

Evaluation of drying and extraction conditions to produce a high value flavoring ingredient from *Physalis peruviana*

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Abstract

Colombia is recognized as the world's leading exporter of *Physalis peruviana*, commonly known as Cape gooseberry (CG), which is recognized for its "exotic" organoleptic characteristics (color, flavor and aroma). This project aimed to evaluate different drying techniques and high intensity ultrasound assisted extraction (UAE) methods to obtain a flavoring ingredient that resembles the flavor of fresh CG. To achieve this, fruits were classified within a range of 12- 13°Brix and subjected to convective drying, freeze-drying and fluidized bed drying. UAE of dried fruits was evaluated using a factorial design where the concentration of ethanol and amplitude were modified. The variable response in each treatment was the volatile composition found on material which was analyzed by HS-SPME-GC/MS, methodology optimized under a Box-Behnken design. The results indicated that after drying, fluidized bed drying (FBD) was the treatment that better conserved the volatile composition when compared to the total initial compound in fresh fruits. Dried fruits obtained by FBD were used in UAE and the final extract was rotary evaporated and lyophilized to get a prototype of ingredient which did not conserve most of the volatile compounds that were present in fresh *Physalis peruviana* that could be associated with desired organoleptic properties, especially those classified as esters and terpenes. Likewise, this productive process could be considered as a starting point to obtain and characterize flavoring ingredients from other fruits considered exotic and that are of interest in national and international markets.

*Keywords***:** *Physalis peruviana fruits,* drying process, ultrasound assisted extraction, volatile compounds, HS-SPME-GC/MS, flavoring ingredient.

Introduction

Physalis peruviana is a native species from the Andes area that can grow in a wide range of altitudes. This herbaceous plant has a characteristic fruit that is known as Uchuva, Uvilla, Golden Berry, which is a globular berry (1.25-2.50 cm in diameter, 4-10 g of weight) with an inflated calyx, an orange-yellow skin and a sweet, aromatic and juicy pulp (Lanchero et al., 2007; Puente et al., 2011). *Physalis peruviana* fruits contains vitamins (C, E, B6, B3, K1), phenolic compounds, carotenoids (β-Carotene), phytosterols (Campesterol, β-Sitosterol, Δ5-Avenasterol, Lanosterol, Stigmasterol and Δ7-Avenasterol ergosterol), minerals (Ca, Fe, Mn, Mg, Zn), physalins, withanolides (Olivares-Tenorio et al., 2016) among others. These chemical and nutritional components provide fruits with medicinal properties including antispasmodic, diuretic, analgesic, sedative, parasiticidal, antidiabetic, antioxidant, anti-inflammatory, antimicrobial, optic nerve strengthening effects, and hypoglycemic (Kupska & Jeleń, 2017; Majcher et al., 2020; Puente et al., 2011).

This specie is not only known for its medicinal properties but also for its color, flavor and odor, this leads to it being considered an "exotic fruit". Different countries are engaged in the production of this fruit with Colombia being one of them. *Physalis peruviana* specie that grows in Colombia is characterized by its quality, size, color, and healthy and clean appearance, factors that make it competitive in international markets. Accordingly, by 2020, Colombia had a cultivated area of 1311 hectares in different departments such as Boyacá, Antioquia, Cundinamarca, Cauca, Nariño, Norte de Santander and Santander with a production of 16.377 tons of raw material; Colombian agroclimatic conditions allows to have fresh fruits throughout the year. In 2015, Colombia exported 5200 tons and in 2022 exported 8500 tons of raw material, repositioning Colombia as the world's leading exporter of fresh *Physalis peruviana* (ICA, 2022; Procolombia, 2021a). Additionally, exports have been expanding into new markets, reaching a total of 53 destinations worldwide such as Azerbaijan, Brazil, Canada, China, Chile, EEUU, Germany, Italy, Malaysia, Mexico, Russia, Spain, Saudi Arabia, Ukraine, United Kingdom, etc. Nevertheless, not all the national production is exported and therefore this vegetable material remains in the domestic market. The exotic flavor and odor of *Physalis peruviana* is generated by the presence of esters, aromatic ethers, aromatic hydrocarbons, alcohols, aldehydes, lactones, ketones, terpenes, terpenoid, saturated fatty acids and derivatives (Beema et al., 2017; Dymerski et al., 2015; Granados Pérez et al., 2019; Gutiérrez et

al., 2010; Kupska & Jeleń, 2017; Majcher et al., 2020; Popova et al., 2022; Yilmaztekin, 2014b). In relation to the foregoing, Colombia has raw material that has been exported, generating an opportunity that allows could be explored to give some additional value throughout engineer process to obtain ingredients or byproducts with the organoleptic properties of *Physalis peruviana*, which could be used in other food matrices such as juices, nectars, yogurts or powdered as food additives and participate in a market that generated income of 7818 million dollars in 2022 (Conway, 2023).

To obtain an ingredient, it must be consider different process steps, such as preparation, where initial characteristics of the fruits like color, acidity, microbiological content, weight or soluble solids content in the fruit are described to classify ripeness state of the raw vegetal material (Bravo & Osorio, 2015; Etzbach et al., 2018; ICONTEC, 1999; Olivares-Tenorio et al., 2016). On the other hand, drying is considered as a conservation step due to its wide use to preserve harvested foods facilitating handling and packaging, reducing weight during transportation, improving processes like grinding and mixing, extending shelf life and enhancing stability during storage. Drying process can even affect the volatile and non-volatile chemistry of the fruits, however, this could be avoid or diminished by the implementation of diverse drying techniques such as convective drying, fluidized bed drying or freeze-drying (Junqueira et al., 2017; Mujumdar, 2014; Nawirska-Olszańska et al., 2017; Puente et al., 2011). Another step involves the selective separation of the desired components through solid-liquid extraction (Bitwell et al., 2023), that can be optimized by high intensity ultrasound since cavitation phenomena can cause surface peeling, erosion, sonoporation and permeabilization of cell walls, leading to better extraction of bioactive compounds (Perera & Alzahrani, 2021).

The present work aimed to establish the process conditions to obtain a prototype of a high value-added flavoring ingredient from *Physalis peruviana* fruits, which guarantee the preservation of its organoleptic properties related to the volatile chemical composition. To achieve this, the volatile composition of the fresh fruit was settled and compared with material obtained from three drying techniques (convective drying, fluidized bed drying, and freeze-drying). Subsequently, the dried material with the highest preservation value underwent high intensity ultrasound assisted extraction (ethanol-water) and the profile of volatiles of the final extract was also compared with the fresh material profile. In this way, it was correlated how each treatment employed affected the volatile chemical composition of the fresh fruit to finally obtain a prototype of freeze-dried

ingredient which preserved a certain volatile proportion identified in the fresh material that could be related to sensory perception.

1 Problem statement

Physalis peruviana, also known as Cape gooseberry or uchuva in Colombia, is a native plant from South America or Andean region. It has been extensively studied, particularly with a focus on Colombian agronomical varieties (Álvarez-Herrera et al., 2021; Fischer, G. Herrera, A. Almanza, 2011; Fischer & Melgarejo, 2020; Herrera et al., 2012; Lanchero et al., 2007; Ramírez et al., 2013). Its fruit is ovoid and its color varies from green to orange according to its ripeness. Based on Colombian Technical Standard (NTC) 4580, *Physalis peruviana* fruit is classified into seventh ranges according to its color, soluble solids content and titratable acidity. Apart from that, it has been shown that volatile and non-volatile chemistry are influenced by the ripeness state (Olivares-Tenorio et al., 2016). Total phenolic compounds and antioxidant activity also vary understanding to the ripeness state as outlined in NTC 4580, however, it did not completely clear how they change through the maturation process since previous reports assert that it decreased conformance to the ripeness stage (Bravo & Osorio, 2015). Valdenegro et al., 2013 propose that it did not vary but Severo et al., 2010 and Mier et al., 2011 confirm that it increased. As published by Bravo et al. 2015, the content of other type of metabolites such as β-Carotene and Ascorbic acid increases depending on different cultivars of *Physalis peruviana*, but the inhibition of oxidation and radical scavenging activity decreased with ripeness stage (Bravo & Osorio, 2015).

