.61±0.06^{C***}
	30			99.94±0.08^{b}	103.61±0.06^{C}	110.09±0.18^{b}	115.35±0.18^{D}
		0.54±0.02	0.88±0.00				
VIT. A	0			0.91±0.05^{abA}		0.79±0.05^{abB}	
	10			0.95±0.12^{a***}	0.67±0.01^{B***}	0.88±0.00^{ab}	0.91±0.05^{A}
	20			0.84±0.01^{ab}	0.88±0.02^{A}	0.90±0.01^{a}	0.87±0.03^{AB}
	30			0.78±0.01^{b}	0.88±0.00^{A}	0.78±0.01^{b}	0.80±0.07^{B}

Regarding crude protein, stability is observed before and after drying. It appears that the MAP treatment (P and N_2) shows no significant differences in crude protein levels throughout the entire analysis period. This may indicate a relative preservation of proteins regardless of the type of treatment applied. The observed stability in crude protein content before and after drying aligns with existing research indicating that dehydration processes, such as hot-air drying, effectively reduce water activity while preserving the nutritional value of fruits [70].

The S.S.G values reveal significant variations among the treatments. In particular, the Green MAP N_2 treatment shows significantly lower S.S.G compared to the other treatments, suggesting a possible influence of the treatment type, regardless of the drying phase. Total sugars show notable differences among the treatments throughout the analysis period. The Green MAP P treatment has higher values at D10, while the Mature MAP P treatment shows greater variability at D20 and D30. This suggests a complex interaction between the treatment type and total sugar content, with possible impacts on the nutritional quality of the substance. The lower S.S.G in the Green MAP N_2 treatment suggests that the type of modified-atmosphere packaging (MAP) can influence the concentration of soluble solids. This observation is consistent with studies demonstrating that MAP conditions can affect the quality attributes of dried fruits during storage [71]. Then, the differences in total sugars among treatments throughout the analysis period indicate a complex interaction between the type of MAP treatment and sugar content. Similar findings have been reported, where different drying methods and storage conditions significantly impacted the nutritional and antioxidant properties of fruits like mango, avocado, and tomato [72]. Finally, the vitamins (B_3, B_1, C, A) highlight differences among the treatments during all observation days. The Green MAP N_2 treatment shows lower levels of vitamins B_3 and B_1 compared to the other treatments, while the Mature MAP P treatment exhibits significant variability in vitamins C and A. These results underscore the importance of considering the type of treatment applied in the preservation of vitamins [73]. The commercial dehydrated mango has a slightly lower water activity than the mango used in the experiment. However, the values of total polyphenols are also much lower in absolute terms than the mango used in the experiment. The degradation of these could be attributed both to much more extreme thermal treatments to produce a lowering of the water activity, and to the fact that the commercial dehydrated mango was also treated with sulphites to avoid the possible development of moulds [73].

Table X. Water activity (aw) and total polyphenols (PT) of fresh, experimentally dried, and commercial mango samples. PT values are expressed as mg gallic-acid equivalents per gram of dry matter (mean ± SD, n = 3).

	AW	**PT (mg GAE $^{g-1}$)**
Fresh G	0,9695	-
GN_2	0,7015	5,65
MN_2	0,6672	4,83
GP	0,6399	7,91
MP	0,6643	7,62
Fresh M	0,9775	2,49

Commercial	0,6455	1,82

Compared with the fresh mature fruit (2.49 mg GAE g^{-1}), all experimental samples exhibited markedly higher total-polyphenol levels. Green MAP P and Mature MAP P showed the greatest concentrations (about 7–8 mg GAE g^{-1}), followed by Green MAP N_2 and Mature MAP N_2 (about 5–6 mg GAE g^{-1}). In contrast, the commercial dried mango had the lowest phenolic content (1.82 mg GAE g^{-1}), even lower than the fresh fruit. These findings indicate that the experimental dehydration protocols—particularly those employing the MAP P help preserve polyphenolic compounds, whereas the harsher processing conditions and use of sulphites in the commercial product led to a pronounced loss of antioxidants.

3.3 Microbiological analysis

According to the World Health Organization (WHO), dried fruits and vegetables—even with their characteristically low water-activity values—can act as carriers of food-borne pathogens [74]. Several of these microorganisms can survive for long periods on dried products and may cause illness because of their low infectious dose [75]. For this reason, the microbiological quality of the dehydrated mango slices produced in the present study was assessed before commercialisation. A culture-dependent approach targeted the principal spoilage and pathogenic microorganisms commonly linked to plant-derived foods [76]. Across all storage times (D0, D10, D20 and D30) and regardless of MAP formulation, counts of Pseudomonas spp., yeasts and filamentous fungi, organisms known to drive physicochemical spoilage in plant products [77], remained below the detection limit (< 2 log CFU g^{-1}). Likewise, no coagulase-positive *Staphylococcus*, *Escherichia coli*, *Listeria monocytogenes* or *Salmonella* spp. were detected during the entire storage period. These results comply with the microbiological criteria for dried fruit established by Commission Regulation (EC) No 2073/2005. The absence of the investigated microbial groups can be attributed both to the lethality of the hot-air dehydration step and to the stringent sanitation and handling procedures applied to the mango slices throughout processing [78].

3.4 Sensory analysis

3.4.1 Acceptance and Purchase Intention

The mean acceptance scores obtained for each dried mango sample after being visually evaluated and tasted are shown in Table 5.

Table 5. Mean acceptance score obtained for the dried mango sample after being visually assessed and tasted. Values are indicated as mean ± standard devi. Different letters indicate statistically significant differences ($p<0.05$).

SAMPLE	*Liking (visual)*	*Liking (tasting)*
Commercial	5.85 b	6.19 c
GP	4.38 a	4.47 a
GN_2	4.77 a	4.14 a
MP	6.18 b	6.12 c
MN_2	6.05 b	5.65 b

Data from visual evaluation revealed a marked effect of the maturity stage on visual acceptance and a non-effect of the packaging atmosphere. Thus, both Mature samples (MP and MN_2) received liking scores around 6.1, without significant differences from the commercial sample [79]. Consumers showed lower acceptance of the Green samples; both GN_2 and GP obtained scores lower than 5. A different pattern was detected when consumers were asked to evaluate the samples after tasting them. In this case, both maturity stage and packaging atmosphere influenced liking scores. Green samples were again the lowest scoring, with no differences between GP and GN_2. However, MN_2 scored lower than MP. Consumers did not show preferences between the commercial sample and MP; both scored slightly above 6.

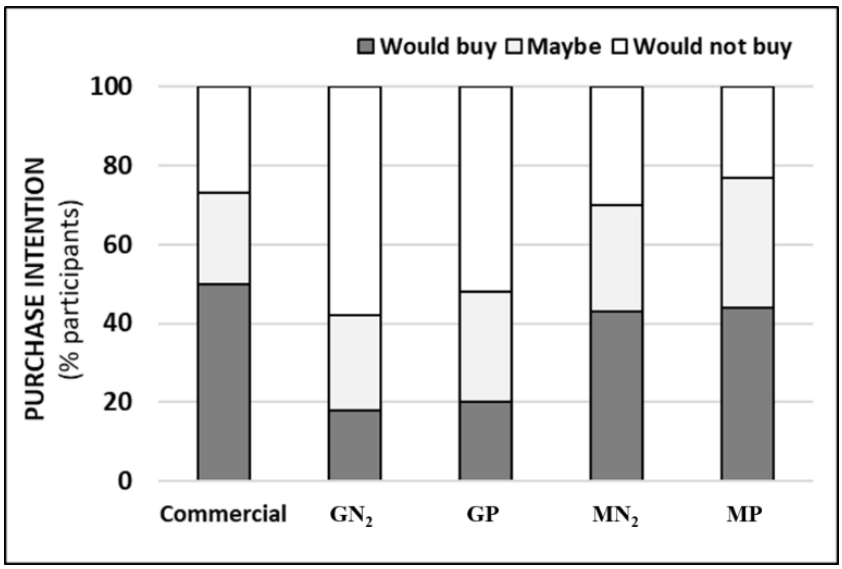


Figure 3. Percentage distribution of purchase intention (n = 100 consumers) for the five dehydrated-mango samples: Commercial, GN_2 (Green + MAP N_2), GP (Green + MAP P), MN_2 (Mature + MAP N_2) and MP (Mature + MAP P). Each stacked bar represents the proportion of participants who stated, "*Would buy*" (dark grey), "*Maybe would buy*" (light grey) or "*Would not buy*" (white).

Participants were asked about their purchase intention after tasting the samples. Results from this question paralleled to that of liking after tasting. Purchase intention was lower for Green samples, with no relevant differences depending to the packaging. Twenty per cent of participants declared be willing to buy them and other 20 % declared that they may buy or not. Purchase intention was markedly higher for Mature samples and for the commercial sample. More than 40% of participants were willing to buy these samples and 20-30% declared that they may buy or not.

3.4.2 Samples and Ideal product description

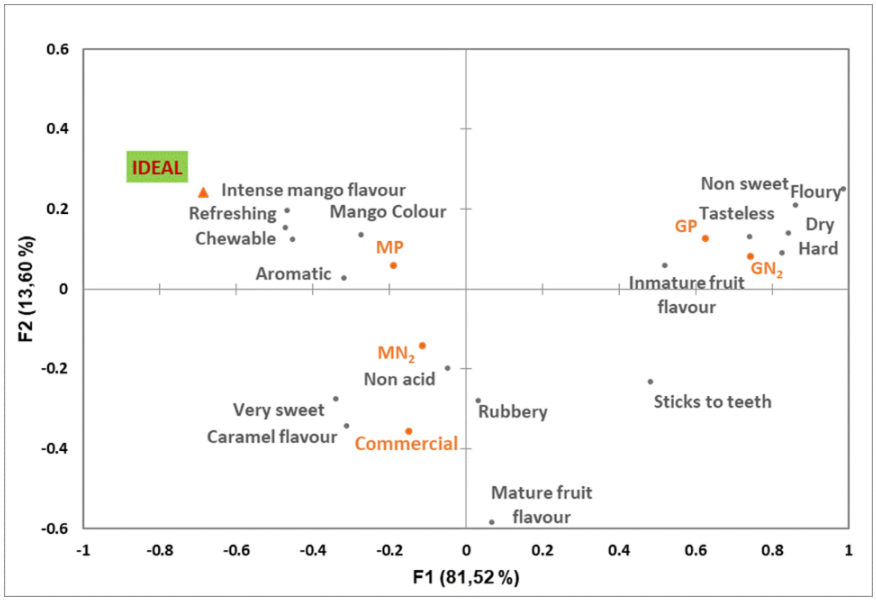


Figure 4. Correspondence-analysis (CA) map generated from the CATA data, displaying the Ideal dehydrated mango (green) together with the five samples evaluated: Commercial, GN_2 (Green + MAP N_2), GP (Green + MAP P), MN_2 (Mature + MAP N_2) and MP (Mature + MAP P).

Figure 4 shows the correspondence analysis performed with the CATA question data including the Ideal dried mango. As previously explained in the material and methods section, only those attributes that contributed significantly to differentiate samples are include in the plot, in which the first and second dimensions explained close to 94% of the variability. The CA plot shows the Ideal dried mango allocated to the left and upper part of the space. Among the evaluated samples, MP was the one that came close to the Ideal. This closeness in allocation meant that consumers found in this sample most of the attributes that they used to describe their Ideal: *Intense mango flavour*, *easy chew*, *refreshing flavour*, *mango colour* and *aromatic* [80]. If we pay attention to the first dimension, which explained most variability, we observed that MN_2 and the commercial samples are relatively close to the MP, that indicating that to some extent the three samples share characteristics among each other. However, participants described the MN_2 and the commercial samples as *sweeter*, *less acid*, *rubbery* and having a more *intense caramel flavour* than the MP sample.

Mature fruit flavour was mainly perceived in the commercial samples. Both Green samples (GP and GN_2) were allocated in the opposite side of the plot, with positive values in the first dimension. These samples had characteristics very different to the consumers' Ideal. Thus, Green samples were described with terms such as *dry*, *floury*, *tasteless*, *hard*, *non-sweet* and *immature fruit flavour*. Moreover, consumers detected that these two samples were the ones that stuck more to teeth.

3.4.3 Identification of key attributes. 'Must-have' and 'must-not-have' attributes

To investigate the relation between acceptability and the sensory description of the samples, a penalty analysis was applied to CATA data to determine the changes in the global liking associated with a deviation of each product from the ideal for each attribute.

A first analysis based on incongruence in which the attribute is missing in the real but not the ideal product allows identifying the 'must have' attributes (Figure A). Thus, 'medium mango flavour', 'aromatic', 'mango colour', 'chewable', 'refreshing' and 'slightly sweet' were attributes that a high percentage of participants applied to their ideal dehydrated mango, and when detected in the samples led to a significant increase in acceptance [81]. In parallel, figure B shows highlighted in blond font those attributes not desirable in the ideal product that have been detected in samples by a high percentage of participants and are associated to low coordinate on the Y axis, i.e., the "must not have" attributes: 'slight mango flavour', 'sticks to teeth', 'hard' and 'dry'.

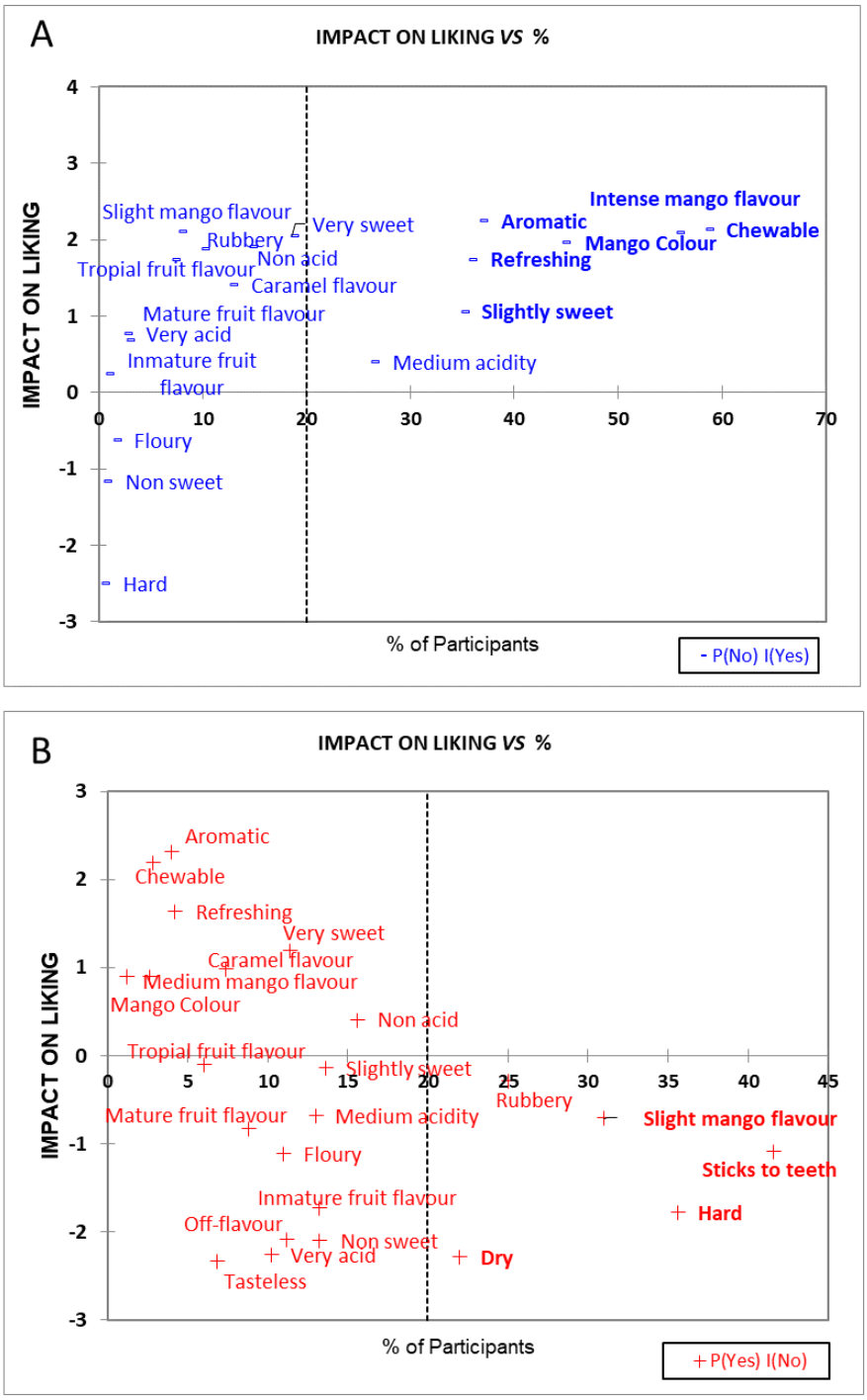


Figure 5. (A) Results of the penalty analysis based on CATA, in which the attribute is selected for the ideal product but missing in the real products. (B) Results of the penalty analysis based on CATA, in which the attribute is not important for the ideal but endorsed for the real products. Attributes highlighted in bold correspond to those in which more than 20% of the consumers considered that it deviated from the ideal and caused a significant decrease in liking according to Kruskal–Wallis test for a 95% confidence level.