Not only ripening has an impact on chemical composition or bioactivity of fruits like *Physalis peruviana*, handling, exposure to sunlight, storage and transporting compromise them. Taking the Olivares-Tenorio et al. 2017 research, they evaluated in *Physalis peruviana* their content of Ascorbic acid, β-Carotene and flavonoids like catechin and epicatechin, They confirm that those components had not thermal stability when they were exposed to different temperatures $(40, 60, 80, 80, 120 \degree C)$ (Olivares-Tenorio et al., 2017). They concluded that at temperatures above 60°C the concentration of these substances decreased at a slower rate, even the concentration of flavonoids increases at lower temperatures.

Aroma and flavor represent two of the most crucial attributes influencing the consumption of fruits and both qualitative and quantitative values are essential for characterizing aroma production. A bibliometric analysis revealed that 317 components are related to the volatile substances of CG, which includes acids, alcohols, aldehydes, alkanes, alkenes, diterpenes,

diterpenoids, esters, ethers, furanones, ionones, ketones, lactones, monoterpenes, monoterpenoid oxides, phenols, phytol derivatives, pyrazines, sesquiterpenes, steroids, sterols, terpenes and terpenoids (Ballesteros et al., 2019; Beema et al., 2017; Berger et al., 1989; Dymerski et al., 2015; Gutiérrez et al., 2010; Kupska & Jeleń, 2017; Yilmaztekin, 2014b). The presence of these compounds depends on CG origin and some of them could vary according to the genotype. The volatile components are commonly identified and quantified using techniques such as liquid or gas chromatography coupled to a mass spectrometer. Other techniques are engaged in the analysis process and each of them have advantages or specific applications, they include headspace solid phase microextraction (HS-SPME), solvent assisted flavor evaporation (SAFE), gas chromatography-olfactometry (GC-O), in-tube extraction (ITEX), electrospray ionization ion trap multiple mass spectrometric (ESI-MS/MS) (Majcher et al., 2020; Mayorga et al., 2001; Popova et al., 2022; Yilmaztekin, 2014b).

Notwithstanding these versatile tools, additional evaluation such as sensory tests could complement organoleptic description of fruits like CG with the purpose of getting the real consumer perception of the flavor, aroma, color and texture. According to NTC 3929, sensory analysis is defined as the science related to the evaluation of the attributes of a product that are perceived by the sensory organs and in relation to a person's experience. In other words, the object under analysis can be described based on the psychophysiological reaction resulting from sensory stimulation. This standard proposes an evaluation on an intensity scale from 1 to 5, either for taste or smell. In previous inspections, CG from different departments of Colombia were submitted to a sensory analysis where five expert panelists identified seven attributes related to taste (acidic, sweet, bitter, fruity, citrus, tree tomato and Lulo) and six attributes related to odor (fruity, citrus, Lulo, tangerine, tree tomato and floral) (Quintero et al., 2012). The conclusion was that "the flavor of uchuva consists of fruity, floral and citrus notes, attributes associated with tropical Colombia fruits". Sensory results can be explained by a potential relationship with the volatile composition when substances such as Butyl acetate, Limonene, Terpinolene, Ethyl octanoate and Ethyl acetate are highlighted as the aromatic notes present in these fruits. Another sensory analysis of Colombia imported CG material performed by ten expert panelists, showed the identification of intense notes of caramel, cotton candy and weak notes of vanilla, melon, citrus and gooseberry (Majcher et al., 2020).

Sensory attributes postulate *Physalis peruviana* as an attractive source of functional products such as flavoring ingredients that could be incorporated into food production (Erkaya et al., 2012; Etzbach et al., 2020). For this purpose, some treatments should be applied to fresh fruits of CG since, in most of the cases, they must be preserved and storage to guarantee a ripening stage that offers the required organoleptic properties. Drying is a preservation method widely studied and applied in different materials such as fruits (Mujumdar, 2014). Drying techniques have been employed to preserve CG, including convective drying, freeze drying, fluidized bed drying, microwave drying, infrared drying and refractive window drying (Karama et al., 2016; Mellor & Bell, 2003; Patra et al., 2022; Puente et al., 2020) to evaluate their final effect in color, hardness, adhesiveness, cohesiveness and gumminess properties (Junqueira et al., 2017; Vásquez-Parra et al., 2013). Additionally, they are usful to model drying kinetics using mathematical models like Newton, Page, Logarithmic, Wang & Singh or Henderson & Pabis (Junqueira et al., 2017). Some studies have been focused in polyphenols content, antioxidant activity, total carotenoids (all-translutein, β-Kryptoxanthin, α-Carotene, all-trans-β-Carotene and 15-cis-β-Carotene), fatty acid composition and amino acids (Nawirska-Olszańska et al., 2017; Puente et al., 2011) and results suggest precaution during handling this fruit avoiding exposure to oxygen, moisture, light and high temperatures (Puente et al., 2020).

Few studies have been conducted with GC under ultrasound treatment. One of them is related with carotenoids, where raw fruits were submitted to ultrasonication extraction and compared with untreated CG, the results did not show significant effect on the total extraction of carotenoids and not correlation was observed between the total carotenoid content and the amplitude, process time and temperature. On the other hand, Ordoñez et al. 2017 studied the effect of ultrasound on CG properties such as color, vitamin C, total phenols and also carotenoids. As a result, ultrasound did not induce any change in the pH, titratable acidity and total soluble solids. However, it was also observed that with increasing sonification time, ascorbic acid decreases, in contrast to total phenolics and some carotenoids that increase.

Based on the above mentioned, Colombia has a wide diversity and availability of fruits, among these is *Physalis peruviana* which is not only attractive for its nutritional composition also for its exotic flavor and smell, which is made up of volatile components such as esters, terpenes, ketones and others. Colombia has a production of approximately 16000 tons of these fruits, but about half of this value is not exported, since not all fruits meet certain chemical or physical characteristics to be exported. These fruits are left for national consumption or transformation, which generates an opportunity to take advantage of this highly attractive fruit and the possible creation of a product or ingredient that captures and preserves the volatile chemistry that represents the specific flavor or smell of this fruit. To achieve this, a scalable industrial process must be established, which preserves and extracts certain components of interest, in addition to evaluating how each stage of this process affects the initial volatile chemistry of the material and thus be able to generate a value-added product such as a flavoring that can be used in other food matrices.

2 Justification

Colombia is the world's leading producer and exporter of *Physalis peruviana* since in 2022 it exported 8.541 tons of fresh fruits to 17 countries by \$38.2 million USD FOB (Free on Board), mainly due to its nutritional content and exotic flavor (ANALDEX, 2022). On the other hand, Colombia has an exportation attention on fresh products since 2020, its contribution to the gross domestic product (GDP) was \$15.1 billion dollars in the category of "Agriculture, livestock, hunting, forestry, and fishing", higher than the category of "Manufacture of food products; manufacture of beverages; manufacture of tobacco products" with a value of \$7.8 billion dollars (Procolombia, 2021b). The food industry is the driver of the economic growth of the region and the entire country, when it interacts with the branches of agriculture, wholesale and retail trade, transport and logistics system, it has a synergetic effect on the economy (Glinskiy et al., 2018). Therefore, this sector serves as an economic engine by enhancing value to raw materials since it requires infrastructure, industrial services and qualified personnel to develop technology and innovation. Finally, this involves the generation of products that can be used or added in the manufacturing of other foods or beverages, such as natural flavorings.

Organoleptic properties of fruits are related to volatile (terpenes, esters, alcohols, aldehydes, etc.) and non-volatile components (phenolic compounds, sugars, etc.), playing a role in imparting distinct aroma, colors and flavor according to the specie (Arshamian et al., 2017). The conservation of those elements can promote final acceptance by the consumer, as their perspective is influenced by microbiological, physical, chemical and biochemical changes that, from their point of view, determine their consumption (Olivares-Tenorio et al., 2017). In order to extendend shel life or reduce the volume of different foods, process like drying are commonly applied, however, they could be improved or developed to minimize any significant alteration to their chemical composition with the purpose of assure their sensory, nutritional or medicinal properties (Oliver-Simancas et al., 2020; Onwude et al., 2022).

This study aimed to evaluate a drying and extraction process applied to Physalis *peruviana*, seeking preservation of the aroma, flavor and color to finally create a flavoring ingredient that resembles the specific organoleptic characteristics of a Colombian variety of *Physalis peruviana*, which could be used as an essential component of other foods or as a final product available to immediate consume. In the same way, findings can set a precedent for a potential applicability to other exotic local fruits such as passion fruit, lulo, gulupa, among others.

3 Objectives

3.1 General objective

To establish the process conditions for *Physalis peruviana* fruit that guarantee the preservation of its organoleptic properties and the volatile chemical composition necessary for the generation of high value-added flavoring ingredients.

3.2 Specific objectives

3.2.1. To establish the drying conditions of ripe fruit of *Physalis peruviana* by evaluating different drying techniques including fluidized bed, convective and freeze drying

3.2.2. To develop a process of cold extraction by high intensity ultrasound in dried *Physalis peruviana* that allows concentrating the characteristic compounds associated with flavor, aroma and color.

3.2.3. To formulate a prototype flavoring ingredient from an extract of *Physalis peruviana* with organoleptic characteristics and volatile chemical composition of interest*.*

4 Hypothesis

4.1 Working hyphotesis

The integration of a drying technique like convective drying, lyophilization, bed fluidized drying with high-intensity ultrasound-assisted extraction might be used in a process to obtain a flavoring ingredient that conserve the fresh *Physalis peruviana* flavor.

4.1.1 Nully hyphotesis

 H_o : It is not possible to establish a drying and extraction process that preserves and allows to create a flavoring ingredient that possesses similar sensory properties of the fresh *Physalis peruviana*.

4.1.2 Alternative hyphotesis

 H_1 : It is possible to establish a drying and extraction process that preserves and allows to create a flavoring ingredient that possesses similar sensory properties of the fresh *Physalis peruviana*.

4.1.3 Variables

Independent variables: for drying methods: convective drying, freeze drying and bed fluidized drying. For ultrasound assisted extraction method: ethanol concentration as solvent and wave amplitude level.

Dependent variables: presence of volatile compounds, concentration of identifiable compounds ((-)-β-Pinene, 1R-α-Pinene and Eucalyptol).

5 Theoretical framework

5.1 Exotic fruits in Colombia

A wide variety of exotic fruits are produced in Colombia with a constant demand in international markets. Due to its geographical location, rich soil and extended periods of light, it leads to fruit harvests throughout the year. This enabled Colombia to establish itself in 2019 as the leading exporter of exotic fruits in Latin America and the ninth country in the world with fruits like Passion fruit, Gulupa, Cape gooseberry, Dragon fruit, Mangosteen, Borojó, Banana, Lulo, Medlar and Guanabana (ANALDEX, 2022). This export sector has been steadily growing since 2014 with sales reaching 80.7 million dollars in 2021. Furthermore, it has expanded into new markets in 2020, reaching a total of 53 diverse destinations such as Azerbaijan, Brazil, Canada, China, Chile, EEUU, Germany, Italy, Malaysia, Mexico, Russia, Spain, Saudi Arabia, Ukraine and the United Kingdom. According to figure 1 both the exports and production have been increasing in Colombia since 2016, despite a market setback in 2020 due to COVID-19. (ANALDEX, 2022; Procolombia, 2021b). The effort made by government entities such as Ministry of Agriculture and Rural Development, the Ministry of Commerce, and associations like the Colombian Agricultural Institute (ICA), *Physalis peruviana* Exporters Committee, Procolombia, Asohofrucol, Agrosavia, among others, has made progress in terms of the eligibility and export of *Physalis peruviana* to different countries. In 2022, *Physalis peruviana* production increased from 7.872 to 8.541 tons and exports from 37.8 USD million to 38.2 USD million. In addition, the U.S. market is one of the most prominent with exports of 3.9 USD million in 2020 to 4.9 USD million in 2021 (ANALDEX, 2022; ICA, 2022).

Source: ANALDEX, 2022.

Additionally, *Physalis peruviana* production has one of the greatest economic and social impacts in the country, not only due to its importance for food security but also because of its demand in international markets. While the economic aspect is of great relevance to Colombia, 45% of the costs related to *Physalis peruviana* cultivation and harvesting are attributed to labor, especially by female heads of households; this contributes to rural development and strengthens family economies. *P. peruviana* production come from various regions of Colombia, table 5.1.1, where Antioquia is the largest producer followed by Cundinamarca, which is an indicator of economic dynamism for rural region in Antioquia since It includes municipalities like Yarumal, Abejorral, Sonsón and Rionegro (MasColombia, 2021; Procolombia, 2021b).

Table 1 Production of P. peruviana in Colombia in 2019

5.2 Physalis peruviana

5*.2.1* **Agronomical chracteristics**

P. peruviana L., commonly known as Cape gooseberry, Goldenberry or Uchuva, is a native plant of the Andes region that belongs to the Solanaceae family. It is herbaceous, perennial and semi-shrubby, It has a straight and sparsely branched, the stem can grow between 0.6 and 0.9 meters. The leaves are alternate, petiolate, densely pubescent, its flowers are yellow with five purple spots, slightly inclined or erect and can be easily pollinated by insects, wind and selfpollination (Dostert et al., 2013; Puente et al., 2011; Yilmaztekin, 2014a). Fruits are berries with 1.25 to 2.5 cm in diameter and weighing 4 to 10 grams. They are orange when ripe and in the peduncle region, the fruits are lighter and more yellow than in other areas due to the lower concentration of carotenoids and changes in the ripening process, figure 2. They are completely covered by the calyx, a bladder-like organ that protects them from insects, birds, diseases and adverse weather conditions. The calyx is small at the beginning of the fruit development, after the flower falls, it expands, ultimately forming a straw-colored husk much larger (5 cm) (Puente et al., 2011).

Figure 2 Anathomy of Physalis peruviana L. (A) plant, (B) calyx, (C) flower and (D) fruits.

Source: (Olivares Tenorio, 2017).