A significant drop in average liking was detected when these attributes are present in samples [81]. It is important to comment that 'slight mango flavour' was the 'must not have' attribute with the lowest impact on liking (less than 1 point of drop). In fact, data shows that mango flavour is a desirable attribute, with 'medium mango flavour' identified as a must have attribute (figure 5A) and 'intense mango flavour' as an attribute that describes the ideal

product (figure 5B). Therefore, it is important to make it clear that consumers want to perceive mango flavour, and the more intense the attribute the better.

4 Conclusion

The findings of this research highlight how preservation treatments, mango ripeness, and dehydration techniques significantly influence product quality and consumer perception. Mango ripeness proved to be a key factor in purchase preference, with ripe samples receiving higher sensory evaluations compared to unripe ones. This emphasizes the importance of tailoring preservation practices based on the fruit's ripeness stage to optimize product acceptability. In terms of preservation treatments, modified atmosphere storage with nitrogen (GN_2) showed a positive effect on colour preservation, ensuring visual stability, a crucial factor for consumers. However, passive atmosphere packaging (MAP P) was more effective in maintaining firmness and colour, particularly in ripe samples, compared to GN_2. These results underline the importance of selecting conservation techniques that balance both visual and structural attributes of the product. From a nutritional perspective, parameters such as protein content, soluble solids, and total sugars remained relatively stable during the analysis period. However, vitamins exhibited significant variations across treatments, highlighting the impact of storage conditions. Specifically, the GN_2 treatment resulted in lower levels of vitamins B3 and B1 compared to other methods, indicating the need to balance conservation effectiveness with the preservation of essential micronutrients. Finally, the comparative analysis with commercial samples revealed substantial differences, particularly in polyphenol levels. These discrepancies were likely due to extreme thermal treatments and the use of preservatives like sulphites. This finding underscores the importance of carefully managing dehydration conditions to better preserve the nutritional and organoleptic qualities of the product while avoiding chemical preservatives.

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CHAPTER 6

Improvement of Antioxidant Activity and Sensory Properties of Functional Cookies by Fortification with Ultrasound-Assisted Hot-Air-Drying Blackberry Powders

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Abstract

In response to the global challenge of food wastage and high perishability of blackberries, this study evaluated the use of ultrasound-assisted hot air drying (US-HAD) to convert downgraded blackberries into powders, comparing it with traditional hot air drying (HAD). US-HAD reduced the drying time and achieved a final moisture content of 12%. Physicochemical analyses (colorimetry, total soluble solids, titratable acidity, and total phenolic content) were conducted on fresh fruit, powders, and fortified cookies. US-HAD cookies exhibited promising antioxidant activity, with ABTS values ranging from 8.049 to 8.536 mmol TEAC/100 g and DPPH values from 8.792 to 9.232 mmol TEAC/100 g, significantly higher than control cookies. The TPC was 13.033 mg GAE/g in HAD cookies and 13.882 mg GAE/g in US-HAD cookies. UHPLC-ESI-MS analysis showed an increase in phenolic compounds content in fortified cookies compared to the control. Sensory analysis highlighted a superior blackberry flavour and overall acceptability in US-HAD cookies, with statistical analysis confirming their superior nutritional and sensory qualities. Integrating US-HAD blackberry powder into cookies helps reduce food waste and enhances the nutritional profiles of baked goods, offering functional foods with health benefits. This work provides a scientific basis for developing enriched functional cookies, offering a healthy and sustainable alternative for utilising damaged fruits.

Keywords: ultrasound-assisted hot air drying; blackberry powder; physicochemical properties; antioxidant properties; polyphenols; sensory evaluation; fortified foods

1. Introduction

Over the past decade, consumer interest in foods rich in beneficial nutrients and bioactive compounds that maintain integrity and freshness has grown significantly [1]. Numerous studies [2–5] have demonstrated the health benefits of diets rich in fruits and vegetables due to the presence of phenolic compounds, which are associated with cancer prevention, cardiovascular health, and immune system enhancement. The blackberry (*Rubus* spp.-Fam. *Rosaceae*) is of Asian origin and is cultivated in Europe, North America, and the temperate regions of Brazil [6]. This fruit is mainly found in the temperate and cool–temperate areas of the Northern Hemisphere but is also found in some warm and subtropical areas [7]. Ripe blackberries have a dark red to dark blue colour with a smooth, shiny skin [8]. They are climacteric, seasonal, and highly perishable fruits [9]. Small fruit crops, such as blackberries, are known for their antioxidant potential due to the presence of various naturally occurring active compounds. These include polyphenols, isoprenoids, and organic sulphur compounds [10–12]. In red berries, the most abundant compounds are phenolic acids, tannins, and flavonoids, particularly anthocyanins, which give the fruits a sour taste and attractive colour. The presence of these compounds, especially anthocyanins, makes these fruits useful in preventing diseases such as diabetes, cancer, and degenerative conditions [13]. There are quality standards for blackberries in the food industry that concern size, consistency, flavour, and nutritional content. Quality criteria include physical and chemical parameters such as a uniform colour, the absence of physical damage or contamination, and proper ripeness. The sugar content in blackberries can vary from 8 to 12 g per 100 g of fresh fruit, while the total acid content usually ranges from 0.6 to 1.5 g per 100 g, depending on the variety and growing conditions [14]. Blackberries are usually consumed fresh but can be processed and marketed frozen or as juice concentrate. In industry, blackberries that do not meet fresh market standards are used to produce food supplements, juices, yoghurt, ice cream, jellies, and other candies [15,16]. Additionally, they could be processed into semi-finished foods, such as vegetable powders, and then used in other preparations, allowing the creation of ‘fortified foods’, with bioactive compounds that offer additional health benefits [17]. This approach opens new possibilities for the use of small fruits in the food industry. For example, Pereira et al. (2019) [18] reported a study on the use of freeze-dried blackberry flour and whole blackberries for the creation of cookies while Rózyło et al. (2019) [19] reported the use of freeze-dried blackberry powder in the formulation of gluten-free crispy bread. However, no

study has focused on the use of blackberry powder from downgraded fruits, obtained by hot-air and ultrasound-assisted drying, as ingredients for the preparation of cookies. Globalization has led to overproduction and overdistribution, causing significant food waste [20]. Addressing this challenge requires the development of food technologies that preserve both the quality and quantity of the product, ensuring efficient resource utilization. Despite their potential commercial, blackberries' high perishability and rapid post-harvest respiration significantly compromise their nutritional and microbiological quality, limiting their shelf life and resulting in the downgrading of the fruit in world markets, leading to increased food waste. It is estimated that around 931 million tons of food are wasted globally each year, and blackberries, being highly perishable, contribute significantly to this problem. This waste not only represents a loss of valuable resources but also incurs considerable economic costs. The economic burden of food waste globally amounts to approximately USD 936 billion annually [21]. Therefore, it is crucial to develop new products using downgraded blackberry fruits to mitigate this phenomenon. The integration of vegetable powders into cookies represents an innovative and advantageous strategy for the food industry [22]. Cookies are a highly favoured treat, but their high fat and sugar content can pose significant health risks. With the global rise in obesity, diabetes, and cardiovascular diseases, there is increasing attention towards reducing the intake of unhealthy fats and sugars in the diet. The use of such powders allows for a significant reduction in fat and sugar content in the final products, thereby addressing the growing consumer concern for health and well-being [23]. Additionally, the use of natural ingredients with low sugar and fat content can enhance a product's image and positively respond to market trends toward more sustainable and nutritious foods. Among the technologies for reusing products that are not suitable for the fresh market or waste products, convective hot air drying is suitable for obtaining vegetable powders that can be used as supplements or ingredients to enrich the diet with nutrients. Drying is an ancient method that reduces water content to a final concentration that ensures microbial stability and preservation (10–14% relative humidity) [24]. Compared to other methods, hot air drying produces a qualitatively superior product using low temperatures and short treatment times, as demonstrated by Tinebra et al. (2022) in a study conducted on some Loquat varieties [25]. This technology can improve processing efficiency, preserve nutritional quality, and reduce the environmental impact of processing operations compared to other techniques such as freeze-drying, which requires much higher energy expenditure [26]. During the drying of fruits and vegetables, numerous physicochemical changes occur, such as colour alterations due to enzymatic or non-enzymatic browning reactions, texture

changes, shrinkage, and the loss or degradation of nutritional compounds (e.g., ascorbic acid, carotenoids, phenolic compounds) [27]. Si et al. (2016) [28] demonstrated that the use of low temperature freeze-drying on raspberries causes a more significant degradation of polyphenols compared to hot air drying (HAD) for 9 h. This result is understandable, considering the long drying time of 36 h required by freeze-drying. Therefore, selecting the appropriate time/temperature parameters or applying pre-treatments such as ultrasound is crucial to minimize the loss of these compounds [29]. For example, studies on the hot air drying of apples have shown that using low temperatures and short processing times can preserve vitamin C content and improve the sensory quality of the final product [30]. Kalra and Bhardwaj (2012) [31] have demonstrated that convective drying is faster and more efficient than solar drying for fruits such as mangoes, papayas, and apricots. Some technologies can be combined with drying to facilitate water diffusion and reduce the processing time. Ultrasound pre-treatment has been shown to cause the extreme expansion and contraction of the fruit structure, making it resemble a 'sponge' [32]. This spongy structure accelerates the removal and evaporation of moisture compared to the control condition, thus preserving the bioactive compounds and nutritional characteristics of the fruit [33]. Several studies [34–37] have demonstrated that ultrasound improves drying efficiency by shortening processing times. When ultrasound interacts with vegetal material, it induces molecular and microscopic vibrations within [38], leading to the formation of micro-cavitations—tiny air bubbles within the matrix. These bubbles subsequently collapse, causing microscopic explosions that alter the structure and composition of the plant material [39]. A reduced drying time offers significant benefits including improved energy efficiency and a reduced environmental impact. Ultrasound pre-treatment has been applied to strawberries, significantly improving anthocyanin retention and antioxidant capacity [40]. These examples show how drying technologies and pre-treatments can be adapted to different types of fruits while maintaining their nutritional and functional properties.

The aim of this study was to evaluate the use of hot air drying assisted by ultrasound to convert damaged and non-marketable blackberries into vegetable powders for use as additives in cookie production. The study focused on assessing the impact of the drying process on the quality of the powders and their effects on the sensory characteristics of the resulting cookies.

2. Materials and Methods

2.1 Plant Material

Blackberries fruits (4 kg) not suitable for the fresh market from the farm 'A piccoli frutti' in Marsala (coordinates 38◦6′44.859" N, 13◦20′47.063" E), Trapani (Italy) were used for the experimentation. The fruits were transported to the post-harvest laboratory of the Agricultural Food and Forestry Sciences Department of the University of Palermo. The following parameters were measured on 50 fruits (300 g) in a single repetition for fruit, representative of the sample: weight (g), colour (RGB colour model), soluble solids content (CSS-◦Brix), titratable acidity (AT-g/L−1 citric acid), and dry residue (%RS). These parameters were calculated to characterize the fruits before they were processed.

2.2 Chemicals and Reagents

Methanol, sodium hydroxide, hydrochloric acid, sodium carbonate, sodium bicarbonate, gallic acid, Folin–Ciocalteu's phenol reagent, and DPPH (2,2-diphenyl-1-picrylhydrazyl) were purchased from Sigma-Aldrich (Sigma-Aldrich, St. Louis, MO, USA). ABTS (2,2′ azino-bis(3-ethylbenzothiazoline-6-sulfonic-acid), potassium persulphate, and Trolox (6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid) were obtained from Fluka (Buchs, Switzerland). N-hexane, ethyl acetate, potassium hydroxide, and 0.45 µm PTFE syringe filter was purchased from Thermo Fisher Scientific Inc. (Thermo Fisher Scientific, Waltham, MA, USA). Ethyl myristate, quercetin, rutin, kaempferol, naringenin, nicotinic acid, cyanidin 3,5-diglucoside, caffeic acid, ferulic acid, *p*-coumaric acid, m- coumaric acid, and chlorogenic acid were purchased from Sigma-Aldrich (St. Louis, MO, USA). ABTS (2,2′ azino-bis(3-ethylbenzothiazoline-6-sulfonic-acid), potassium persulphate, and Trolox (6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid) were obtained from Fluka (Buchs, Switzerland. HPLC grade water was obtained by purifying double distilled water in a Milli-Q Gradient A10 system (Millipore, Bedford, MA, USA).

2.3 Experimental Design

2.3.1 Drying Design

To obtain a dried product with a residual moisture content of 12% that ensures micro-biological stability [24,41,42], two different treatments were tested:

- HAD (hot air drying) 75 ◦C;
- US-HAD (ultrasound hot air drying) 75 ◦C.

Before drying, the fruits were washed and disinfected in an aqueous solution containing distilled water at 25±1 ◦C and 200 µL/L−1 NaClO (5–6.5% NaClO solution, CHEMLAB)

for 10 min. The samples were then divided into two lots (each 2 kg) that were homogeneous in terms of size, colour, and weight. The first lot of blackberries, after washing, was placed in a dryer as described by Roppolo et al. (2023) [43]. The second lot underwent pre-treatment in an ultrasound bath (US) (DU-32, ARGOlab, Modena, Italy) by dipping and then drying. The US treatment protocol involved heating the water to a temperature of 30 ◦C and then dipping the fruits in the ultrasound bath for 30 min at 22 kHz and 70W. The drying temperature was chosen based on preliminary tests and literature concerning the drying of similar fruits [28,44–46]. During the drying process, the weight of the trays was measured every two hours to ensure the process stopped when a residual moisture content of 12% was achieved, preventing the loss of bioactive compounds. After drying, the weight was measured to determine the dry residue (%RS) and the colour to determine the degree of browning of the fruit.

Finally, the two lots were ground in an ultracentrifugal mill (Fritsch, Pulverisette 14, Lainate, Italy) at 10,000 rpm for 10 s to obtain vegetable powders with a suitable grain size (800±50 µm), labelled as follows:

- HAD-BP (blackberry powder)
- US-HAD-BP (blackberry powder)

The powders were colour-detected and stored in hermetically sealed polyamide/polyethylene (PA/PE) bags at room temperature (20±1 °C).

2.3.2 Cookie Design

Vegetable powders, HAD-BP and US-HAD-BP were used as ingredients for shortbread cookies. The preparation protocol involved adding the powders at a rate of 10% to "00" type flour (CTRL-P) [47,48]. The flours were mixed as follows:

- HAD-BP 10% + "00" flour 90%;
- US-HAD-BP 10% + "00" flour 90%;
- CTRL-P "00" flour 100%.

The dough was prepared by adding eggs, yeast, and vegetable oil and mixing all ingredients for 3 min with a planetary mixer (model XBM10S; Electrolux Professional SpA, Pordenone, Italy) at Speed 4. The dough was stored at 4 ◦C for 3 h and 30 min before tempering at room temperature (20±1 °C). A stainless-steel rolling pin was used to roll the dough to a target thickness (3±1 mm). The dough was then cut into 3.00±1.00 mm square shapes, placed on a baking tray, and baked in a laboratory oven (Compact Combi, Electrolux, Pordenone, Italia) at 165 ◦C and a time of 20 min. The resulting cookies were divided as follows:

- HAD-Cookies (HAD-BP 10% + “00” flour 90%):
- US-HAD-Cookies (US-HAD-BP 10% + “00” flour 90%):
- CTRL-C (“00” flour 100%).

The cookies were first left to cool at room temperature (20±1 °C) for 2 h and then packed in hermetically sealed polyamide/polyethylene (PA/PE) bags under passive modified atmosphere conditions (21% O_2 and 0.04% CO_2) at room temperature (20±1 °C) for 10 days.

2.4 Chemical-Physical Analysis

The weight of the fruits (g), fresh and dried, was measured with a precision electronic balance (Gibertini EU-C 2002 RS, Novate Milanese, Italy) to assess the percentage of dry residue (%RS), evaluated using Formula (1) [43]:

$$\%RS = \left(\frac{c-a}{b-a}\right) \times 100 \quad (1)$$

Here,

a. = weight of empty tray;

b. = weight of the tray with the product before drying;

c. = weight of tray with product after drying.