P. peruviana can grow at different altitudes above sea level, ranging from 1500 to 3000 meters. It can withstand low temperatures but can suffer irreversible damage at temperatures below 0° C and its growth is affected at temperatures below 10 $^{\circ}$ C. High temperatures can also affect its flowering and fruiting, the optimal growth temperature is 18°C. Furthermore, It can thrive in poor but well-drained and slightly acidic soils, It has low fertilization requirements and cannot tolerate clayey soils due to its shallow roots (Fischer, G. Herrera, A. Almanza, 2011). The harvest begins seven to nine months after planting and the fruits are harvestable approximately 60 to 80 days after the development stage when the calyx changes color from green to brown. Additionally, it has been classified into ecotypes, Colombian type is characterized by having small fruits with vivid color and higher sugar content (Ramírez et al., 2013).

5.2.2 Uses and properties

Although most of the fruit of *Physalis Peruviana L*. is exported as fresh fruit, the raw material is useful to produce numerous products such as juices, syrups, raisin, candies, pomaces, sauces, jams, jellies and snacks which are currently available in the domestic and international markets. This fruit contains a source of a variety of bioactive compounds that are present in the pulp, seeds and peel, which has potential health benefits that make the fruit an option for the development of phytotherapeutics with activity against various diseases (Ramadan, 2011).

Different medicinal properties have been attributed, including antispasmodic, diuretic, analgesic, sedative, parasiticidal, antidiabetic, optic nerve strengthening effects, among others (Kupska & Jeleń, 2017). Traditionally in Colombia, It has been used to purify the blood, reduce albumin, eliminate cataracts, calcify and control amoebiasis. In Peru, It is empirically used to treat cancer, asthma, malaria, dermatitis and hepatitis (J. Wu et al., 2021). Although no adverse effects have been demonstrated from consuming the fruit, some studies indicated that eating CG fruit lowers blood glucose levels, causing a hypoglycemic effect, which could pose a risk to populations with conditions associated with this pathology. In addition to the fruit, the calyx is also used in traditional medicine due to its anticancer, anti-inflammatory, immunomodulatory, antimicrobial, diuretic, and antipyretic properties (Majcher et al., 2020; Puente et al., 2011; Ramadan, 2011). In this way, the potential health benefits of CG are associated with the presence of phytochemicals in different ethanolic, ethyl acetate, water: methanol: acetonitrile extracts which can contain chemical

compounds such as withanolides, physalins, vitamin C, flavonoids, among others. The main mechanism of these potential health benefits is claimed to be the radical scavenging activity of health promoting compounds that can maintain an adequate level of non-enzymatic and enzymatic antioxidant defense. Nevertheless, mechanisms are not yet clear (Olivares-Tenorio et al., 2017).

Diverse traditional uses have been confirmed through in vitro studies in rats through the evaluation of CG extracts. For example, extracts of the leaves in water, ethanol, and hexane from *P. peruviana* showed an effect against induced hepatotoxicity. Other extracts obtained with $CO₂$ showed prevention of cell cytotoxicity induced by lipopolysaccharide (LPS) in murine macrophages, inhibition of the induction of inducible nitric oxide synthase by LPS, and inhibition of 2-Cyclooxygenase expression, imparting an anti-inflammatory effect (Ramadan, 2011). The ethanol extract of the plant exhibited a superior antioxidant effect compared to α -tocopherol (S. J. Wu et al., 2005). Its consumption has also demonstrated effects on blood coagulation, bone metabolism, heart diseases, dermatological conditions, due to the high levels of vitamin K_1 present in *P. peruviana* oil, which serves as a coenzyme, participates in the synthesis of various proteins, reduces the risk of heart disease, kills cancer cells and improves skin health. The oil also has a high content of linoleic acid that has been associated with the prevention of cardiovascular diseases and is involved in the composition of plasma membranes (Ramadan, 2011). El-Akad et al, 2022 obtained an extract with water: methanol:acetonitrile, 50:25:25 V/V and it had the capacity to extract metabolites such as organic acids, phenolic and phenylpropanoid derivatives, flavonoids, coumarins, alkaloids, withanolides and fatty acids, all of which possess anti-arthritic activity (El-Akad et al., 2022) Ethyl acetate extract showed the highest α-Amylase (200.52 \pm 0.87 μg/ml), β-Glucosidase (25.19 \pm 2.65 µg/ml) and lipase concentration (22.52 \pm 1.43µg/ml), which evaluated in an induction of diabetes in rats by Streptozotocin and showed a decrease in the blood glucose level (Nowak & Jakubczyk, 2020).

5.2.3 Chemical composition

Physalis peruviana fruits exhibits an organoleptic and nutritional quality thanks to their solids soluble content, acidity (Marin et al., 2010), fiber, protein, lipid, carbohydrates (Corrales-Bernal et al., 2015), ash, calcium, phosphorus (Álvarez-Herrera et al., 2014), phenolic content (Bravo & Osorio, 2015), ascorbic acid, riboflavin, citric acid, tartaric acid and malic acid (Palomino, 2010; Puente et al., 2011).

In relation to nutritional composition, the fruit has shown a presence of approximately 31.8% of essential amino acids, mainly leucine, lysine and isoleucine (Puente et al., 2011). Among the main carbohydrates, sucrose, glucose and fructose are the main. Around 2% of oil can be found in the fruit, where 1.8% is present in the seeds and 0.2% in the pulp and skin. This oil contains approximately 15 fatty acids, with linoleic acid and oleic acid being the main ones. There is also the presence of saturated fatty acids such as palmitic acid and stearic acid, as well as polyunsaturated fatty acids like γ-Linolenic acid, α-Linolenic acid and Dihomo-γ-linolenic acid. The oil has high levels of phytosterols such as Campesterol, β-Stosterol, Stigmasterol, Δ5- Avenasterol, Lanosterol, Δ7-Avenasterol and Ergosterol. Campesterol, Δ5-Avenasterol and Argosterol are the most abundant in the fruit pulp (Puente et al., 2011; M. F. Ramadan, 2011). The high levels of vitamin A, B (thiamine, riboflavin, niacin) and C (Ascorbic acid) also make *Physalis peruviana* fruits highly nutritious. The concentration of Ascorbic acid (46 mg/100 g) in Cape gooseberry is higher than that of pear $(4 \text{ mg}/100 \text{ g})$, apple $(6 \text{ mg}/100 \text{ g})$, peach $(7 \text{ mg}/100 \text{ g})$, and comparable to that of orange $(50 \text{ mg}/100 \text{ g})$ and strawberry $(60 \text{ mg}/100 \text{ g})$. The main active components of vitamin A are α-Carotene, β-Carotene and β-Cryptoxanthin which are also responsible for the orange color of the fruit. Vitamin K₁ (Phylloquinone) and vitamin E (α -Tocopherol, β-Tocopherol, γ-Tocopherol and δ-Tocopherol) are present in the fruit's oil; vitamin E levels in the pulp are high when compared to those in the seeds. The level of vitamin K_1 is considered high, representing more than 0.2% of the total lipids in the pulp oil, which is significant because the level of this vitamin in most foods that contain it is low $\left($ < 10 mg/100 g), and most of it is obtained from some green vegetables and leaves like spinach (Puente et al., 2011; Ramadan, 2011).

The epicarp of the fruit contains a waxy film primarily composed of terpenes and pectins (Ramadan, 2011). The phenolic content, which includes quercetin, rutin, myricetin, epicatechin,

catechin and kaempferol, may differ between varieties, cultivars, ecotypes or maturity stage (Bravo et al., 2015). Furthermore, the presence of withanolides, which are a group of steroidal lactones which have been isolated from the genera *Acnistus, Datura, Jaborosa, Lycium, Physalis* and *Withania* of the family Solanaceae (Ramadan, 2011). Those components have antimicrobial properties, antitumor, anti-inflammatory, hepatoprotective or immunomodulatory and antiparasitic activity. Additionally, thirty withanolides have been found in *Physalis peruviana* but there are 650 withanolides identified in the Solanaceae family (Fang et al., 2012; Puente et al., 2011; Sang-ngern et al., 2016). Moreover, there are other types of derived steroids known as physalins which are a group of specially and highly oxygenated ergostane-type withanolides with a wide array of pharmacological activities, including anticancer, anti-inflammatory, immunoregulatory, antimicrobial, trypanocidal and leishmanicidal, antinociceptive, antidiabetic and some other activities. Some physalins found in *Physalis peruviana* species are 25-Hydroxyphysalin F and 25- Hydroxyphysalin J (Wu et al., 2021).

5.2.4 Volatile subtances

Aroma and taste are some of the most important attributes in the quality of fruits and directly affect their consumption. The taste of fruits is related to the ripeness index and the total soluble solids, which are mainly determined by sugars and organic acids (Alencar et al., 2022). Generally, during ripening the sugar content increases while the acid content decreases, because of dilution and metabolism. Additional to those metabolites, fruits such as *P. peruviana* produces volatile compounds that provide them attractive organoleptic properties and that are related to the plant's defense and tolerance to abiotic stress, imparting systemic acquired resistance, allelopathy effects and inhibiting pathogen growth(Murali-Baskaran et al., 2022). Approximately 1700 volatile compounds exist in nature and are represented by terpenoids, phenylpropanoids and fatty acid derivatives (Muhlemann et al., 2014) and their presence dependent of the fruit and their maturity state (Alencar et al., 2022; Cozzolino et al., 2021; Dursun et al., 2021; El Hadi et al., 2013; Mashilo et al., 2022).

In general, aldehydes are also common in fruit flavors specifically in *Physalis peruviana* fruit table 2, they represent approximately 1.63-7.05% of the volatile substances, with Benzaldehyde, Hexanal, Nonanal and β-Cyclocitral being the most predominant ones

(Yilmaztekin, 2014b). The presence of aldehydes imparts the characteristic scent of cherry, which is waxy and green in aroma (Yilmaztekin, 2014a).

Table 1 Aldehydes and aroma descriptor referenced in Physalis peruviana.

Thus, some studies have demonstrated that lactones such as γ -Octalactone and γ -Hexalactone, along with Ethyl octanoate, 2-Heptanone and nonanal are directly related to the odor in *Physalis peruviana*, table 3. Additionally, these can represent between 2.09% to 24.1% of the total identified components (Yilmaztekin, 2014b).

Table 2 Lactones and aroma descriptor referenced in Physalis peruviana.

Other studies highlight the importance of esters, table 4, such as Ethyl butanoate, Methyl benzoate, Bethyl octanoate, Hexyl hexanoate and Butyl decanoate and as high-value compounds in creating the aroma, which can serve as quality markers for the fruit (Majcher et al., 2020). These changes may be associated with factors such as the cultivar, cultivation practices, ripeness stage, post-harvest handling, stress factors, and pest attacks. Among these, the ripeness stage is relevant in the development of fruit volatiles; therefore, if one wanted to enhance flavor and aroma, It could be beneficial to harvest the fruit at its ripeness for consumption to optimize the volatile content. However, in most cases, this strategy is considered unfeasible because fruits are generally harvested in their unripe state to extend their shelf life and ensure marketability (Balaguera et al., 2014).

Table 3

NF: no found.

Among the group of alcohol type constituents, *Physalis peruviana* has 1-Hexanol, 2- Heptanol, 2-Methylpropanol, Benzyl alcohol and Butanol (Mayorga et al., 2001). These are also associated as significant contributors to the characteristic fresh aroma with herbal and green notes. On the other hand, compounds like terpenes, table 5, are found in abundant quantities and are commonly associated with the aroma of almost all fruits and other foods, some are responsible for the aroma of flowers and most of them have an unspecified fruity odor (Kupska & Jeleń, 2017).

Table 4 Terpenes and aroma descriptor referenced in Physalis peruviana.

p-Cymen-8-ol 150.22 $C_{10}H_{14}O$

(Yilmaztekin, 2014a)

NF: not found.

5.3 Drying techniques

Drying is perhaps the oldest used method of preserving food and agricultural products (Devahastin & Jinorose, 2020), widely used to preserve harvested foods with the purpose of facilitating handling and packaging, reducing weight in transportation, improving processes like grinding and mixing, extending shelf life and enhancing stability during storage (Karama et al., 2016; Onwude et al., 2017). The drying process is energy intensive, requiring between 10 and 20% of the total energy used in global food production, in this way, one of the objectives of dry is remove water but exist different types of techniques and equipment, within materials is related to the forces exerted between water molecules, which can be termed free water, bound water and interstitial water (Ling et al., 2022).

The complexity of the drying process is due to the interrelation between the transport phenomena of heat and mass transfer at the same time, therefore physical and chemical changes occur (Aversa et al., 2007). Factor such as type of dryer, the exchange of momentum between systems (convection, conduction and radiation), the diffusion of thermal energy and mass transport within moist porous substances, as well as the phase change of liquid water vaporization are all factors to consider (Mujumdar, 2014). It is important to understand that regardless of the chosen drying equipment, it can affect the attributes or properties of the material to a greater or lesser extent (Pateiro et al., 2022). For the drying of fruits and vegetables, various techniques have been developed, which involve the application of drying modes that utilize heat or a combination of these. However, the selection of the most appropriate technique will depend on additional factors such as the type of product, energy consumption, the quality of the final product, conditions industry, efficiency and cost of drying (Onwude et al., 2017).

Various drying techniques (Sagar & Suresh Kumar, 2010) such as freeze-drying, convective drying and fluidized bed drying are employed with different material and industrial applications (Mujumdar, 2014). In the context of evaluating the drying process, it is crucial to refrain from exclusively prioritizing metrics such as drying speed, energy consumption or process capacity. This caution arises from the documentation impact of drying process on material attributes, especially in relation to volatile compounds (monoterpenes, sesquiterpenes, alcohols, aldehydes, ketones, furans) (Oliver-Simancas et al., 2020; Zhao et al., 2023), color expressed in L*a*b* (Li et al., 2023), carotenoids ((Z)-β-Carotene) (Onwude et al., 2022) or total phenolic content (Kittibunchakul et al., 2023).

Convective drying is a complex phenomenon involving the simultaneous transfer of heat and mass, liquid and vapor transport, as well as structural and chemical transformations within the material. These phenomena lead to changes in product quality and affect the drying mechanisms, which typically occur through two processes. In the first process, the transfer of energy (heat) occurs from the surrounding air to the wet product, which can be due to convection, conduction, radiation or a combination of these mechanisms. In the second process, moisture content from the interior of the material is transferred to the outer boundary of the solid. In a typical practice, thermal energy is transferred to the surface of the wet product and then into the interior of the product. The first process depends on environmental conditions such as air temperature, air humidity, flow conditions, product surface, and pressure. The second process depends on the movement of internal moisture within the solid and is influenced by the physical structure, temperature, and moisture distribution of the product. These parameters governing the first and second processes act as limiting factors that control the drying rate and total drying time (Karama et al., 2016; Patra et al., 2022).

Fluidized bed dryers are widely used for drying materials with particles sizes from 50- 2000 µm, including wet particles, sludges, pastes, and suspensions that can be fluidized within beds of inert solids. In a conventional fluidized bed, a stream of gas is passed through a column of solid particles at low gas velocities, the bed is considered static or packed. The particle bed rests on a gas distributor plate and the fluidizing gas passes through the distributor, evenly distributing throughout the bed. The pressure drops across the bed increases as the fluidizing gas velocity increases, and at certain gas velocities, the bed becomes fluidized when the gas stream fully supports the weight of the entire bed (Mujumdar, 2014).