Colours of fresh fruits, vegetable powders, and cookies were evaluated using a digital colourimeter (Minolta, mod. CR-300; Osaka, Japan) according to the CIELab colourimetric system, which identifies the colour of the fruit with three different coordinates: L* (brightness; L* = 0 for black and L* = 100 for white), a* (green/red colour index; a* = −100 for green and a* = +100 for red), and b* (blue/yellow colour index; b* = −100 for blue and b* = +100 for yellow). Values were converted to RGB format using ‘e-paint.co.uk Convert Lab’ software [49]. The colour differences (ΔE) between the fresh fruit and the obtained powder and between the cookies were measured according to Equation (2) [50]:

$$\Delta E = \sqrt{(L^* - L_0^*)^2 + (a^* - a_0^*)^2 + (b^* - b_0^*)^2} \quad (2)$$

Here, $L0*$, $a0*$, and $b0*$ represent the values of the colour parameters measured after drying. The total soluble solids content (TSSC) was measured on the fresh fruit with a digital refractometer (Atago, Tokyo, Japan) and expressed in ◦Brix while the titratable acidity (TA) was measured with a pH titrator (Titromatic 1S, Crison, Barcelona, Spain) and expressed in grams of citric acid per litre of fruit juice (g citric acid/L−1).

2.5 Evaluation of Total Phenolic Content (TPC)

Total phenolic content (TPC) was determined using the optimized Folin–Ciocâlteu colourimetric method according to previous research [47,51] with slight modifications. In particular, 0.5 g of each sample of powder was mixed with 10 mL of methanol/water solution (80:20 *v/v*), sonicated for 50 min, and filtered through Whatman 0.45 µm PTFE filters. An aliquot of the filtrate (0.110 mL), 125 µL of a 7% Na_2CO_3 solution, and 625 µL of Folin–Ciocâlteu reagent (1:5) were incubated in the dark at 25 ◦C for 50 min. The Abs were evaluated at 765 nm using a UV-Vis spectrophotometer (Varian Cary 50, Agilent, Santa Clara, CA, USA). Methanol was used as the blank and gallic acid was used for calibration of the standard curve (0.001 to 0.30 mg/mL). TPC was expressed as mg gallic acid equivalents per g (mg GAE g−1) of the sample. Data were analysed in triplicate and reported as means ± SEMs.

2.6 Radical Scavenging Properties Evaluation, DPPH and ABTS Assay

The measurement of powder sample antiradical activity follows procedures previously described [52,53]. DPPH and ABTS assays allow us to determine the antioxidant power through the reaction of the sample with a solution of DPPH [2,2-diphenyl-1- picrylhydrazyl] and ABTS [2,2′-azino-bis (3-ethylbenzothiazolino-6-Sulphonic acid] that causes a discolouration of the solution proportional to the antioxidant charge present in the sample. Two grams of sample were extracted using 10 mL of methanol, sonicated for 60 min, and filtered through Whatman 0.45 µm PTFE filters. The filtrate (120 µL) was mixed with 4 mL of DPPH (60 µm) and incubated in the dark at 25 ◦C for 30 min. Methanol was used as the blank. Scavenging activity was measured at 515 nm using a UV-Vis spectrophotometer (Varian Cary® 50). A calibration curve using Trolox at increasing concentrations [2.5 µm–25 µm] was constructed. The results were reported as Trolox equivalent antioxidant activity (TEAC) and expressed as mmol Trolox equivalents (TEs)/100 g of sample. Five mL of MeOH was added to 2 g of each sample, sonicated, and filtered through Whatman 0.45 µm PTFE filters. The absorbance was read 10 min after the addition of 4 mL of diluted ABTS+ to 120 µL of sample. The decrease in absorbance caused by antioxidants, recorded at 734 nm against ethanol, reflected the ABTS+ free radical scavenging capacity. A calibration curve using Trolox in a concentration range of 2.5 µm–25 µm was constructed. Obtained values were reported as Trolox equivalent antioxidant activity (TEAC) and expressed as mmol Trolox equivalents (TEs) per 100 g of sample. Data were analysed in triplicate and reported as means±SEMs.

2.7 Polyphenols Extraction

Phenolic compound phenolics were extracted and analysed using previously modified methods [54,55]. Five grams of sample were homogenized for 45 min in 20 mL of 80% methanol solution using an ultrasound bath. The samples were centrifuged at 5000× *g* for 15 min and the supernatant was recovered. The pellet was re-extracting four times (repeating the protocol described above) and the supernatant was collected and evaporated using a rotary evaporator under vacuum at 45 ◦C. The residue was redissolved in 1 mL of methanol. This solution, containing free phenolic compounds, was filtered through a 0.22 µm PTFE syringe filter into glass vials before UHPLC-ESI-HRMS analysis. Data were analysed in triplicate and reported as means ± SEMs.

2.8 UHPLC-ESI-HRMS Analysis

All the extracts were characterized by UHPLC-ESI-HRMS as reported by Frisina et al. (2023) [56] with slight modifications. The instrument set up consisted of a Dionex Ultimate 3000 RS (Thermo Scientific—Rodano, MI, Italy), interfaced with a high-resolution Q-Exactive orbitrap mass spectrometer (Thermo Scientific, Rodano, MI, Italy) with electrospray ionization source, operating in negative mode. Chromatographic separation was performed with an Hypersil Gold C18 column (100 mm × 2.1 mm, 1.9 µm particle size) from Thermo Scientific, maintained at a temperature of 30 ◦C and a flow rate of 300 µL min−1. The chromatographic column was equilibrated in 98% solvent A (ultrapure water containing 0.1% of formic acid) and 2% solvent B (methanol). The concentration of solvent B was linearly increased from 2% to 23% in 6 min, remaining isocratic for 5 min, then linearly increased from 23% to 50% in 7 min and from 50% to 98% in 5 min, remaining isocratic for 6 min, and finally returned to 2% in 6 min, remaining isocratic for 3 min. Volume of injected sample was 5 µL. The total run time, including column wash and equilibration, was 38 min. Heated electrospray ionization (HESI) was selected in negative polarity, with the following operating conditions: 70,000 resolving power (defined as FWHM at *m/z* 200), IT 100 ms, ACG target = 1 × 106, and scan range (100–900 *m/z*). MS/MS analyses were performed according to the following operating conditions: resolution: 35,000, AGC target = 1 × 105, maximum IT 200 ms, and collision energy (stepped NCE): 20, 30, 40. The quadrupole isolation window was set to 2.0 *m/z*. High purity nitrogen was used as the sheath gas (30 arb units) and auxiliary gas (10 arb units). The instrument was calibrated before each analysis using the calibration solution supplied by Thermo Fisher Scientific. Compounds were characterized according to the corresponding HRMS spectra, accurate masses, characteristic fragmentations, and retention times and

quantified using calibration curves of analytical reference standards. Standard solutions were daily prepared in methanol. Xcalibur software (version 4.1) was used for instrument control, data acquisition, and data analysis. Individual concentrations of the considered molecules were derived by the external calibration curves of the respective commercial analytical standards. In particular, the concentrations of protocatechuic acid (*m/z* 153.0183), benzoic acid (*m/z* 121.0284), and caffeic acid (*m/z* 179.0342) were obtained with respect to a calibration curve of caffeic acid (r2 = 0.9999) in a range between 0.1 and 5 mg/L. A preset kaempferol standard calibration curve (r2 = 0.9978) in the concentration range of 0.1–10 mg/L. was used to determine the content of dihydrokaempferol (*m/z* 287.2229). Ellagic acid (*m/z* 300.9991) and quercetin-3-O- glucoside (*m/z* 463.0884) were quantified using the calibration curve of the corresponding reference standards in ranges of 0.1–10 mg/L and 0.5–10 mg/L, respectively.

2.9 Sensory Analysis

The sensory evaluation was conducted by a panel of 14 semi-qualified judges based on 19 descriptors: surface colour (SC), internal colour (IC), colour homogeneity (CO), cookie odour (BO), blackberry odour (MO), burnt odour (BRO), unpleasant odour (UO), hardness (H), softness (S), crunchiness ©, chewiness (CH), cookie taste (BT), blackberry taste (MT), burnt taste (BRT), unpleasant taste (UT), sweetness (SW), bitterness (BI), sourness (SN), and overall acceptability (OA). The evaluation was conducted from 10:00 to 12:00 in a room under white lights. Each panel member received, in random order, a control sample (CTRL-C) and the two samples with the added powders (HAD-Cookies and US-HAD-Cookies) (Figure 1). Between each sample, water was provided for mouth rinsing. The judges rated the intensity of each descriptor on a hedonic scale, assigning score from 1 to 9, representing different levels of intensity of the quality descriptors: 1—no sensation, 2—barely recognizable, 3—very weak, 4—weak, 5—slight, 6—moderate, 7—intense, 8—very intense, and 9—extremely intense.

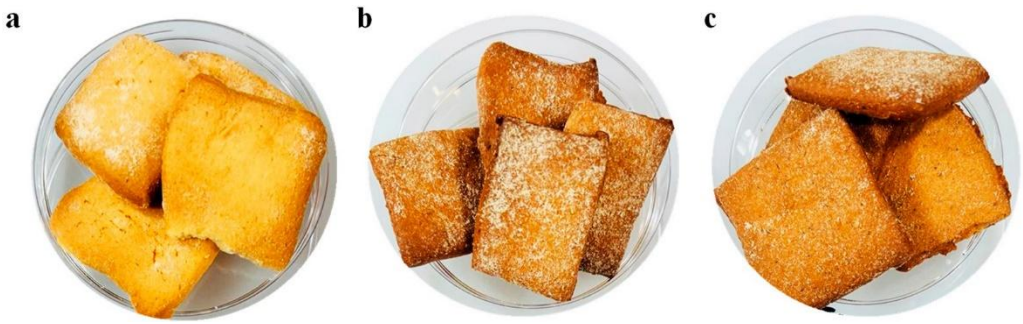

Figure 1. Cookies samples analysed in the sensory panel: (**a**) CTRL-C (control); (**b**) HAD-Cookies (hot air drying); (**c**) US-HAD-Cookies (ultrasound-assisted hot air drying).

2.10 Statistical Analysis

Results are expressed as means ± SEMs of *n* separate experiments conducted in triplicates. Statistical comparisons were performed by one way analysis of variance (ANOVA) followed by Tukey's correction for multiple comparisons. The data were statistically processed using XLStat software version 16.0 for Microsoft Excel (Addinsoft, New York, NY, USA). In all cases, significance was accepted if the null hypothesis was rejected at the $p < 0.05$ level.

3. Results

3.1 Plant Material

Before the drying process, a physicochemical characterization was conducted on a sample of 50 fresh fruits (Table 1). A TSSC of 8.36±0.08 ◦Brix suggests that blackberries have a satisfactory level of soluble sugars, ensuring adequate sweetness. Titratable acidity measures the content of organic acids in fruit, contributing to flavour and shelf life. A TA of 0.29±0.07 g/L indicates that blackberries have a good balance between sweetness and acidity, ensuring a well-rounded flavour profile that enhances overall sweetness. Values of L*, a*, and b* and RGB collectively indicate that blackberries have a dark colour, typical of ripe, high-quality fruit.

Table 1. Quality characteristics of fresh blackberry fruits: TSSC (total soluble solids content, ◦Brix); TA (titratable acidity, g/L−1 citric acid); L* (brightness); a* (red); b* (yellow); RGB (conversion of CIELab* values). Values are represented as means ± standard deviations of the data obtained.

	TSSC (°Brix)	TA (g citric a./L^{-1})	L*	a*	b*	RGB
F. Blackberries	8.36±0.08	0.29±0.07	18.72±0.74	1.46±0.27	1.95±0.72	

3.2 Drying Process

Table 2 presents the results of drying experiments conducted on blackberries using different treatment methods at 75 ◦C. The values represent the percentages of dry residue (%RS) obtained after specified durations, illustrating the efficiency of each method in preserving fruit integrity.

Table 2. Results in terms of drying time and dry residue of blackberries. ◦C represents the treatment temperature, *h* represents the treatment time expressed in hours, and %RS represents the percentage of dry residue. The values of %RS are expressed as the means ± standard deviations of the results obtained. Different letters represent statistically significant differences ($p < 0.05$).

Samples	h	°C	%RS
HAD	7	75	11.72±0.74^{a}
US/HAD	6	75	10.37±1.67^{b}

The results show that HAD treatment at 75 ◦C produced a dry residue (%RS) of 11.72±0.74 after 7 h while the US-HAD treatment, at the same temperature, resulted in a dry residue (%RS) of 10.37±1.67 after 6 h.

3.3 Chemical-Physical Analysis

An important quality parameter for consumers is the visual appearance of the fruit. Table 3 illustrates the colour differences (ΔE) in blackberry powders obtained from both treatments compared to fresh blackberries.

Table 3. Colour differences (Δ*E*) of blackberry powders measured on the fresh samples after drying process. RGB column represents the conversion of CIELab* values. Values are represented as means ± standard deviations of the data obtained. Different letters represent statistically significant differences ($p < 0.05$).

Samples	Δ*E*	RGB
F. Blackberries		
HAD-BP	28.27±0.78[a]	
US-HAD-BP	26.70±0.44[b]	

The results show that the blackberry powder samples obtained through the two drying treatments exhibited significant colour differences compared to fresh blackberries, as indicated by the ΔE values. The powder from hot air drying (HAD-BP) had a ΔE value of 28.27±0.78, indicating a substantial colour difference from the fresh blackberries. Conversely, the powder from ultrasound-assisted hot air drying (US-HAD-BP) had a ΔE value of 26.70±0.44. Although this also signified a significant colour difference from the fresh blackberries, it was less pronounced than that observed in the HAD-BP sample. It was also visually evident that US-HAD-BP caused significantly less colour variation than HAD-BP, as shown in Figure 2.

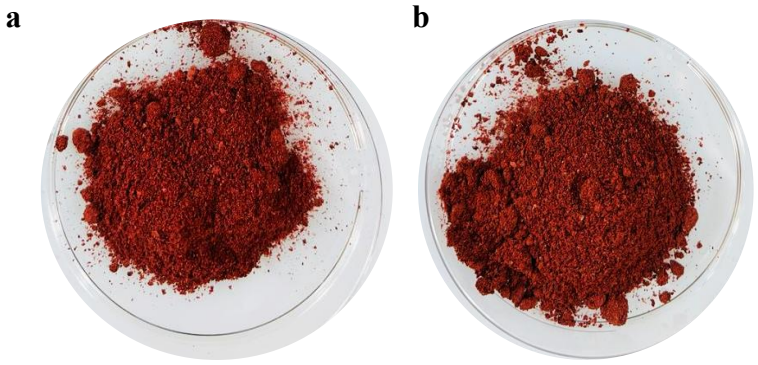

Figure 2. Visual characteristics of the blackberry vegetable powders analysed. a) Blackberry powder HAD-BP (hot air drying); b) Blackberry powder US/HAD-BP (ultrasound-assisted hot air drying).

Figure 3 shows the values of the colourimetric parameters L* (brightness), a* (red/green), and b* (yellow/blue) for blackberry powders obtained by two drying methods (HAD and US-HAD). The L* values for the HAD-BP and US-HAD-BP powders were 25.80±1.04 and 24.94±0.36, respectively, indicating a significantly lower brightness, with reference to Table

1, but closer to the values of the fresh samples for US-HAD treatment. The parameter a* showed a more marked difference compared to the fresh samples (Table 1), with values of 24.99±0.38 for HAD-BP and 23.25±0.35 for US-HAD-BP. The statistical analysis performed also showed significant differences between the two treatments. For the parameter b*, the values were similar: 15.87±0.82 for HAD-BP and 16.05±0.26 for US-HAD-BP, but significantly different from the fresh samples (1.94).