Freeze drying, which is also known as lyophilization, is the process of removing water from a product by freezing followed by sublimation across three states (sublimation, primary drying,

and secondary drying) (Mellor & Bell, 2003). In those states, six main physical phenomena can be distinguished that have a significant impact on the course of the process, the quality of the obtained material, and the overall costs of the process. Those are: the phase transition of the water contained in the product into ice, the ice to vapor phase transition, the desorption of water molecules from material structures, the obtainment of a sufficiently low pressure, the re-sublimation of water vapor removed from the material on the surface of the condenser and finally the removal of a layer of ice from the surface of the capacitor (Nowak & Jakubczyk, 2020).

5.4 Effects of drying on chemical fruit properties

The acceptability of dehydrated fruit and vegetable products is highly dependent upon their flavor attributes. Unavoidable, there will always be losses of flavor constituents during pre-drying, drying and storage operations, since conditions like rough handling, delay in processing, exposure to light, high temperature and chemical reactions (oxidation and non-enzymatic browning) contributed to flavor deterioration. As it is shown in figure 3, levels of relevant substances such as vitamins (vitamin C or ascorbic acid) can be affected and decrease due to prolonged exposure to temperatures (30-70°C) due to their sensitivity to heat. As in the case of Kiwi, its dried fruits have lost up to 83% of Vitamin C, which represents a high loss if it is considered a substance of interest (Mujumdar, 2014; Onwude et al., 2022).

Figure 3

Source: Onwude et al., 2022.

Another example of susceptible substances in fruits are pigments (carotenoids and chlorophylls) which can diminish due to their exposition to oxygen, light and heating, figure 4. They are degraded easily thanks to the interactions with radical intermediates like unsaturated fatty acids, which are produced by autoxidation or lipoxygenases catalyzed co-oxidation. Additionally, long term storage of dehydrated generates a discoloration due to browning by enzymatic and nonenzymatic reactions, which affects organoleptic properties and consumer acceptance (Mujumdar, 2014; Onwude et al., 2022).

Source: Onwude et al., 2022.

Likewise, bioactive compounds that give fruits some medicinal properties could be altered, those bioactive compounds include the total phenolic content (TPC), which significantly decreases after being submitted to high temperatures, figure 5. Once TPC diminished, effects such as antioxidant and antitumor could disappear, avoiding taking the adequate profit of fruit consumption (Mujumdar, 2014; Onwude et al., 2022)

Source: Onwude et al., 2022.

On the other hand, temperature in the drying process also affects flavor and aroma. For example, peel mango was submitted to different temperatures and two different drying techniques table 6, it was evident that its volatile components changed even when the freeze-drying process was applied. The lowest losses were detected when an oven at 45 °C were used, undermining the capacity of lyophilization to preserve this type of compounds, table 6 (Oliver-Simancas et al., 2020).

Compound	FRESH		DRIED AT 45°C		DRIED AT 60°C		FREEZE DRIED	
	MEAN	SD	MEAN	SD	MEAN	SD	MEAN	SD
Hydrocarbon monoterpenes								
α -Pinene	0.39 ± 0.08^b		0.37 ± 0.08^b		0.20 ± 0.03^b		0.39 ± 0.08^b	
β -Pinene	0.02 ± 0.00 ^c		$0.01 \pm 0.00^{\circ}$		0.01 ± 0.00^b		$0.01\pm0.00^{\rm a}$	
Camphene	$0.01 \pm 0.00^{\circ}$		$0.01 \pm 0.00^{\circ}$		0.01 ± 0.00^b		0.00 ± 0.00^a	
3-Carene	67.19 ± 15.72^b		61.33 ± 5.86^b		$27.54 \pm 4.21^{\circ}$		$16.37 \pm 3.20^{\circ}$	
α -Phellandrene	5.62 ± 0.47 °		3.95 ± 0.91^b		1.86 ± 0.12^a		0.89 ± 0.21 ^a	
o-Cymene	0.07 ± 0.01^b		0.08 ± 0.00^b		0.04 ± 0.00^a		0.04 ± 0.00^a	
α -Terpinene	1.12 ± 0.23^b		0.87 ± 0.20^b		0.43 ± 0.02^a		$0.24 \pm 0.03^{\text{a}}$	
D-Limonene	4.68 ± 0.98 ^c		3.31 ± 0.31^b		1.77 ± 0.28 ^a		1.02 ± 0.21 ^a	
trans- β -Ocimene	0.40 ± 0.04 ^d		0.20 ± 0.02 ^c		0.09 ± 0.01^b		0.04 ± 0.01^a	
α -Terpinolene	12.18 ± 1.06^d		8.51 ± 1.10^c		3.54 ± 0.72^b		1.69 ± 0.43^a	
$\frac{1}{2}$ Hydrocabon (23 monoterpenes)	97.89 ± 0.03 ^c		83.38 ± 8.13^b		$39.79 \pm 5.57^{\text{a}}$		22.07 ± 4.28 ^a	

Table 5 Hydrocarbon and oxygenated monoterpenes compounds detected in fresh and dried mango peel samples.

Source: Oliver-Simancas et al., 2020.

"Fresh", no treatment applied; "Dried at 45 ºC", oven dried at 45 for 18 h; "Dried at 60ºC", oven dried at 60 ºC for 12h; "Freeze dried", Freeze dried at -53.2 ºC and 1.1 x 10-2 mbar for 12 h.

5.5 Extraction techniques

Bioactive extraction is the process of separating or distributing a solute between two immiscible liquids or phases. This intricate procedure is influenced by various factors, such as technique (Bitwell et al., 2023; Patra et al., 2022), viscosity, surface tension, vapor pressure, and polarity (Kislik, 2012). The extraction process consists of three steps. First, the sample is brought into contact with the extraction solvent through diffusion. Second step involves dissolving the analyte in the extraction solvent, the affinity of the analyte for the extraction solvent must be greater than its affinity for the matrix sample. Finally, the extraction phase (containing the analyte) must diffuse through the sample, separating into a distinct phase, which is removed from the insoluble matrix of the solid sample (Zhang et al., 2018). Conventional extraction methods include decoction extraction, Soxhlet extraction, steam distillation, high hydrostatic pressure extraction, pulse electric field process, high-pressure process, among others. However, traditional methods have the disadvantages of low extraction rate, large solvent consumption, high energy consumption and long time consuming. In addition, these methods are difficult to use to achieve the desired condition of crushing effect (Zhang et al., 2018).

5.5.1 Improvement of fruit extraction using ultrasound assisted extraction

Ultrasound-assisted extraction (UAE) is based on the partition of the analyte between the solid matrix and the extraction solvent by the assistance of high frequency (>20 kHz). Piezoelectric systems convert electrical energy to sound energy, the sound waves are spread through the medium and thereby cause rarefaction and compression cycles creating small bubbles. When the intensity of the ultrasound is large enough, the expansive cycle leads to the violent collapse of bubbles (cavitation) to generate pressure and temperature at a minuscule level. During the expansion cycles the bubbles increase in size, while throughout the compression cycles their size decreases. When those micro implosion occurs in the surface of the cell membrane generate a shear disruption or microporous, resulting in enhanced solvent penetration into cells and amplification of mass transfer of target compounds into the solvent (Tiwari, 2015) even this extraction technique induces in the material fragmentation, erosion, sonocapillary effect, sonoporation, local shear stress and detexturation (Chemat et al., 2017).