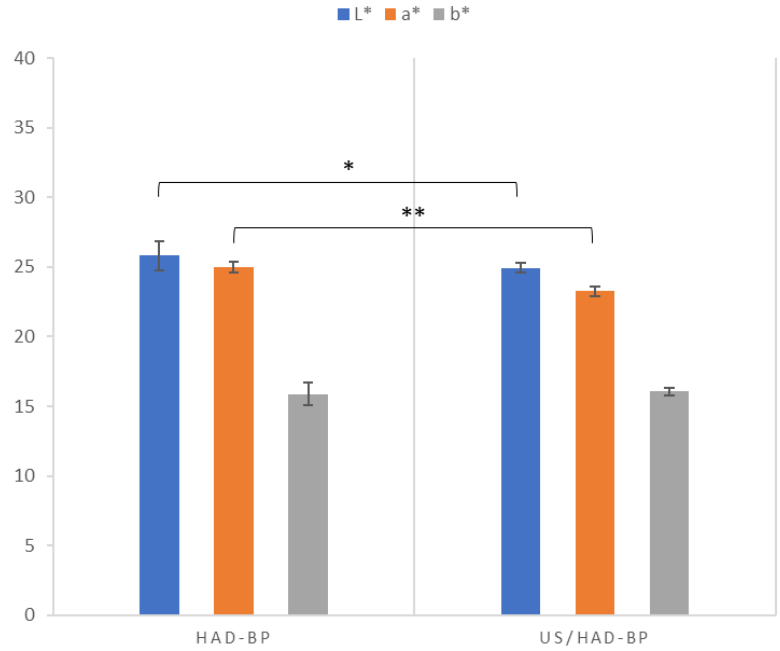

Figure 3. CIELab* values of blackberry powders (HAD-BP and US/HAD-BP) after drying and mill. Results indicate the mean values ± standard deviation. Asterisks indicate statistically significant differences between treatments with * for $p<0.05$, ** for $p<0.01$, *** for $p<0.001$; no asterisk indicates a non-significant difference.

Table 4 displays the colour difference (ΔE) values observed in cookies containing blackberry powder, comparing two different drying treatments. The ΔE values quantify the perceptible differences in colour between the control cookies and those processed with the HAD and US-HAD methods, providing insights into the impact of drying techniques on final product appearance.

Table 4. Colour difference (ΔE) of blackberry cookies measured on the samples after baking. RGB represents the conversion of CIELab* values. Values are represented as mean ± standard deviation of the data obtained. Different letters represent statistically significant differences ($p<0.05$).

Samples	ΔE	RGB
CTRL-C		
HAD-Cookies	22.55±1.19^{a}	
US/HAD-Cookies	31.06±1.32^{b}	

Table 4 presents the colour difference values (ΔE) for three samples of cookies. The HAD-Cookies sample exhibited a colour difference of 22.55±1.19 compared to the control cookies whereas the US-HAD-Cookies sample showed a larger difference of 31.06±1.32. In

addition, figure 4 illustrates the colourimetric parameters L*, a*, and b* of cookies containing 10% blackberry powder. Both treatments showed significant differences in CIELab values compared to the control (CTRL-C). The US-HAD-Cookies sample exhibited a lower L* value (40.12±0.86) than the HAD-Cookies sample (48.23±1.47) when compared to CTRL-C (69.30±1.40). Specifically, a 30% loss was observed for HAD-Cookies and a 42% loss for US-HAD-Cookies, highlighting a significant difference between the two treatments. The a* parameter was higher in US-HAD-Cookies (15.13±0.61) than in HAD-Cookies (14.14±0.92), but both were lower than CTRL-C, with respective losses of 25% and 30%. Similarly, the b* parameter was higher in HAD-Cookies (26.85±1.26) than in US-HAD cookies (24.46±2.22), with a significant difference between treatments and compared to the control. In fact, both HAD-Cookies and US-HAD-Cookies showed reductions of 32% and 38%, respectively, compared to CTRL-C.

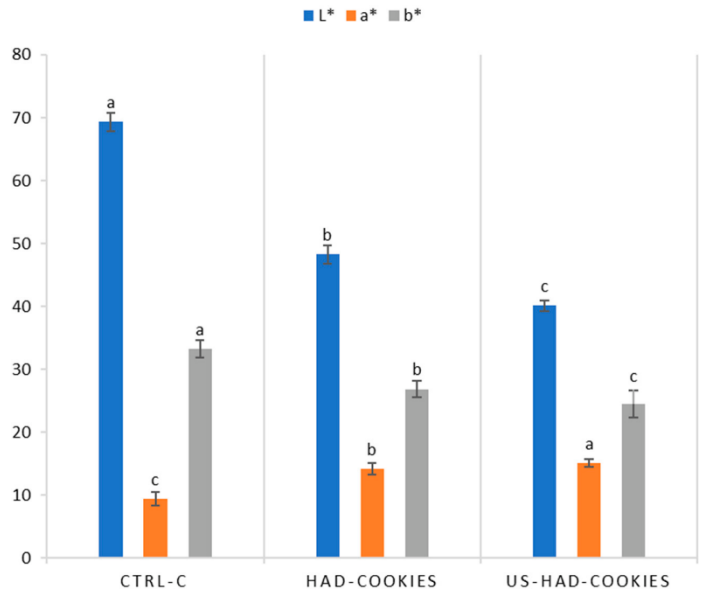

Figure 4. CIELab* values of blackberry cookies (HAD-Cookies and US-HAD-Cookies) after baking. Results indicate the mean values ± standard deviations. Values in the histograms followed by a different letters (a, b,c) are significantly different ($p < 0.05$).

3.4 Evaluation of Total Phenolic Content (TPC) and Radical Scavenging Properties

The total phenolic content (TPC) and radical scavenging activity values of HAD-BP, US-HAD-BP, and "00" control flour (CTRL-P) and of their 10% fortified cookies (HAD-Cookies, US-HAD-Cookies) and control cookies (CTRL-C) were measured. The hot-air-dried samples (HAD-BP) showed lower values of both antiradical activity and total polyphenolic content compared to the hot-air-dried flour sample combined with ultrasound (US-HAD-BP). As shown in Table 5, a higher TPC value was highlighted in the US- HAD-BP sample (33.054 mg GAE/g) compared to the HAD-BP (32.111 mg GAE/g) and "00" flour samples (CTRL) (3.676 mg GAE/g). The highest increase in antiradical activity was

also observed in US-HAD-BP with values of 49.067 mmol TE/100 g and 42.632 mmol TE/100 g for the DPPH and ABTS tests, while the lowest was recorded for the control flour (CTRL-P) (3.915 and 4.423 mmol TE/100 g for the DPPH and ABTS tests, respectively).

Table 5. Antioxidant and antiradical activity of blackberry powders prepared with two different treatments, HAD (HAD-BP) and US + HAD (US-HAD-BP), and "00" type control flour (CTRL-P). Results indicate mean values ± SEMs. Data within a column followed by different letters were significantly different according to Tukey's test

	CTRL-P	HAD-BP	US/HAD-BP	SEM	*p* value
TPC mg GAE/g	2.972 a	32.111 b	33.054 b	4.948	<0.0001
DPPH mmol TEAC/100 g	3.915 a	48.484 b	49.067 c	7.477	<0.0001
ABTS mmol TEAC/100 g	4.423 a	39.576 b	42.632 c	6.130	<0.0001

Statistical analysis showed significant increases in all experimental treatments compared to the controls, especially for the US-HAD-BP sample, both for antioxidant activity and antiradical activity. The HAD-BP and US-HAD-BP samples did not show significant differences from each other for all tests performed. Same analyses were conducted on cookies fortified with 10% HAD-Cookies and on cookies fortified with 10% US-HAD-Cookies. The addition of the blackberry powder to the "00" flour significantly improved the antiradical and antioxidant activity of the samples. As shown in Table 6, the fortified US-HAD-Cookies had higher values of TPC (13.882 mg GAE/100 g) and antiradical activity (9.232 and 8.536 mmol TE/100 g for the DPPH and ABTS tests, respectively) compared to the control cookie (CTR-C) products with only "00" flour (2.326 and 3.101 mmol TE/100 g for the DPPH and ABTS tests, respectively).

Table 6. Antioxidant and antiradical activity of fortified cookies prepared with two blackberry powders HAD-Cookies and US-HAD-Cookies—and "00" type control cookies (CTRL-C). Results indicate mean values ± SEMs. Data within a column followed by different letters were significantly different according to Tukey's test.

	CTRL-C	HAD-Cookies	US/HAD-Cookies	SEM	p value
TPC mg GAE/g	2.232 a	13.033 b	13.882 c	1.753	<0.0001
DPPH mmol TEAC/100 g	2.326 a	8.792 b	9.232 c	1.116	<0.0001
ABTS mmol TEAC/100 g	3.101 a	8.049 b	8.536 c	0.653	<0.0001

Analysis showed significant increases in all experimental treatments compared to controls, especially for the US-HAD-Cookies sample, both for antioxidant activity and anti- radical activity. The HAD-Cookies and US-HAD-Cookies samples did not show significant differences from each other for all tests performed.

3.5 Evaluation of Phenolic Compounds

UHPLC-ESI-MS analysis of the polyphenolic profile of the fresh blackberry fruits (F. Blackberries) showed (Table 7) an abundance of phenolic acids, organic acids, and flavonoids; most detected compounds were protocatechuic acid (123.3 mg/100 g), ellagic acid (292.25 mg/100 g), quercetin 3-O-glucoside (678.1 mg/100 g), and dihydrokaempferol (276.4 mg/100 g). The two blackberry powders prepared with two different HAD (HAD-BP) and US + HAD (US-HAD-BP) treatments showed lower values compared to the fresh fruit but a qualitatively higher profile than the control flour of type "00" (CTRL-P). Samples subjected to the US-HAD treatment showed slightly higher values, especially for ellagic acid (78.67–38.9 mg/100 g for US-HAD-BP and HAD-BP). The quali-quantitative analysis of fortified cookies (HAD-Cookies and US-HAD Cookies) highlighted a slight increase compared to the control cookies. In this case, the values of the phenolic compounds detected were like each other, except for benzoic acid (0.9–1.8 mg/100 g for HAD-Cookies and US-HAD-Cookies, respectively). Protocatechuic acid and caffeic acid, absent in the control cookies (CTRL-C), were instead revealed in the HAD-Cookies and US-HAD-Cookies samples, assuming that their presence was only due from the fortification with blackberry powder.

Table 7. Phenolic compound evaluation of fresh blackberry fruits and blackberry powders prepared with two different treatments—HAD (HAD-BP) and US + HAD (US-HAD-BP)—and "00"-type control flour (CTRL-P) and of fortified cookies prepared with two blackberry powders—HAD-Cookies and US-HAD-Cookies—and "00"-type control cookies (CTRL-C). Results indicate mean values ± SEMs; n.d.: not detected.

	F. Blackberries	**CTRL-P**	**HAD-BP**	**US/HAD-BP**	**CTRL-C**	**HAD-Cookies**	**US/HAD-Cookies**
				mg/100g			
Phenolic acids							
Protocatechuic acid	123.3	n.d.	34.6	34.3	n.d.	3.1	3.2
p-coumaric acid	37.3	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Ellagic acid	292.25	n.d.	38.9	78.67	n.d.	n.d.	n.d.
Organic acids							
Benzoic acid	20.1	n.d.	0.4	1.2	0.5	0.9	1.8
Caffeic acid	92.55	n.d.	n.d.	3.22	n.d.	n.d.	3.22
Flavonoids							
Quercetin-3-O-glucoside	678.1	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Dihydrokaempferol	276.4	18.6	38.2	25.4	17.6	21.2	20.9

3.6 Sensory Analysis

The sensory evaluation of the cookies was conducted 24 h after baking. The results are shown in Figure 5. From the results obtained, it can be observed that about the visual descriptors (SC, IC, CO), the judges, although there was a clear difference in colouring between the control (CTRL-C) and theses (HAD and US-HAD), evaluated these descriptors positively in the treated samples; in fact, no significant difference was found compared to the control. In the olfactory descriptors, the cookie odour (BO) was found in all samples while the blackberry odour (MO) was only detected in the HAD thesis. For the taste descriptors, important results can be seen in the texture. In particular, the descriptors H, S, C, and CH showed no differences between the samples, indicating appreciation, but above all that, the addition of the powders did not cause a change in the rheological and mechanical characteristics of the treated samples compared to the CTRL-C sample. This is of critical importance, both for shelf life but also because it makes it possible to standardize production and place on the market products that, in terms of texture, are perfectly identical to a classic cookie made with 100% wheat flour "00' '. However, significant differences were found in the blackberry flavour (BT), indicating that the addition of 10% of the powder was sufficient for the characteristic taste of the small fruit to be detected by the judges. Confirming this, the SN descriptor was reported to be intense in line with the classic taste of fresh blackberry fruits. Negative descriptors such as UO and UT received the lowest scores, so, in addition to the health aspects, we can confirm that the addition of the powders was also highly appreciated from a sensory point of view with no significant difference between the HAD-Cookies and US-HAD-Cookies samples.

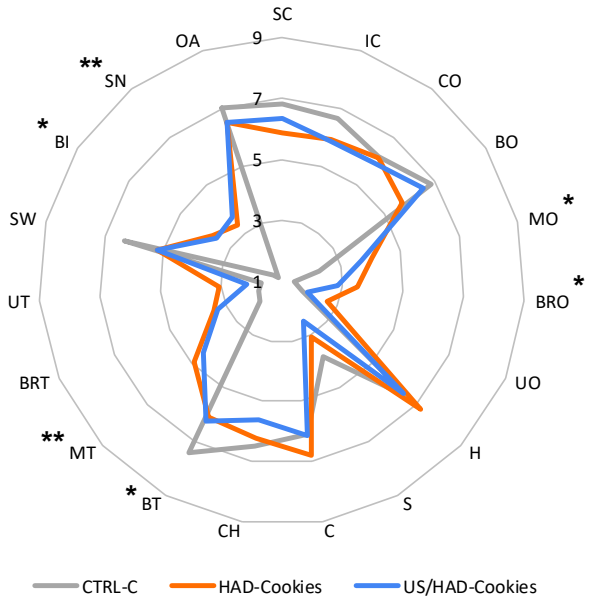

Figure 5. Sensory evaluation of blackberry cookies (HAD-Cookies and US-HAD-Cookies) after baking. The spider plot indicates the average intensity values of the descriptors, ascribed by judges, on a hedonic scale from 1 to 9. Asterisks indicate statistically significant differences between treatments with * for $p < 0.05$, ** for $p < 0.01$; no asterisk indicates a non-significant difference. Legend: SC (surface colour); IC (internal colour); CO (colour homogeneity); BO (cookie odour); MO (blackberry odour); BRO (burnt odour); UO (unpleasant odour); H (hardness); S (softness); C (crispness); CH (chewiness); BT (cookie taste); MT (blackberry taste); BRT (burnt taste); UT (unpleasant taste); SW (sweetness); BI (bitterness); SN (acidity); OA (overall acceptability).

4. Discussions

TSSC is a critical indicator of fruit sweetness. The value of TSSC is crucial for assessing fruit quality for both fresh consumption and processing as a higher TSSC enhances flavour and perceived sweetness in the final product [57]. Our data showing a TSSC of 8.36±0.08 ◦Brix confirmed that the analysed blackberries had a substantial level of sweetness, making them suitable for both direct consumption and processing. With a TA of 0.29±0.07 g/L, our findings indicate that the blackberries had a good balance of sweetness and acidity, which not only contributed to their flavour but also helped in maintaining microbial stability [14,58]. The values of L*, a*, b*, and RGB collectively indicated that the blackberries possessed a dark colour, typical of ripe fruits (Table 1) [14]. This colouration is significant not only for visual appeal but also for antioxidant and nutritional properties as a darker colour is often associated with higher contents of anthocyanins and other beneficial phenolic compounds [59]. The data confirmed that the analysed blackberry fruits were suitable for both direct consumption and processing, affirming their quality for intended uses. These findings are consistent with those of Hassimotto et al. (2008) and Schulz et al. (2019) [60,61], highlighting their ideal maturity and quality for consumption and processing. Drying is a critical process in food preservation, with the aim of removing moisture from the fruit while maintaining its nutritional content and sensory attributes. Effective methods of drying play a crucial role in maintaining product quality, including texture, flavour, and colour [62]. Moreover, shorter drying durations minimize prolonged exposure to high temperatures, which can degrade bioactive compounds such as vitamins, anthocyanins, and phenols [63]. This preservation helps maintain the nutritional value, colour, flavour, and texture of the food product. Our results indicate that pre-treatment with ultrasound reduced the drying time by one hour to achieve a comparable dry residue. Thus, ultrasound-assisted hot air drying (US-HAD) technology emerges as a preferred option for the food industry, combining operational efficiency and sustainability while optimally preserving product characteristics. An important quality parameter for consumers is the visual appearance of the fruit. Colour is the attribute that most influences consumers when purchasing fruits [64]. In fact, it is considered the most important visual attribute in quality perception [65]. Fruit drying, as reported in the literature [26,66,67], influences changes in the texture, flavour, and

taste and in the characteristic colour of the fruits subjected to the process. Table 3 illustrates the colour differences (ΔE) in blackberry powders obtained from both treatments compared to fresh blackberries. The drying process induced a colour difference (ΔE) signifying an alteration of the overall colour in both treatments. These results indicate that ultrasound treatment (US-HAD-BP) causes significantly less colour variation compared to conventional treatment (HAD-BP) (Figure 2). This significant difference in the a* value of US-HAD-BP powder suggests that ultrasound pre-treatment helped preserve the red component better in the final powders due to the presence of anthocyanins. Pre-treatment with ultrasound improves the preservation of the red component in US-HAD-BP powder as it facilitates a faster extraction of water from fruits, thus minimizing their exposure to heat and light, factors that contribute to their degradation [68]. This process also helps stabilize the anthocyanins, ensuring their subsequent processing at lower temperatures during drying, thus preserving the integrity of these heat sensitive compounds. Reduced colour variation is generally preferred as it suggests the better preservation of the original colour, which is a key indicator of visual quality and may reflect the lesser degradation of pigmented compounds such as anthocyanins [69,70]. Anthocyanins are sensitive to various physical and chemical treatments including high temperatures [70]. Ultrasound, however, may have helped preserve these compounds better than conventional HAD treatment, as well as reducing the general browning caused by the drying process due to the shorter treatment time. This hypothesis is supported by previous studies showing that ultrasound can increase cell permeability, improve the efficiency of the drying process and preserve bioactive compounds [71]. Li et al. (2020) [72] reported that ultrasound treatment with different powers for 20 min significantly improved the values of L*, a*, and b* in wine samples by stabilizing the colour loss. This indicates that the yellow component of the powders remained unchanged between the two treatments, suggesting that ultrasound had no significant effect. Also, baking is a complex process that induces physical, chemical, and biochemical changes in the grain matrix, including volume expansion, water evaporation, the formation of a porous structure, protein denaturation, starch gelatinization, crust formation, and browning [73]. The colour of the cookie surface is a fundamental characteristic closely linked to aroma, texture, and appearance—critical aspects for consumers. These findings indicate that the HAD-Cookies sample closely resembled the colour of the control cookies, experiencing less colour variation compared to the US-HAD-Cookies sample. The interaction of anthocyanins with other ingredients in the cookie matrix, such as proteins and fats, can influence the final colour [74]. The better-preserved anthocyanins from the ultrasound pre-treatment might

interact differently during baking, causing a higher colour difference. In fact, the better preservation of the a* parameter after baking in the HAD-US-Cookies samples could be attributed to the higher preservation of anthocyanins. Considering that the control sample represented the standard consumer reference colour, it is likely that consumers would prefer the HAD-Cookies sample over the US-HAD-Cookies sample due to their closer resemblance in colour to the standard. Often, colour serves as an indicator of baking degree. The formation of colour and aroma in the crust of bakery products during baking primarily results from the Maillard reaction [75]. These data demonstrate the consistent trend in results across all colourimetric parameters influenced by the baking process, albeit with higher values due to baking. Our results revealed also that the incorporation of blackberry powder into cookie formulas led to significant increases in antioxidant activity, total phenolic content (TPC), and polyphenolic profile proportionate to the percentage replacement of blackberry powder used in the production recipe. Results obtained from the evaluation of total phenolic content (TPC) and radical scavenging activity were in accordance with those obtained by Gil-Martínez et al. (2023) [76], which highlighted TPC values for blackberries equal to 31.1±4.9 mgGAE/g and antiradical activity values equal to 57.6±8.3 mmol TEAC/100 g; in this study, the blackberries were dried at 45 ◦C and the ground dry material was extracted with an ethanol/water solution (50:50 *v/v*) at 50 ◦C with constant stirring for 14.5 h. Albert et al. (2022) [77] found a similar range of antioxidant activity values (21.43–33.11 mg GAE/g) on fresh frozen blackberries extracted with different solvents (80% ethanol, 70% acetone + 2% acetic acid, 60% methanol + 3% formic acid, 90% acetonitrile + 10% HCl 6 M). Similar values were found in the work of Jazic et al. (2018) [78]; the authors researched antioxidant activity in the ethanolic extracts of previously dried wild and cultivated blackberries. The results showed a range of close values between 21.59 and 40.18 mg GAE/g. Dai et al. (2007) [79], however, obtained lower levels of TPC (17.32 mg GAE/g) and antiradical activity (66.98 µmol TEAC/g d.w.). In this study, frozen blackberry puree was freeze-dried and extracted in ethanol acidified with 0.01% HCl. Santos et al. (2023) [80], for example, analysed an extract using ultrasound-assisted extraction, reporting slightly higher values equal to 52.36 mg GAE/g for TPC and for antiradical activity (55.56–60.86 mmol TEAC/100 g for DPPH and ABTS tests, respectively). Another study evaluated TPC in blackberries using near infrared spectroscopy (NIRS) and showed values between 17.36 and 36.78 mg GAE/g [81]. In a recent study by Sik et al. (2024) [82], the antioxidant properties of wild blackberry and its possible use as a functional ingredient to produce fortified muffins were evaluated. The work involved ultrasound-assisted methanolic extraction acidified with 0.5%

HCl. The total polyphenol content of the fruits was equal to 53.8 mg GAE/g; this process highlighted an increase in antioxidant activity and greater acceptability in the finished product. The results of the study suggest that blackberries can be used as functional ingredients to increase the antioxidant activity values of a food [82]. Higher phenolic acid values than those found in the literature were highlighted in fresh blackberry fruits. Sellappan et al. (2002) [83] found ellagic acid values in a range between 30.01 and 30.8 mg/100, like those found (35.7–54.7 mg/100 g) by DJuric et al. (2014) [84]. In the study by Da Silva et al. (2018) [85], the beneficial health effects attributable to the polyphenols present in blackberries were evaluated and the availability of phenolic compounds after the transformation of blackberries into jam was investigated. Significantly fewer phenolic compounds were identified in the jam, indicating degradation during the heating process; this could also explain the significant loss of phenolic compounds in the samples analysed in this study. However, the finished product improved its antioxidant activity (ABTS assay) of the processed products after storage respect to the control, which may be related to the development of new compounds with higher antioxidant activity. Therefore, the transformation of blackberries into food products and fortified foods represents a valid alternative to increase the antioxidant activity of the finished product [85]. Finally, the sensory evaluation shows that the addition of blackberry powder was well received by the judges, with positive ratings for visual, aromatic, and taste attributes. The negligible differences between the HAD-Cookies and US-HAD-Cookies samples further emphasise the effectiveness of ultrasound pre-treatment in maintaining sensory quality attributes comparable to conventional drying methods. Belšcak-Cvitanovic´–Zoran and Opalìc (2013) [86] demonstrated that pear samples subjected to longer ultrasound treatments (35 and 45 min) and dried for extended periods show darker colouring, harder texture, and the presence of cracks, leading to lower sensory acceptability. This effect could probably also be transferred to foods when these products are used as ingredients as in our case. These results not only validate the sensory appeal of blackberry-enriched cookies but also suggest potential for the standardized production and marketability of fruit-enriched baked goods.

5. Conclusions

In this study, the effects of the addition of blackberry powder dried by two different methods, hot air drying (HAD) and ultrasound-assisted hot air drying (US-HAD), on the physical and sensory characteristics of functional cookies were examined. The US-HAD method showed a significant reduction in drying time compared to the conventional HAD method, saving approximately one hour of time and energy to reach a final moisture content of about 12%.

The results showed that cookies enriched with blackberry powders, both HAD and US-HAD, presented a significant increase in antioxidant activity compared to the control cookies (CTRL-C). The values of antioxidant activity measured with the ABTS and DPPH tests, were higher than those of the control cookies. UHPLC-ESI-MS analysis revealed an increase in polyphenol content in the fortified cookies, with the main phenolic compounds detected including protocatechuic acid and dihydrokaempferol. Fortified cookies showed promising values of total phenolic content (TPC), directly associated with improved antioxidant properties. The sensory evaluation showed that cookies fortified with US-HAD powder scored higher in terms of blackberry flavour and general acceptability than HAD and control cookies. This suggests that the use of US-HAD technology not only improves the nutritional but also the sensory properties of cookies. The incorporation of dried blackberry powders, especially those obtained by the US-HAD method, into functional cookies not only reduces food waste but also enriches the nutritional profile of baked goods, offering a healthy and sustainable alternative in the use of damaged fruits. The absence of added sugar and fat in blackberry powder has significant health benefits for consumers, reducing the intake of potentially unhealthy ingredients and promoting a more balanced diet. From an economic point of view, the food industry can capitalize on the growing demand for healthy, fortified foods by positioning products enriched with dried blackberry powder as attractive and nutritionally advantageous options for health-conscious consumers.

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CHAPTER 7

Exploring the Addition of Mango Peel in Functional Semolina Sourdough Bread Production for Sustainable Bio-Reuse

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Abstract

Mango, a tropical fruit celebrated for its delightful fragrance and high nutritional value, generates significant waste during processing, with approximately 35–60% of the fruit being discarded. However, this waste contains valuable components, such as fibre, carotenoids, polyphenols, and other bioactive compounds. To repurpose mango peel, this study dehydrated it to create mango peel powder (MPP), which was then incorporated into sourdough bread to produce functional breads with enhanced nutritional value. Semolina was replaced with MPP at levels of 5% (MPP-5) and 10% (MPP-10) (*w*/*w*). After dehydration, the mango peel had a yield of 22%, and the procedure used did not cause any organoleptic changes. The bread fermentation process was conducted at 30 ◦C for 8 h. During dough fermentation, the pH was monitored, showing a value of 4.14 ± 0.02 in the MPP-10 dough. Overall, the MPP-10 bread received a higher score (6.51) than the control (CTR) bread (5.6) and the MPP-5 bread (6.11). The total phenolic content of the fortified breads ranged from 44.760 to 98.931 mg gallic acid equivalents (GAE)/g, and the antiradical activity ranged from 15.213 to 29.461 mmol Trolox equivalent antioxidant activity (TEAC)/100 g, depending on the percentage of enrichment.

Keywords: mango peel powder; functional bread; lactic acid bacteria; sourdough; polyphenols; antioxidant properties

1. Introduction

In the quest to enhance production quality and nutritional value, the food industry is increasingly focusing on sustainable supply chains. Often overlooked, waste materials contain valuable bioactive compounds that can be harnessed to fortify various products [1]. By utilizing by-products from plants and fruits, innovative functional foods have been successfully developed [2]. Mango, a tropical fruit prized for its fragrance, taste, and nutritional value, grows naturally in South Asia and is cultivated in diverse regions world-wide, including India, Mexico, Thailand, and the coasts of Sicily, where favourable weather conditions prevail areas [3]. The steady increase in demand for mangoes in the European market has led to a rise in exports, both within Italy and abroad. Being a climacteric fruit, mangoes undergo rapid ripening after harvest and are often used in the production of fresh products, juices, or dried or freshly cut fruit [4]. A substantial part of the mango (35–60%) is discarded during processing, without adequate treatment, resulting in environmental issues and economic losses [5]. Mango peel is rich in fibre, with approximately 16% to 28% soluble fibre and 29% to 50% insoluble fibre. It also contains valuable phytochemicals, including carotenoids, polyphenols, and other bioactive compounds, which are associated with potential health benefits [6–8].

Traditional bread is conventionally leavened using either baker's yeast or sourdough technology [9]. Sourdough, in particular, benefits from a diverse microbial environment within which lactic acid bacteria (LAB) and yeasts coexist, adding various desirable qualities to the final product [10]. Compared with solely using baker's yeast, sourdough fermentation offers numerous nutritional and functional advantages. These include lowering the glycaemic index, enhancing the properties of dietary fibre complexes, improving mineral and vitamin absorption, and promoting the production of beneficial metabolites during fermentation, such as peptides and amino acid derivatives like aminobutyric acid [11,12]. In alignment with European initiatives aimed at reducing waste and enhancing the sustainability of the food industry, this study explores the use of mango peel (MP) in powdered form (MPP) as an ingredient for making semolina bread. Although MPP has been used to enrich bread made with wheat flour and baker's yeast [13–15], there are no published studies on incorporating these by-products into sourdough Italian-style breads. The objective of this research is to functionalize traditional sourdough bread produced in southern Italy. By repurposing MPP, valuable polyphenols can be introduced into a staple food within the nutritional pyramid.

2. Materials and Methods

2.1 Isolation and Identification of LAB from Mango Peel

Mango fruits (*Mangifera indica* L., variety Keitt) were harvested from "Azienda Agricola Tripodo-Collura" in Acquedolci, Sicily, Italy (38◦03′27″ N–14◦36′38″ E 50 m s.l.m). The peel was manually removed using stainless steel knives and then stored at 20 ◦C until microbiological analysis. Presumptive LAB were isolated from the mango peel at the highest cell densities, using the following specific media for plate counting: De Man–Rogosa–Sharpe (MRS), M17, and sourdough bacteria (SDB). Incubation occurred at 30 ◦C for 48 h anaerobically for MRS and M17 plates, while SDB plates were incubated aerobically under the same conditions. To maximize LAB biodiversity, at least four colonies with similar appearances in terms of shape, size, colour, edge, surface characteristics, and elevation were isolated. All isolates underwent preliminary characterization through Gram staining following the method described by Gregersen [16] and a catalase test as reported by Koneman et al. [17]. Only Gram-positive cultures negative for catalase expression were further purified through successive sub-culturing and stored in glycerol stocks at -80 °C. The acidification kinetics were assessed following the method described by Alfonzo et al. [18], employing a sterile semolina extract (SSE) at 20% concentration as the growth medium. LAB cultures were propagated, washed, and inoculated into SSE without maltose. The pH decrease was monitored at 0, 2, 4, 6, 8 and 24 h after inoculation. The LAB isolates underwent genetic characterization. Overnight cultures were grown in their optimal growth media at 30 ◦C, and genomic DNAs was extracted from pelleted cells using the Insta Gene Matrix kit (Bio-Rad, Hercules, CA, USA) following the manufacturer's instructions. Crude cell extracts served as templates for PCR. Random amplification of polymorphic DNA (RAPD)-PCR analysis was performed to differentiate the isolates at the strain level. Specifically, the M13 primer, as described by Stenlid et al. [19], was used, and the resulting amplicons were separated and visualized according to Ventimiglia et al. [20]. RAPD profiles were analysed using Gelcompar II software, version 6.5 (Applied-Maths, Sint-Martens-Latem, Belgium). The strains were identified at the species level by 16S rRNA gene sequencing. PCR reactions were performed using the primers fD1 (5′-AGAGTTTGATCCTGGCTCAG-3′)/rD1 (5′-AAGGAGGTGATCCAGCC-3′) as described by Weisburg et al. [21]. The resulting PCR products had a molecular size of about 1600 bp, which was confirmed using agarose gels. After purification with the QIAquick purification kit (Quiagen S.p.a. Milan, Italy), the amplicons were sequenced using the same primers used for PCR amplification at AGRIVET (University of Palermo, Italy). The obtained sequences

were then compared with those available in the GenBank/EMBL/DDBJ https://www.ncbi.nlm.nih.gov (accessed on 10 June 2024) and EzTaxone databases https://eztaxon-e.ezbiocloud.net (accessed on 10 June 2024).

2.2 Mango Peel Powder Production and Characterization

MPP was prepared from mango peels that were first washed under running water (25±1 ◦C), then sanitised with 2% NaClO solution for 15 min and finally rinsed thoroughly to remove residual chlorine. To ensure uniformity during the dehydration process, the peels were sliced to a thickness of 0.5±0.1 mm and stored at -20 ◦C until further processing. Initially, the peels underwent treatment in an ultrasonic bath (DU-32, ARGOlab, Modena, Italy) at 30 ◦C for 15 min at 22 kHz and 70 W, significantly reducing the overall drying time [22]. Subsequently, the mango peels were dehydrated in a tray dryer, as described by Roppolo et al. [23], at 60 ◦C for 3 h. During the drying process, the trays with peels were weighed every 30 min to halt the process once a 20% dry residue (DR) was reached, thereby preserving bioactive compounds. The %DR was calculated using the following Equation (1):

$$\%DR = \left(\frac{c-a}{b-a}\right) \times 100 \quad (1)$$

where

a is the weight of the empty tray;

b is the weight of the tray with the product before drying;

c is the weight of the tray with the product after drying.