For the successful application UAE is related to the extraction efficiency parameters,however, it is necessary to consider that yield is not always the only objective of the extraction process. Moreover, the clean, green and sustainable influence factors should also be considered. Process parameters can be considered including design of a system, frequency, ultrasonic intensity, amplitude, electricity consumption, solvent type and matrix particle size, among others. Thus, the choice of the appropriates parameters should be optimized in ultrasonic assisted extraction process (Wen et al., 2018), which could be used in the extraction of volatile substances (Kimbaris et al., 2006; Murali-Baskaran et al., 2022; Pizani et al., 2022), phenolic components (Carrillo-Hormaza et al., 2020), saponins, proteins, anthocyanins, polysaccharides,, capsaicinoids, phenylpropanoids, terpenes, from different matrices such as pomegranate peel, Zyzyphus lotus fruit, *Garcinia madruno*, jabuticaba peel, longan fruit, orange peel, red raspberries, garlic cloves, pepper, caraway seeds, tomato pomace, rosemary, marjoram, saffron, spearmint and tremella (Yusoff et al., 2022; Zhang et al., 2018).

5.6 Solid phase microextraction followed by gas chromatography coupled to mass spectrometry (SPME-GC/MS)

Solid phase microextraction (SPME) was developed to address the need for rapid sample preparation, both in the laboratory and at the site of the investigated system (Pawliszyn, 2012) it's a modern, non-exhaustive sample preparation technique, which has integrated sampling, preconcentration and extraction into a single step and the trapped analytes can be directly introduced to analytical instruments like chromatographic systems. The advantages of this technique include simplicity, rapidity, improved sample clean-up, accurate analysis and low organic solvent consumption (solvent-free or solvent-minimized) (Jalili et al., 2020) and it has been used in analysis of environmental, food, pharmaceutical and biological samples (Sajid et al., 2019).

This type of sampling and extraction (SPME) can be performed in three modes including headspace, direct immersion and protected membrane. Headspace solid phase microextraction (HS-SPME) was first introduced in 1990 and is preferred for volatile compounds and complex samples such as biological ones (urine, whole blood, plasma, and hair). In HS-SPME, a fused silica fiber coated with an adsorbent is exposed in the headspace above the sample; volatile or semivolatile compounds are distributed among the sample, headspace and sorbent. HS-SPME is useful for samples with high molecular weight interferences. In this mode, volatile and semi-volatile compounds that equilibrate between the sample and the headspace are trapped by the fiber coating. In addition, the sample matrix is not in direct contact with the coating, it significantly increases the life span of the sorbent (Jalili et al., 2020; Mills & Walker, 2000).

5.6.1 Analysis of volatiles in food and fruits by HS-SPME

HS-SPME is a selective, sensitive and environmentally friendly process in food analysis. Food is a complex matrix and several procedures are typically used to prepare a food sample for gas chromatography, liquid chromatography or other analysis. Sampling and sample preparation depends on the type of matrix. To prepare a representative sample, a solid matrix needs to be homogenized and liquid or gaseous samples must be properly stirred prior to the isolation of the target analytes from the examined matrix. Fast isolation of the analytes from food matrices is particularly important to minimize or prevent changes in sample associated with enzyme activity, lipid oxidation, microbial growth and physical changes that are likely to occur in the unstable food systems (Kudlejova & Risticevic, 2012). Table 7 summarizes some applications of HS-SPME in various matrices with the aim of analyzing volatile components present in them, in addition to different separation and detection techniques.

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Analysed matrix	Target analytes	Extraction techniques	Separation and detection system				
Jasmine rice	Aroma profile	HS-SPME	GC-qMS/EI				
Mango	Unsaturated fatty acid esters	HS-SPME	GC-qMS/EI				
Strawberries	Aroma profile	HS-SPME	GCxGC-FID				
Avocado	Aroma profile	HS-SPME	$GC-qMS/EI$				
Passion fruit	Aroma profile	HS-SPME	GC-FID				
Apple juice	Off-flavor compounds	HS-SPME	GC-qMS/EI				
Cocoa	Pyrazine	HS-SPME	GC-FID				
Coffee	Aroma profile	HS-SPME	GC-FID				
Tomato plant	Methyl salicylate	HS-SPME	GC-qMS/EI				

Table 6 Different matrix analyzed by solid phase microextraction with head space.

Source: Kudlejova & Risticevic, 2012.

5.7 Functional ingredients and flavoring

For centuries, ingredients have played important roles in a wide variety of foods. Our ancestors used salt to preserve meats, added herbs and spices to enhance flavor or sugar for fruit preservation, among many others (Hacelas, 2020). Nowadays, natural ingredients are common in a wide range of products, especially in healthy and beauty markets related to cosmetics and food. Trends such as "natural ingredients, organic personal care ingredients, or bioingredients" are becoming more persistent. Additionally, the modern consumer is more critical and pays attention to the ingredient content in a final product, so it is seen as a positive fact, especially for functional ingredients (Amberg & Fogarassy, 2019). Moreover, it has been evidenced that foods can be used as vehicles for the release of bioactive and micronutrients at the levels required to achieve health benefits, suggesting a need for the development of new ingredients (Day et al., 2009).

The market of functional ingredients and foods has experienced growth in recent years due to people's conscious consumption, the increase in the promotion of healthy eating and lifestyles, and the need for products rich in flavor and color (Day et al., 2009; Industryarc, 2020). Countries

in the Asian-Pacific region and in North America have the largest functional food markets, representing 34% and 25%, respectively. It is expected that by the year 2026, the functional food market will reach 7.8 billion dollars. Other analyses of the market for ingredients reveal a promising outlook for growing industries. According to records from the year 2015, more than 3.5 million tons of botanical ingredients were used in the production of food, personal care products, and health products. Additionally, an annual global growth of 2% and a national growth of 7% are projected. In Latin America, nearly one-third of the population (approximately 36% of Brazil, 31% of Mexico, 28% of Argentina and 32% of Colombia) prefers to consume food products made from botanical ingredients, with a focus on a cleaner and health-preserved food consumption (Conway, 2023; Lau et al., 2021). It is clear, therefore, that the market is constantly demanding new natural ingredients with scientifically supported claims, this has led to increased attention on sources of various natural compounds and their associated bioactivities. Furthermore, the growing concern for the environment is a central theme in the development of natural ingredients, not only among consumers but also among manufacturers who are progressively focused on maintaining a sustainable supply of ingredients, which in turn generates more interest in the concept of all-natural products. As a result, significant opportunities are emerging for natural ingredients in all global markets (Hacelas, 2020). However, the limited capacity of the country to meet the domestic demand for secondary transformation products from natural ingredients is reflected in the statistics reported by the United Nations Statistics Division Trade Map Database, which demonstrated the country's limited capacity within ingredients area, for example, in 2019, the value of imports of gums, resins and plant extracts was 24117 billion dollars, while the value of exports was only 1071 billion dollars, resulting in a 24 to 1 imbalance between imports and exports. Similarly, many of these imported products are obtained from agricultural sources cultivated on a large scale in Colombia but it lacks the transformation process needed to generate extracts and ingredients of higher added value. The same occurs with essential oils, in 2019 alone, the volume of imports was 710.8 Ton, while the volume of exports was 30.3 Ton, a 23-fold imbalance. The same effect occurs with fats and oils, as during 2019, 2.791.126 dollars were imported while the value of exports was 831.8 billion dollars, resulting in a 3.4-fold imbalance (Trademap, 2023).