The colour of raw materials (MPP and semolina) was assessed using a digital colourimeter (Minolta, mod. CR-300; Osaka, Japan) to evaluate the degree of browning based on the CIELab colourimetric system. This system determines colour using three coordinates: L* for brightness (with L* = 0 representing black and L* = 100 representing white), a* for the green/red colour index (where a* = -100 indicates green and a* = +100 indicates red), and b* for the blue/yellow colour index (with b* = -100 signifying blue and b* = +100 signifying yellow). Chroma values (C*), indicating the quantitative attribute of colour intensity, were calculated using Equation, as follows (2):

$$C^* = \sqrt{a^2 + b^2} \quad (2)$$

Hue angle (h◦), describing the colour tone, was calculated using the following Equation (3):

$$h^\circ = arctan\left(\frac{b^*}{a^*}\right) \quad (3)$$

The mango peel was processed using an ultra-centrifugal mill (Fritsch, Pulverisette 14, Lainate, Italy) at a speed of 10,000 rpm for 10 s. The resulting mango peel powder (MPP) was sieved using a Retsch centrifugal apparatus (Mill ZM1, Haan, Germany) equipped with a 250 µm stainless steel ring sieve. The MPP was then analysed for colour, weighed, packed into sealed glass containers, and stored at room temperature (20 ±1 ◦C). MPP underwent microbiological analysis to identify unwanted microbial groups during food fermentation. Ten grams of MPP were homogenized using a BagMixer® 400 stomacher (Interscience, Saint Nom, France) and then serially diluted. All cell suspensions were inoculated in agar media to promote the growth of different microbial groups, as follows: total mesophilic microorganisms (TMM) as well as total psychrophilic microorganisms (TPM) were cultivated on plate count agar (PCA) and incubated aerobically at 30 ◦C for 72 h and at 7 ◦C for seven days, respectively; yeasts were cultivated on yeast peptone dextrose (YPD) under aerobic incubation at 28 ◦C for 48 h; members of the Enterobacteriaceae family were incubated on violet red bile glucose agar (VRGBA) under microaerobic incubation at 37 ◦C for 24 h; total coliforms were incubated on violet red bile agar (VRBA) under microaerobic incubation at 37 ◦C for 24 h; pseudomonads were incubated on *Pseudomonas* agar base (PAB) supplemented with cephaloridine sodium fusidate cetrimide under aerobic incubation at 25 ◦C for 48 h; *Escherichia coli* and *Salmonella* spp. were incubated on Hektoen enteric agar (HEA) under aerobic incubation at 37 ◦C for 24 h; and *Listeria monocytogenes* was incubated on *Listeria* selective agar base (LSAB) added with SR0140E supplement under aerobic incubation at 37 ◦C for 48 h. All media and supplements were purchased from Oxoid (Milan, Italy). Plate counts were performed in duplicate.

2.3 Sourdough Propagation

A mixture of LAB strains from the Culture Collection of the Agricultural Laboratory, University of Palermo (Italy) was used to initiate a sourdough inoculum. The strains included *Fructilactobacillus sanfranciscensis* SD22, *Weissella cibaria* SD123, *Leuconostoc holzapfelii* SD148, and strains isolated in this study, which were considered intrinsically resistant to the MPP polyphenols. These strains were reactivated from -80 ◦C glycerol stocks and propagated in SSE at 30 ◦C for 24 h. The mixed culture was then diluted in sterile tap water to achieve a final volume of 187.5 mL. This cell suspension was added to 312.5 g of semolina, resulting in a 500 g of dough with a dough yield (DY = weight of the dough/weight of semolina x100) of 160. The cell density in the dough was approximately 106–107 CFU/g. The dough was fermented at 28 ◦C for 16 h and underwent seven consecutive daily refreshments to develop a mature sourdough inoculum. The semolina used for bread

production was sourced from the brand “La Molisana” (C.da Colle delle Api 100/A, Campobasso, Italy). The labelled nutritional values per 100 g were as follows: 14 g of protein, 70.0 g of carbohydrates, 1 g of fat, 0.3 g of saturated fat, 3 g of fibre, and 0.02 g of salt.

2.4 Production of Dough

The bread production process exclusively utilized sourdough developed from the selected LAB strains. No baker’s yeast or salt was added to assess the impact of MPP on LAB performance. The control (CTR) trial (800 g dough) was prepared by adding 228.6 mL of sterile tap water and 457.2 g of semolina to 114.2 g of mature sourdough to reach a final DY = 175. The experimental MPP trials followed the same procedure with the same amount of water and sourdough but reduced the semolina to 417.1 g and to 377.2 g for the MPP-5 (containing 5% *w*/*w* MPP) and MP-10 (containing 10% *w*/*w* MPP), respectively. The ingredients were mixed using a planetary mixer (model XBM10S Electrolux Professional, SpA, Pordenone, Italy) equipped with a paddle at speed 4 for 15 min. Aliquots of 100 g per dough were transferred into trapezoidal stainless steel baking pans with the dimensions specified by the American Association of Cereal Chemists—Method 10-10B of AACC [24] [143x79 mm (top inside), 129x64 mm (bottom outside), 57 mm (depth inside)]. The pans were covered with aluminium foil and fermented at 30 ◦C for 8 h. The baking process included an initial exposure of the doughs to hot air/steam at 200 ◦C for 5 min, followed by 15 min of convection heat (hot air only) at the same temperature. Each production was performed in duplicate (technical repeats), and the experiment was conducted twice (independent replicates).

2.5 Fermentation Monitoring

During sourdough fermentation, the acidification process and the levels of the main fermenting microorganisms were analysed at two time points, as follows: the beginning (T_0) and the end (T_2) of fermentation. The pH was measured using a Russell RL060P pH meter (Thermo Fisher Scientific, Beverly, MA, USA). The total titratable acidity (TTA) was determined by titration with 0.1 N NaOH and expressed as the volume of NaOH (in mL) required for 10 g dough. This method follows that of the official American Association of Cereal Chemistry [25].

2.6 Microbiological Analysis

Microbial loads were assessed in the raw materials, the doughs immediately after ingredient mixing, and the sourdoughs at different fermentation stages. For each sample, 10 g were

suspended in 90 mL Ringer's solution, homogenized using a stomacher (BagMixer® 400, Interscience, Saint Nom, France) for 2 min at the highest speed, and then subjected to decimal serial dilution. Dilutions were plated and incubated as follows: TMM on plate count agar (PCA), incubated aerobically at 30 ◦C for 72 h; sourdough LAB on SDB, incubated at 30 ◦C for 48 h; and total yeasts on yeast extract peptone dextrose (YPD), also incubated at 30 ◦C for 48 h. To inhibit fungal growth, cycloheximide (10 mg/mL) was added to SDB, while chloramphenicol (0.05 mg/mL) was added to YPD to inhibit bacterial growth. Additionally, the investigation included the detection of members of the Enterobacteriaceae family, total coliforms, and the presence of spore-forming aerobic bacteria, as explained in Section 2.2. Microbiological analysis was conducted in duplicate, and the results are expressed as log colony forming units (CFU)/g.

2.7 Analysis of Bread Attributes

After cooling at room temperature for 30 min, bread quality attributes were assessed as described by Viola et al. [26]. The evaluation included the following parameters: weight loss (%), moisture (%), specific volume (cm3/g bread), firmness (N/mm2), crumb and crust colour determination, image analysis to determine void fraction (%), cell density, and mean cell area (per cm2). The analyses were performed in duplicate.

2.8 Total Phenolic Content Analysis of Raw Materials and Bread Samples

Total phenolic content (TPC) was determined using the Folin–Ciocalteu method as described by Viola et al. [26] with slight modifications. Specifically, 0.5 g of each sample (semolina, MPP, CTR, MPP-5, and MPP-10) was added to 8 mL of methanol/water solution (80:20 *v*/*v*). The mixtures were kept in an ultrasonic water bath and sonicated for 45 min. The extracts were filtered through 0.45 µm PTFE filters (Whatman, Milan, Italy). Then, 100 microliters of filtrate solution were added to 625 µL of Folin–Ciocalteu reagent and 120 µL of 7% (*w*/*v*) Na_2CO_3 solution. The mixtures were incubated in the dark at 25 ◦C for 60 min. The reaction formed a blue-coloured complex, with colour intensity proportional to the phenolic compounds present. The resulting colourimetric reaction was measured at 765 nm using a UV–Vis spectrophotometer (UV-1600PC, VWR, Radnor, PA, USA) with methanol as the blank. TPC was calculated by interpolating from a calibration curve of gallic acid (GA), used as the standard (ranging from 0.01 to 0.5 mg/mL). The results are expressed as milligrams of gallic acid equivalents per gram of the sample [mg gallic acid equivalents (GAE)/g]. Experiments were performed in triplicate.

2.9 Antiradical Activity

The antiradical activity of the samples was evaluated using the 2,2-diphenyl-1-picrylhydrazyl (DPPH) and 2,2′-azino-bis (3-ethylbenzothiazoline-6-sulfonic acid) (ABTS) assays, as previously described by Di Stefano et al. [27]. For both tests, scavenging activity was assessed by measuring the decrease in the solution's colour, which is proportional to the amount of antioxidant in the sample. One gram of each sample was added to 4 mL of methanol, sonicated for 40 min, and filtered through Whatman 0.45 µm PTFE filters. The filtrate was then subjected to DPPH and ABTS tests. For the DPPH assay, 0.100 mL of the filtrate was mixed with 3 mL of DPPH (60 µM) and incubated in the dark at 25 ◦C for 30 min. Scavenging activity was measured by spectrophotometric analysis of the absorbance at a wavelength of 517 nm using a UV–Vis spectrophotometer (UV-1600PC, VWR). For the ABTS assay, the ABTS+ radical cation was produced by reacting ABTS stock solution with 2.45 mM potassium persulfate, according to Re et al. [28]. An aliquot of filtered samples (0.100 mL) was mixed with 3 mL of ABTS, and, after 5 min, the absorbance of the mixture was read at 734 nm using a UV–Vis spectrophotometer (UV-1600PC, VWR). Methanol was used as the blank for both assays. Two calibration curves, using Trolox as the standard at increasing concentrations (1–75 µM), were constructed. Experiments were performed in triplicate. The results are reported as mmol Trolox equivalent antioxidant activity (TEAC) per 100 g of sample.

2.10 Analysis of Volatile Organic Compounds

Solid-phase microextraction (SPME) was followed by gas chromatography–mass spectrometry (GC–MS) to analyse volatile organic compounds (VOCs) in bread with or without MPP addition. Each sample was chopped, and an amount of 5 g of each sample was exposed to an SPME fibre [30 µm divinylbenzene (DVB)/carbowax (CAR)/polydimethylsiloxane (PDMS)] (Supelco, Bellefonte, PA, USA) at 25 ◦C for 60 min. The SPME fibre was subjected to 250 ◦C for 5 min inside the GC injection port after adsorption. A DB-624 capillary column (Agilent Technologies, Santa Clara, CA, USA, 60 m, 60 m, 0.25 mm, 1.40 µm) was used for chromatographic separation. The GC oven temperature ramped up from 40 to 230 ◦C at a rate of 4 ◦C/min, remained isothermal for 40 min, and the final temperature was held for 2 min. Mass acquisition was performed in full scan mode in a range from 40 to 400 *m*/*z*, with the interface temperature at 230 ◦C. VOCs were identified by comparing their mass spectra with the NIST05 library. Results were expressed as percentages, obtained by normalizing peak areas with the total area of significant peaks. Each bread sample was analysed in triplicate.

2.11 Sensory Evaluation

Sensory analysis was conducted with a trained panel to evaluate bread attributes. The sensory characteristics of the final bread were analysed according to ISO 6658 guidelines. The judges assessed descriptors related to bread, including crust colour, thickness, crumb colour, porosity, alveolation, and alveolation uniformity [29–31]. They also considered sensory aspects, such as bread aroma intensity, unpleasant odour, unpleasant aroma, salty taste, acidic taste, astringent taste, bitter taste, taste persistence, mouth adhesiveness, crispness, and overall assessment. The judges used a visual analogue scale, marking a point between "dislike/low quality" (left end) and "like/high quality" (right end). This scale captured their preferences and acceptability. Results from the hedonic scale were then converted into distances (in centimetres) from the left end of the line. This approach helps quantify sensory preferences and quality perception.

2.12 Statistical Analyses

The experimental data were analysed using R, version 3.2.2. Analysis of variance (ANOVA) was used for pair comparisons. Post-hoc analysis to identify differences between groups was performed using Tukey's test from the Agricolae package, version 1.3-5. Heat map cluster analysis was used to investigate the distribution of VOCs among breads. The sensory traits were graphically represented as a radar chart created with the fmsb package. In all cases, a significant level of 5% was used.

3. Results and Discussion

3.1 Isolation and Identification of LAB from Mango Peel

No growth was observed on MRS, M17, and SDB media from fresh mango peel dilutions. Consequently, 10 g of mango peel were directly added to 50 mL of MRS and M17 media. Colonies with morphologies consistent with LAB grew on the MRS medium. Phenotypically, the strains were catalase-negative and lacked an external membrane. RAPD–PCR was used to differentiate the strains of presumptive LAB. All isolates sharing a given RAPD profile were considered to represent the same strain. Thus, the dominant LAB populations in the mango peel samples included only four strains. These selected strains underwent 16S rRNA gene sequencing, which revealed the presence of three LAB species. Specifically, three obligate homofermentative LAB were identified as *Latilactobacillus sakei*. This LAB plays a crucial role in fermenting meat products due to its ability to produce lactic acid and enhance food safety and flavour [32]. *Latilactobacillus sakei* has also been

used to enhance flavour impact in fermented wheat gluten [33], but its application in bread remains unexplored.

3.2 Characteristics of Mango Peel

The MP yield, calculated from the dry weight of the peels, was 22%, exceeding that previously obtained by De León Monzón [34]. This result highlights the efficiency of our drying method in maximizing yield while effectively preserving the nutritional content of the dried product. Drying is a critical process in food preservation, designed to eliminate moisture from fruits while retaining their nutritional content and sensory qualities. Effective drying techniques are essential for maintaining product quality, including flavour, colour, and texture [35]. Moreover, shorter drying times reduce the risk of prolonged exposure to high temperatures, which can degrade bioactive compounds like anthocyanins, phenols, and vitamins [36]. This preservation process ensures that the nutritional and quality attributes of the food product remain intact. As demonstrated by Roppolo et al. [22], ultrasound treatment reduces the drying time required to achieve a dry residue comparable to that of food dried without its use. Therefore, based on these studies, the same ultrasound-assisted hot-air-drying technology was adopted as a preferred option for the food industry. This approach makes it possible to produce dried products that faithfully retain the characteristics of their fresh counterparts. Both fresh mango peel and powdered mango peel were found to be safe for consumption, as pathogen levels were below the detection limit. In terms of colour, semolina and MPP exhibited significant differences (Table 1).

Table 1. Comparative analysis of colour parameters.

Samples	L*	C*	h*	RGB
Semolina	91.57±0.06a	23.00±0.14[a]	-1.43±0.0[a]	
MPP	59.13±1.80b	35.02±1.51[b]	0.801.35[b]	
***p* value**	<0.0001	<0.0001	<0.0001	

Results indicate mean value S.D. (standard deviation) of lightness (L*), chroma (C*), and hue angle (h◦) parameters of colour between semolina and mango peel powder (MPP). Data within a column followed by different letters are significantly different ($p < 0.05$). n.a. = not analysed.

The semolina sample showed higher brightness (91.57±0.06) compared to MPP (59.13±0.06), indicating a visually lighter appearance. However, semolina exhibited a lower chroma value (23±0.14), suggesting a less intense colour. Conversely, MPP showed a higher C* value (35.02±1.51), indicative of greater colour intensity, despite having a lower h^{o} value (0.80±1.35), corresponding to a light brown hue. These results show a trend like that observed in avocado powder [26]. The higher brightness of semolina may be linked to its refined nature, whereas the richer chroma and distinct hue of MPP reflect the presence of

natural pigments and bioactive compounds in mango peel powder, which are retained during the drying and milling processes. These findings highlight the potential of MPP as a natural colourant and nutrient source, aligning with the trend to incorporate more natural ingredients into food products.

3.3 Monitoring of the Fermentation Process

The fermentation process carried out by LAB strains, added as starters, was closely monitored. It began with the preparation of a liquid inoculum in SSE. As the LAB grew in SSE medium, the initial pH decreased from 5.6 to specific values for each strain—3.80 (*F. sanfranciscensis* SD22), 3.88 (*W. cibaria* SD123), 4.38 (*Ln. citreum* SD168) and 3.95 (*Lt. sakei*)—with an average of 4.00±0.26. These findings align with the typical pH changes observed in LAB cultivated in flour and semolina extracts, as documented by Viola et al. [26]. After three days of propagation, a mixture containing all four LAB strains was obtained. This mixture was then used to develop sourdough by adding semolina and sterile tap water. The resulting mature sourdough had a pH of 3.82 and a TTA of 14.88 mL NaOH 0.1 N per 10 g. The pH and TTA values of the sourdough were like those recorded in *Ln. holzapfelii* SD148, *Lp. plantarum* SD96, and *Lp. pentosus* SD130 [37]. Additionally, comparable results were found for *Lv. brevis*, *Ln. citreum*, and *W. cibaria* [38] in multiple-species sourdough starter inocula. After seven consecutive days of refreshment, the sourdough was deemed ready for use as a leavening agent in bread production, with a pH of approximately 4, as reported by Corona et al. [39]. The pH and TTA data for doughs at times points T_0 and T_2 are presented in Table 2.

Table2. Chemical parameters of doughs.

Time (h)	Parameter	Sourdough	CTR	MPP-5	MPP-10	p Value
0	pH	3.82±0.00[a]	5.15±0.17[b]	4.95±0.00[b]	4.58±0.00[b]	0.024
	TTA	14.88±3.27[a]	3.78±0.78[b]	6.23±0.62[b]	7.96±2.39[b]	0.023
2	pH	n.a.	4.9±0.15	4.69±0.09	4.73±0.13	0.338
	TTA	n.a.	5.97±2.31	7.89±0.15	7.57±1.79	0.550
4	pH	n.a.	4.41±0.22	4.54±0.09	4.50±0.01	0.700
	TTA	n.a.	7.94±1.80	8.80±2.49	8.34±1.73	0.917
6	pH	n.a.	4.16±0.17	4.22±0.22	4.32±0.04	0.955
	TTA	n.a.	8.37±0.38	11.16±1.42	10.85±1.65	0.201
8	pH	n.a.	4.01±0.07	4.03±0.03	4.14±0.02	0.151
	TTA	n.a.	9.25±1.55	11.05±2.26	12.48±1.23	0.316

The results show the mean value standard deviation (S.D.) of 4 determinations, performed in 2 technical repeats across 2 independent experiments. Data within a line marked by different letters are significantly different ($p < 0.05$). Abbreviations: TTA, total titratable acidity; CTR, control bread; MPP-5, experimental bread enriched

with 5% (*w*/*w*) mango peel powder (MPP); MPP-10, experimental bread enriched with 10% (*w*/*w*) MPP; n.a. = not analysed.

Notably, there was a significant negative correlation between pH and TTA ($R^2 = 0.8$, $p < 0.05$), consistent with other studies [26,37,40]. Initially, the pH and TTA differed significantly between the control group and both the CTR and MPP-5 doughs ($p < 0.05$). However, by the end of the fermentation process, there were no significant differences in pH and TTA among the groups ($p \geq 0.05$). The pH values at T_2 (approximately 4) align with those obtained for powdered-almond-skin-enriched breads [37].

3.4 Microbiological Analysis

Table 3 reports the results of dough plate counts, focusing on the primary microorganisms during sourdough fermentation across three bread production trials.

Table 3. Microbial loads of doughs.

Microorganisms	Time (h)	Sourdough	CTR	MPP-5	MPP-10	p Value
TMM	0	7.6±0.54[a]	6.20±0.14[b]	6.61±0.41[b]	5.62±0.44[b]	0.00987
	8	n.a.	7.23±0.44	7.11±0.12	6.77±0.08	0.391
Sourdough LAB	0	8.54±0.93	7.52±1.31	7.41±0.24	7.78±0.69	0.316
	8	n.a.	9.20±0.17[b]	9.14±0.11[b]	8.37±0.25[b]	0.026
Yeasts	0	7.26±0.14[a]	6.33±0.00[b]	6.19±0.14[b]	5.95±0.32[b]	0.00826
	8	n.a.	7.37±0.09	6.89±0.27	7.28±0.03	0.247

Results indicate mean value S.D. (standard deviation) of 4 plate counts (carried out in 2 technical repeats for 2 independent experiments), expressed as log CFU/g. Data within a line followed by different letters are significantly different according to Tukey's test. Abbreviations: TMM, total mesophilic microorganisms; LAB, lactic acid bacteria; CTR, control bread; MPP-5, experimental bread enriched with 5% (*w*/*w*) of mango peel powder (MPP); MPP-10, experimental bread enriched with 10% (*w*/*w*) of MPP; n.a.= not analysed.

The sourdough, developed using selected LAB, exhibited cell densities of 8.54 log CFU/g for LAB and 7.26 log CFU/g for yeasts. Notably, LAB counts in the doughs (ranging from 7.41 to 7.78 CFU/g) exceeded those of TMM (5.62 to 6.61 log CFU/g), indicating successful transfer of LAB from the sourdough inoculum to the bread doughs. This observation is due to the substantial nutritional requirements of LAB, which were only partially satisfied by PCA [26]. At 0 h postproduction, yeast levels were significantly lower than those of LAB. Yet, after 8 h of fermentation, LAB cell densities increased for all trials. Notably, LAB levels in both the CTR and MPP-5 doughs were similar (9.20 and 9.14 log CFU/g, respectively), while the MPP-10 dough had a lower density (8.37 log CFU/g). Yeast levels also increased significantly, with no observed differences between doughs ($p \geq 0.05$). Regarding hygiene indicators, spore-forming bacteria and the Enterobacteriaceae family remained below the detection limit at both T_0 and T_2 of fermentation (and are hence not included in Table 3).

3.5 Bread Quality Attributes

Baking is a complex process that induces physical, chemical, and biochemical changes in the grain matrix. These changes include water evaporation, volume expansion, the creation of a porous structure, starch gelatinization, protein denaturation, browning, and crust formation [41]. The characteristics of the breads are summarized in Table 4.

Table 4. Quality attributes of bread samples.

Attributes	CTR	MPP-5	MPP-10	p Value
Weight loss (%)	14.87±1.35	14.55±5.15	12.98±2.59	0.116
Humidity (%)	46.11±0.98	47.77±3.75	55.21±4.39	0.138
Specific volume (cm^3/g)	2.45±0.66^{a}	1.97±0.15^{ab}	1.38±0.47^{b}	0.01
Firmness (N/mm^2)	0.034±0.006^{a}	0.036±0.006^{a}	0.047±0.004^{c}	<0.0001
Void fraction (%)	40.39±2.32^{a}	46.57±1.29^{b}	50.94±1.80^{c}	<0.0001
Cell density (n/cm^2)	60.44±1.31^{a}	90.0±2.83^{b}	96±4.00^{b}	<0.0001
Mean cell area (cm^2)	0.72±0.02^{a}	0.54±0.02^{b}	0.47±0.02^{c}	<0.0001
Crust colour				
Brightness	56.67±9.0^{a}	51.39±7.3^{b}	50.92±4.9^{b}	0.005
Redness	8.24±4.61	8.66±7.18	6.99±2.82	0.356
Yellowness	34.13±3.34	32.87±11.74	30.26±2.16	0.328
Crumb colour				
Brightness	68.02±2.12^{a}	57.86±3.96^{b}	54.52±2.74^{c}	<0.0001
Redness	–3.36±0.19^{a}	–1.01±0.63^{b}	0.56±0.22^{c}	<0.0001
Yellowness	22.01±0.60^{a}	19.69±0.93^{b}	22.39±0.94^{ac}	<0.0001

The results show the mean value standard deviation (S.D.) of 4 determinations, performed in 2 technical repeats across 2 independent experiments. Data within a line marked by different letters are significantly different ($p < 0.05$). Abbreviations: TTA, total titratable acidity; CTR, control bread; MPP-5, experimental bread enriched with 5% (*w*/*w*) mango peel powder (MPP); MPP-10, experimental bread enriched with 10% (*w*/*w*) MPP; n.a. = not analysed.

No significant differences were observed in weight loss percentage, moisture percentage, and firmness among different breads ($p < 0.05$). As MPP is added, the moisture percentage increases, likely due to its fibre content [42]. However, the weight percentage decreases, as also reported by Pathak et al. [13]. Additionally, the textural properties, such as hardness, cohesiveness, and springiness, also increase with the level of MPP incorporation [13]. The specific volume of the breads decreased as the percentages of MPP increased. Specifically, CTR bread had a volume of 2.45 cm3/g, while the MPP-10 bread had a volume of 1.38 cm3/g, as also observed by Chen et al. [43]. Image analysis indicated a statistically significant increase in void fraction percentage with the addition of MPP, suggesting that

MPP affected the air pockets within the bread structure. Cell density increased with the addition of MPP, consistent with findings from other studies on bread enriched with waste and by-products [26,37]. Interestingly, there was no difference in cell density between MPP-5 and MPP-10, but a significant difference was observed between fortified breads and CTR bread ($p < 0.05$). Alveolation size decreased with MPP addition, and there was a significant difference in alveolation size between CTR bread and both MPP-5 and MPP-10 trials. One of the key factors in consumer decision-making is the visual appeal of a product. Among visual attributes of foods, colour is the most influential character for consumers [4]. Indeed, it is regarded as the most critical element in how consumers perceive quality [44]. The colour of the bread surface is a crucial characteristic closely associated with flavour, texture, and appearance, which are critical factors for consumers. Incorporating MPP changed the colour parameters of both the crust and crumb in the breads. As the percentage of MPP increased, the L* and a* values gradually decreased, while all trials showed negative values for the crumbs. However, despite these results, Chen et al. [43] found that adding MPP increased the L, a, and b* values in bread. Generally, reduced colour variation is preferred as it indicates better preservation of the original colour, which is a crucial indicator of visual quality and suggests less degradation of pigmented compounds [45].

3.6 Total Phenolic Content and Antiradical Activity by DPPH and ABTS Assays

The TPC and radical scavenging activity, measured by DPPH and ABTS assays, were analysed to evaluate the functional attributes of MPP addition. The TPC is fundamental for assessing the presence of phenolic compounds, which are known for their beneficial effects, including antioxidant and anti-inflammatory properties, as well as their ability to protect against oxidative stress [46]. Additionally, the DPPH and ABTS assays allow for the determination of antioxidant capacity. The raw materials results are shown in Table 5.

Table 5. Antioxidant and antiradical activity of semolina and mango peel powder.

Samples	TPC (mg GAE/g DM)	DPPH (mmol TEAC/100 g DM)	ABTS (mmol TEAC/100 g DM)
Semolina	5.02 ^{a}	0.24 ^{a}	0.44 ^{a}
MPP	276.93 ^{b}	70.26 ^{b}	281.99 ^{b}
SEM	35.25	9.05	0.07
p value	0.002	<0.0001	0.0043

Results indicate mean values of 6 determinations (carried out in triplicate for 2 independent productions). Abbreviations: TPC, total phenolic content; mango peel powder (MPP); SEM, standard error of the mean. Letters that are different within the same column indicate significant difference ($p < 0.05$).

The results of the TPC analysis in MPP showed a significantly higher value of 276.93 mg GAE/g. Literature data report variable TPC values in mango peels; for in- stance, Dorta et

al. [47] determined TPC values to be around 70 mg GAE/g (dry weight), while Castañeda-Valbuena et al. [48] reported values around 112 mg GAE/g (dry weight). This variability can be attributed to several factors, including differences in mango varieties and preservation methods [47,48]. Interestingly, our findings reveal a notably higher TPC, even in comparison with other fruits [49,50]. Furthermore, previous studies have highlighted how mango peels contain a higher polyphenol content than other parts of the mango, including its pulp [51], indicating the potential of this by-product as a functional additive. MPP also showed substantial antiradical activity, with values of 70.26 mmol TEAC/100 g (DPPH assay) and 281.99 mmol TEAC/100 g (ABTS assay), highlighting the valuable functionality of this by-product. The TPC values obtained for the control bread (CTR) and mango-peel-powder-fortified bread samples (MPP-5, MPP-10) are reported in Table 6. Bread samples fortified with MPP showed very promising TPC values compared with control bread (CTR). Indeed, our results show that, in experimental bread samples (MPP-5, MPP-10), the addition of MPP increased the TPC, especially in those fortified at 10% (98.93 mg GAE/g). In a study by Pathak et al. [13], bread enriched with 5% MPP exhibited a TPC of 7.57 mg GAE/g. In contrast, our current study found that bread enriched with the same amount of MPP had a significantly higher TPC of 44.76 mg GAE/g, suggesting that experimental bread is a promising carrier for delivering antioxidant compounds. Additionally, in the evaluation of antiradical activity (Table 6), DPPH and ABTS values, expressed as mmol TEAC/100 g, were higher in all fortified bread samples compared with the control, according to percentage of fortification. Bread fortified with 10% of MPP showed values of 29.46 and 116.62 mmol TEAC/100 g for DPPH and ABTS, respectively, compared with the control values of 1.02 and 0.16 mmol TEAC/100 g for DPPH and ABTS, respectively.

Table 6. Antioxidant and antiradical activity of control and fortified bread samples.

Samples	TPC (mg GAE/g DM)	DPPH (mmol TEAC/100 g DM)	ABTS (mmol TEAC/100 g DM)
CTR	5.21 ^{a}	1.02 ^{a}	0.16 ^{a}
MPP-5	44.76 ^{b}	15.21 ^{b}	75.37 ^{b}
MPP-10	98.93 ^{c}	29.46 ^{c}	116.62 ^{c}
SEM	6.13	2.55	10.52
p value	<0.0001	<0.0001	<0.0001

Results indicate mean values of 6 determinations (carried out in triplicate for 2 independent productions). Abbreviations: TPC, total phenolic content; CTR, control bread; MPP-5 bread, experimental bread enriched with 5% (*w*/*w*) of mango peel powder (MPP); MPP-10 bread, experimental bread enriched with 10% (*w*/*w*) of MPP; SEM, standard error of the mean. Letters that are different within the same column indicate significant difference ($p < 0.05$).

Additionally, when compared with bread containing avocado waste powder [26], our study showed higher values for TPC, DPPH, and ABTS. The results from our analyses indicate that the antioxidant potential is improved by enriching bread with different percentages of MPP compared with the CTR bread. Overall, these results underscore the potential of bread fortified with MPP as a valuable vehicle for delivering bioactive compounds.

3.7 Volatile Organic Compounds Emitted from Breads Samples

VOCs were analysed in bread with and without MPP addition using the SPME–GC/ MS technique (Figure 1). In the control breads, a total of 18 compounds were identified, including four alcohols, six aldehydes, two ketones, and two acids. Short-chain alcohols and fatty acids were linked to sugar fermentation, while higher molecular weight alcohols were found to be associated with amino acid metabolism [52]. Across all breads, the most notable alcohols detected were isoamyl alcohol (3-methyl-1-butanol) and 2-phenylethanol. This observation is common in other studies [38,53–55]. Isoamyl alcohol provides balsamic, alcoholic, and malty notes to the bread [52,56]. Interestingly, this compound is commonly found in sourdough fermentation and is produced by various LAB groups, especially lactobacilli, leuconostocs, and weissellas [57]. Phenylethyl alcohol, known for its mild-warm and rose-honey-like odour, arises during fermentation or results from the Maillard reaction [58]. Another significant group in the bread's aromatic profile consists of carbonyl compounds, with hexanal and benzaldehyde being prominent constituents [53,55]. Hexanal, along with nonanal and pentanal, originates from lipid oxidation [59] and can contribute to a pleasant grassy odour [60]. Benzaldehyde, known for its bitter almond note [61], can derive from the metabolic breakdown or thermal degradation of phenylalanine [62]. Furan compounds arise from thermal reactions such as the Maillard reaction and caramelization [63]. These compounds contribute to the crust's typical odour notes. Similar VOC profiles have been reported by several authors in bread studies [38,53–55].

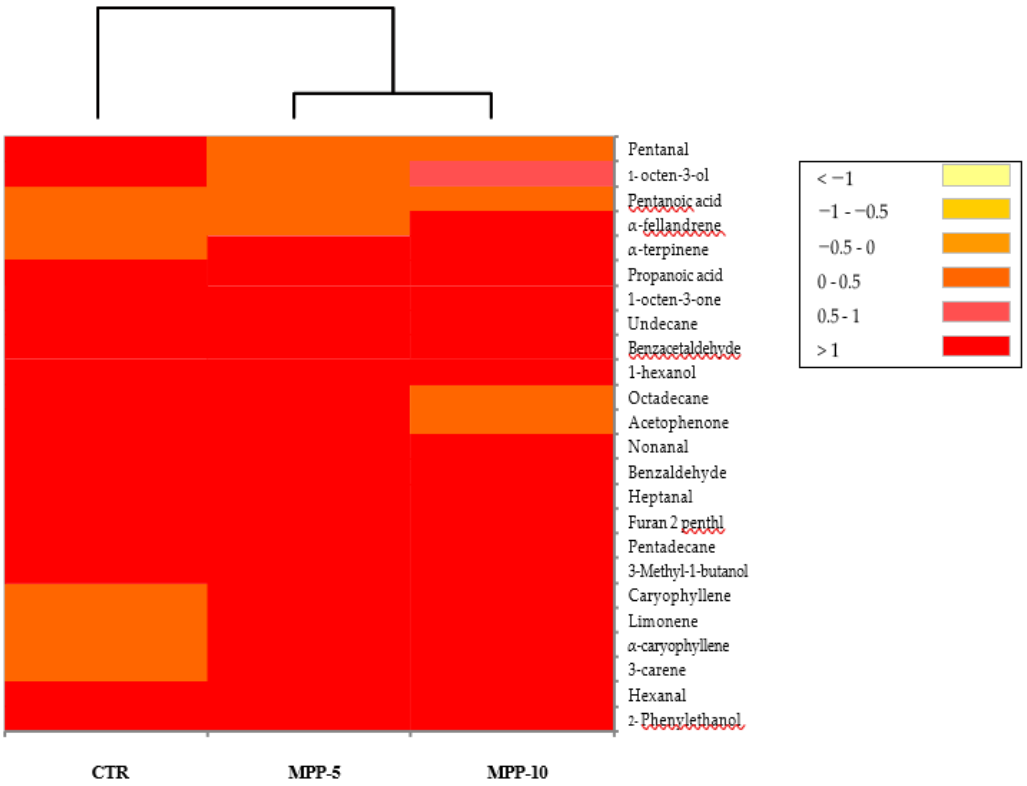

Figure 1. Distribution of volatile organic compounds emitted from breads. The heat map plot depicts the relative concentration of each VOC. Abbreviations: CTR, control bread; MPP-5, experimental bread enriched with 5% (*w*/*w*) of mango peel powder (MPP); MPP-10, experimental bread enriched with 10% (*w*/*w*) of MPP.

In bread samples with added MPP, several terpenes were identified. Specifically, monoterpenes such as 3-carene, limonene, α-terpinene, and α-fellandrene, as well as sesquiterpenes such as caryophyllene and α-caryophyllene, were present. Notably, these compounds were absent in the control samples, indicating that they specifically result from the addition of mango. Among these terpenes, 3-carene stood out as the most abundant. Mango is known for its elevated levels of d-3-carene, which typically constitute 60% and 80% of the total terpene content [64,65]. The flavour profiles of the breads were influenced by varying mango fruit percentages, resulting in higher proportions of terpenes and sesquiterpenes in the MPP-10 samples. Terpene compounds are known for their crucial role in enhancing aromatic perception [66]. Therefore, the presence of these compounds in the bread, due to the addition of MPP, indicates that mango significantly influenced the volatile profile of the breads. As previous research on various fruits and vegetables has demonstrated the ability of terpenes to enhance flavour perception and boost consumer preference [67–71], our study suggests that MPP enrichment could positively impact consumer acceptability of the bread [72].

3.8 Bread Sensory Attributes

A panel of 13 judges (eight women and five men, aged 23 to 40) conducted sensory analysis on CTR and MPP breads, and the results are graphically presented in Figure 2. Traits significantly different from the CTR bread included crust and crumb colour, aroma intensity, and taste persistency ($p < 0.05$). Except for alveolation size, bread odour, and crumb elasticity, all other traits received higher scores for the MPP breads compared with the CTR bread. In particular, the MPP-10 bread had the highest scores for crust and crumb colour, crust thickness, alveolation regularity, odour and aroma intensity, sweetness, acidity, and taste persistency. However, it is well known that the enrichment of cereal-based fermented products with fruit by-products strongly affects the sensory properties of the final products [26,37]. Overall, the MPP-10 bread received a higher score (6.51) than the CTR bread (5.6) and MPP-5 (6.11). MPP addition undoubtedly influenced sensory characteristics of the breads, with MPP-10 standing out in various sensory aspects.

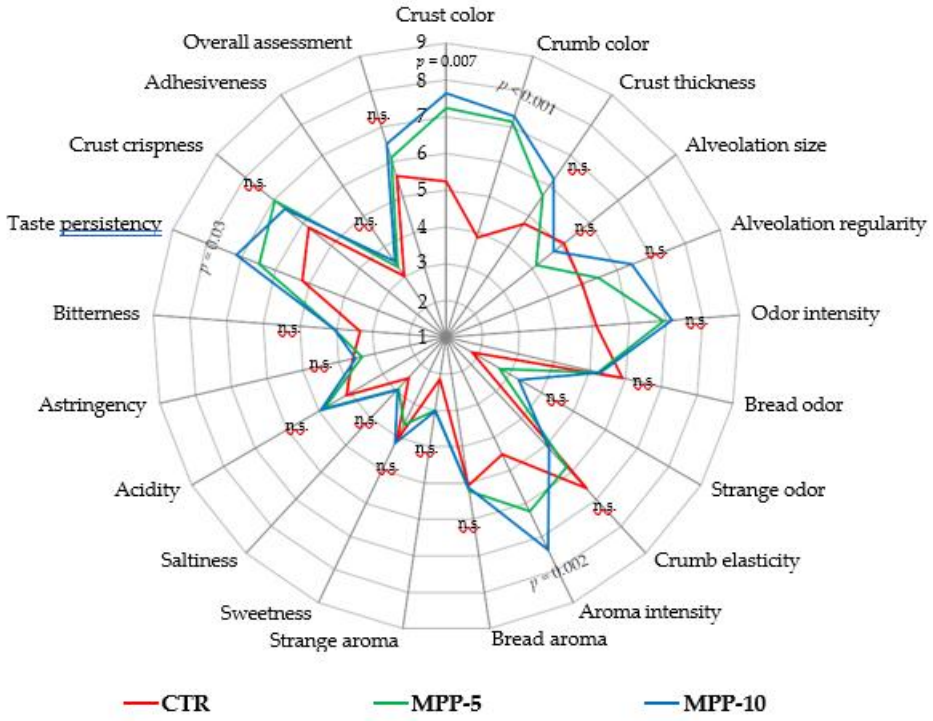

Figure 2. Radar chart of sensory analyses in breads. Statistical differences between doughs are indicated ($p < 0.05$). Abbreviations: CTR, control bread; MPP-5, experimental bread enriched with 5% (*w*/*w*) of mango peel powder (MPP); MPP-10, experimental bread enriched with 10% (*w*/*w*) of MPP; n.s., not significant.

4. Conclusions

This study highlights that MPP, as a by-product, is well suited for functional bread production, enhancing the nutritional profile, particularly in terms of antioxidants. There is a positive correlation between the proportion of added MPP and antioxidant content. The inclusion of 10% MPP in the dough significantly boosts the bread's antioxidant profile compared with the control bread. Furthermore, sensory evaluations revealed that the aroma

and colour of bread made with MPP are highly appreciated. In conclusion, enriched bread holds significant potential as a functional and sustainable staple for consumers.

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CHAPTER 8

General conclusions

The thesis followed a single goal of showing that combining conventional dehydration techniques with pre-treatment and proper post-process management lets you balance productivity, quality, and waste recovery in a 'sustainability-by-design' way. The experimental setup, organised by chapters/product, produced consistent and repeatable results.

On the model matrix (apricot), the combined use of pulsed electric fields (PEF) and air-born ultrasound (US) intensified mass transfer and substantially shortened drying kinetics (−30/−35% compared to HAD), with excellent adaptation of the finite plate diffusion model ($R^2 \geq 0.998$; %var ≥ 99.8; MRE < 0.13). The contributions of the two pre-treatments were found to be complementary. PEF acts mainly on D_{eff} with a quadratic trend as the pulses increase, while US also increases the surface exchange coefficient *k*. PEF×US interaction is statistically significant and explains the maximum efficiency observed in the combined protocols. This intensification did not come at the price of an indiscriminate loss of quality but revealed some design trade-offs. In terms of colour, PEF is the main driver. Increasing the intensity from 0 to 200 pulses reduces ΔE from 21 (HAD) to 6, values close to the threshold of perceptibility, while US alone does not bring significant benefits. Very strong combinations (PEF200+US) can even increase ΔE, suggesting an adverse mechanical effect when permeabilization is already high. For phenolic retention, the best compromise emerged with moderate PEF without ultrasound (PEF50-HAD), while US can promote leaching/oxidation of water-soluble antioxidants. However, total antioxidant capacity (FRAP) remains comparable to the control in PEF treatments. Dimensional deformations (shrinkage) showed dependence on PEF intensity in specific directions (TD and THICK significant; LD stable), with US not determining the final geometry. In summary, the identified operating windows allow for conscious modulation of productivity and final quality: PEF+US when time is the constraint; PEF200-HAD when visual appearance is the priority; PEF50-HAD when maximum phenolic retention is the goal.

The approach was transferred to other plant systems and matrices. On blood oranges (Moro, Tarocco, Sanguinello), treatment at 70 °C for 12 hours followed by modified atmosphere packaging ensured microbiological stability and sensory integrity for up to 100 days at 20 ± 5 °C; active MAP (100% N_2) preserved firmness and colour more

effectively than the passive regime, with perceived differences also in terms of aroma profile (HS-SPME) and a concrete intention to purchase by consumers (56% for the active MAP sample). These results confirm the role of the post-process "second barrier". Packaging atmosphere becomes an extension of the drying strategy, capable of protecting key VOCs and texture beyond the three-month threshold.

The same logic was valid for other seasonal species and formulations. In white-fleshed peach, the combination of drying and MAP (with a prevalence of N_2) limited browning and maintained mechanical and visual properties during storage, providing operational guidelines (peeling, thicknesses, gas mixtures) that can be transferred to an industrial scale.

The study on mangoes (var. *Tommy Atkins*) extended the 'process and post-process' paradigm, showing how the pre-harvest ripening stage and modified atmosphere packaging interact in determining the stability and acceptability of the dehydrated product. 'Green' and "Mature" samples dehydrated at 70 °C for 12 hours and packaged in active MAP (100% N_2) showed different behaviours: Green-N_2 samples maintained greater consistency and less dimensional collapse, while Mature samples better preserved colour (h*) and, after drying, polyphenol, mineral and vitamin (A, B1, B3, C) content, remaining microbiologically safe. On a sensory level, Mature-N_2 achieved a 40% purchase propensity, indicating that MAP active as a "second barrier" capable of protecting colour and texture beyond thermal stress; comparison with commercial samples also suggests better nutrient preservation in the absence of sulphites and with milder thermal regimes.

Finally, the thesis shifts the focus from the "object" to the "functional ingredient". Blackberry powder (BP), obtained by convective drying with or without ultrasonic assistance (US-HAD), is an effective carrier of polyphenols and an enhancer of antioxidant activity in baked goods. Ultrasonic pre-treatment accelerates drying and promotes the preservation/extraction of bioactive compounds compared to hot air alone. In fortified biscuits (10% BP), BP improves the phenolic profile with markers such as ellagic and protocatechuic acid and intensifies the fruity aroma without compromising workability and texture; the inevitable darkening of colour is consistent with the addition of anthocyanins and does not affect overall acceptability.

At the same time, mango peel powder (MPP), obtained by drying with a yield of 22%, proved to be a carrier of antioxidants and terpene aromas in complex fermented matrices. Enriching sourdough bread with 5% and 10% MPP increased the total phenolic

content (up to 99 mg GAE/g at 10%) and antiradical activity (DPPH/ABTS) in a dose-dependent manner; the volatile profile showed the appearance of monoterpenes (e.g. 3-carene, limonene) and sesquiterpenes that support aromatic intensity. Fermentation maintained a safe pH (4.14) and, sensorially, MPP-10 bread achieved the highest overall score (6.5 vs 5.6 for the control). To our knowledge, these are the first data on Italian sourdough breads with MPP, and they outline a concrete path of bio-reuse that transforms a waste product rich in fibre and polyphenols into an ingredient for high value-added baked goods. These applications shift the focus from "dried fruit" to functional ingredients and demonstrate that dehydration is also an enabler of the circular economy.

Methodologically, the thesis contributes with a factorial design that separates and quantifies the main and interaction effects of PEF and US on kinetics and quality, a validated diffusion framework (joint estimation of D_{eff} and k) that returns physically consistent parameters along even combined protocols, and a micro-macro reading of the tissue response (link between electrical permeabilization, reduction of internal/external resistance and selective dimensional collapse), useful for the "design" of target textures and colours. The consistency of the results between different matrices and the convergence between instrumental and sensory indicators reinforce the robustness of the conclusions and their transferability throughout the supply chain.

Overall, the research shows that integrating non-thermal pre-treatments with air drying and targeted post-process packaging is a viable way to reduce processing time and energy without compromising quality, preserving nutrients and critical sensory attributes (colour, texture, aromas) and upgrading by-products and off-calibre batches into functional ingredients. This triple result of efficiency, quality and circularity provides a solid technical basis for industrial adoption and positions "mild" dehydration as a strategic component of a more resilient, competitive and sustainable fruit supply chain.

CHAPTER 9

Future prospects

In consideration of the results obtained, the next step is the continuous transposition of the most promising configurations, such as PEF200-HAD when the constraint is colour stability, PEF50-HAD to maximise phenolic retention, and PEFxUS when productivity is the primary requirement, using industrial prototypes capable of ensuring uniform treatment on heterogeneous matrices. This involves the design of PEF and US protocols that can be integrated on conveyor belts and vibrating beds, the verification of the field distribution and intensity of the ultrasounds on real batches, energy balances including heat recovery and active dehumidification, as well as the definition of robust design spaces that maintain quality as cultivars, thicknesses and initial water content vary.

At the same time, digital control will have to evolve towards multi-objective optimisation, coupling in-line sensors (NIR/hyperspectral spectra for moisture and colour, microbalances for mass flows, thermography for thermal profiles) with digital twins and predictive control schemes capable of orchestrating time, energy, quality and microbiological safety. Data-driven models, ideally constrained by physical laws, will be able to update diffusion and exchange parameters during operation, preventing process drifts and reducing waste.

In terms of sustainability and cost-effectiveness, LCA and techno-economic assessments will be decisive. In addition to the carbon footprint, these assessments will consider water consumption, cost per kilogram of water removed, cost per point of ΔE avoided and per milligram of polyphenols preserved, including the systemic benefits deriving from the reduction of organic waste and the valorisation of by-products. With a view to low thermal impact “hurdle technology”, the exploration of PEF/US combinations with UV-C, cold plasma, low-dose ozone or short wet tempering phases could enhance safety and oxidative stability; however, it will be necessary to map sequencing, synergies and possible reactive intermediates. A mechanistic approach to microstructure, using μCT, SEM, confocal and MRI, will make it possible to link porosity, tortuosity and dimensional collapse to electro-acoustic parameters, with implications for the design of target fabrics and the prediction of rehydration in domestic or industrial use.

Another area concerns the eco-design of packaging. Tailor-made gas mixtures and low-impact barrier materials will need to interact with active systems (oxygen and moisture absorbers, ethylene scavengers) and with “dynamic” MAP strategies capable of

modulating the gas profile during storage using smart sensors, to preserve key VOCs and texture. Validation on different cultivars and stages of ripeness will require bio accessibility and bioavailability testing of bioactive compounds using static and dynamic in vitro models, non-targeted metabolomics to capture secondary effects on phenolic and aromatic pathways, and studies with consumers in real-world contexts to translate instrumental indicators into purchasing preferences.

The industrial transfer will involve the standardisation of operating procedures for PEF/US, drying and MAP, shared acceptance metrics (chromatic, phenolic, antioxidant, microbiological), labelling specifications (absence of sulphites, traceability and origin of by-products), and health and hygiene criteria including limits on contaminants and residues. In this context, the circular "ingredientisation" of mango peel powder (MPP) and blackberry powder (BP) will require quality and oxidative stability standards, study of the impact on rheology and fermentation in baking (gluten network, specific volume, gas retention, acid-lactic kinetics), microencapsulation or co-processing strategies with fibres to protect terpenes and anthocyanins, as well as supply chain and business models that ensure supply, sanitation and batch homogeneity. Rapid consumer science methodologies and predictive liking models will allow physical-chemical descriptors to be linked to market response, while nutritional personalisation will guide recipes and processes towards specific targets for fibre, glycaemic index and antioxidant profile.

Finally, supply chain resilience can be strengthened by placing PEF-US-HAD modules upstream, at collection centres, to quickly stabilise seasonal surpluses. Dedicated logistics for by-products and supply agreements that reward the quality of the material destined for functional ingredients will complete the architecture. In this process, the vision of sustainability-by-design becomes operational: from phenomenological optimisation in the laboratory to predictive control on the plant, to regulatory standardisation and circular valorisation, with measurable reductions in waste, energy consumption and nutritional loss, and the creation of new markets for high value-added ingredients.