There are thousands of ingredients used in food preparation, many of which are consumed daily by consumers, these include spices, colorants, flavorings, sweeteners, and more. However, consumers express concerns about the consumption of many of these ingredients, primarily due to the lack of knowledge regarding the chemical components they contain, which are directly involved in preserving and enhancing the taste, color, and texture of food. Considering this, all food additives must be carefully regulated by federal authorities and international organizations to ensure they are safe and properly labeled (FDA, 2010). Plants are abundant sources of bioactive compounds that contribute to improving and enhancing the quality and safety of various food products. Extracts derived from plants have gained significant importance in recent years as their are useful as flavoring components in foods and as active components due to their biological functions that give them properties as antioxidants, antimicrobial agents, colorants, nutrients and flavor enhancers (Mir et al., 2022; Modupalli et al., 2022).

Obtaining plant-derived products or bioproducts begins with the collection of the material that will serve as a functional ingredient. This material is typically collected from open environments and its composition can vary depending on factors such as climate, geographical location, and so on. Therefore, It is important to ensure functionality and safety by employing standardized production conditions that minimize the range of variability (Gendel, 2021; Iranshahi et al., 2023).

On the other hand, flavoring ingredients are among the most complex components of the food industry, made up of a large and varied array of ingredients from different sources. Flavor is a relevant factor for sensory acceptance and consumer decision making processes, the specific taste sensation in each food is a combination of sensory perceptions of aroma and taste, which is due to the presence of characteristic chemical components that act as stimuli for the taste receptors in the mouth. The production, marketing and consumption of exotic fruits have significantly increased in both domestic and international markets, thanks to their sensory properties. This is attributed to a wide range of essential volatile compounds responsible for providing color, aroma and flavor. These volatile substances have been widely used as flavoring agents and are generally recognized as safe (Ayala-Zavala et al., 2011; Butrym et al., 2019; Modupalli et al., 2022). Likewise, color is another important attribute in food quality that is receiving increasing attention due to consumer rejection of synthetic pigments. Unlike the perception associated with natural colorants or those derived from natural alternatives, which are perceived as healthy and of good quality, whether they are approved or not (Modupalli et al., 2022).

6 Materials and methods

6.1 Materials

Cape gooseberry (CG) was purchased in a local market, reagents were provided by Grupo de Investigación en Sustancias Bioactivas (GISB) and they included ethanol (purity, 99.6%), NaCl (purity, 99.99%), deionized water and standard volatile compounds ((-)-β-Pinene (purity, 98.5%), 1R-α-Pinene (purity, 98.5%), Eucalyptol (purity, 99.99%) and Carvone (purity, 99.99%).

6.2 Fresh fruits classification

In this section, correlations between *Physalis peruviana* fresh fruit variables like diameter, weight, color and soluble solids content were explore to understand and stablish criteria variables to homogenize the fruits through a classification process.

6.2.1 Diameter

Overripe fruits and those with any signs of mechanical damage were removed. The material was washed, and its diameter was measured on it´s equatorial zone and classified according to the NTC 4580 in five groups to obtain a homogeneous groups.

Table 7 *Diameter classification of Physalis peruviana based on NTC 4580.* **Diameter (mm)** ≤ 15.0 15.1-18.0 18.1-20.0 20.1-22.0 ≥ 22.1 Caliber A B C D E

NTC: Colombian techbical standard.

6.2.2 Soluble solids content

A refractometric method was used in this methodology. First, the material was washed and based on its weight, then it was grouped into five categories, table 9.

Next, fruits were cut in half and its juice was squeezed out to measure the soluble solids content (°Brix) using a refractometer (Atago model 3810 PAL-1), table 10.

°Bx: solids soluble content

6.2.3 Color scale in CIE L*a*b* format

Applying the methodology described in point 6.2.2 and before cutting the material in half, the color was measured, only in the equatorial zone, using a high-quality colorimeter NR200 in the space color CIE L*a*b* where L* refers to luminosity, a* red/green and b* yellow/blue.

6.3 Volatile component analysis by headspace solid phase microextraction and gas chromatography coupled to mass spectrometry (HS-SPME-GC/MS)

The analysis was performed using an Agilent 7890 gas chromatograph couplet to a mass spectrometer 5975C. The chromatographic separation was carried out with a HP-5MS capillary column (30m x 0.25mm x 0.25μm), initially the oven was heated at 35°C for 4 min, next the temperature was increased from 35°C to 180°C at a rate of 3°C/min, The injector temperature was set to 220 °C and Helium was used as the gas carrier at a flow rate of 1.1 mL/min. Samples were prepared through the extraction of volatile compounds by headspace solid phase microextraction (HS-SMPE) with a DVB/CAR/PDM fiber, Mass spectrum of each volatile compound was obtained at 70 eV at a rate of 1 scan per second and overing a mass range of 33-400 m/z (Yilmaztekin, 2014a). The compounds identification were separated into four classifications: confirmed, high, medium and low, table 11. Based on identification confidence and the NIST MS library match factor score (match factor refers to a comparison of the unknown's mass spectrum's peaks to those of the peaks in the library's spectra) was assigned.

Peak identification confidence criteria.					
Identification confidence	NIST MS match factor score criteria				
Confirmed	Identification confirmed by reference standard				
High	850-1000				
Medium	700-849				
Low	500-699				
NA	500				

Table 11 Peak identification confidence criteria.

Table 12

Some compounds as 1R-α-Pinene, (-)-β-Pinene and Eucalyptol were confirmed and contrasted with retention times of standards

6.3.1 Optimization volatile analysis conditions

The fiber DVB/CAR/PDM was tested to select the best conditions for the extraction of the *P. peruviana* volatiles compounds which were represented in total area, table 12. HS-SPME methodology was improved with a Box-Behnken design with three variables (Incubation temperature, extraction time and desorption time) with three levels and the response variable was the total area, the instrument raw data files were processed using a data analysis workflow through Agilent Masshunter Analysis software version B.07.00 (Agilent Technologies; Santa Clara, CA)

box-bennken design wun incubation temperature, extraction time, desorption time.							
Factor	Name	levels					
		- 1			Response variable		
X1	Incubation temperature $({}^{\circ}C)$	20	30	40			
X2	Extraction time (min)	20	30	40	Total area		
X3	Desorption time (min)						

Box-Behnken design with incubation temperature, extraction time, desorption time.

The HS-SPME-GC/MS methodology with the optimized variables were subjected to a response surface optimization design, additionally a Pareto chart was used to understand the prioritization variables. Finally, linearity, range, repeatability, limit of detection (LOD) and limit of quantification (LOQ) of the method was validated with three standards (-)-α-Pinene, (-)-β-Pinene and Eucalyptol. Five different standard concentrations were prepared and subsequently dissolved in 15 mL of NaCl solution (13% w/w). Samples were analyzed in triplicate using Carvone as internal standard.

$$
LOD = \frac{3.3 \text{ (Standard deviation)}}{\text{Slope of the line}} \tag{6.3.1}
$$

$$
LOQ = \frac{10 (Standard\ deviation)}{Slope\ of\ the\ line}
$$
 (6.3.2)

6.3.2 Volatile analysis associated with different maturity stages of fresh fruits

Before analyzing the volatile composition according to the fruit ripeness stage based on Brix degrees, trials were conducted with fresh fruits, only selecting those in good condition or visually immature. However, the results did not consistently replicate across each trial, indicating significant variability in volatile composition. Therefore, considering the fruit's climacteric nature, it was deemed necessary to assess the volatile composition through a more rigorous classification based on ripeness stage, estimating the total soluble solids content.

To understand how the volatile composition changes throughout the ripening process of *P. peruviana* fruits a HS-SPME-GC/MS analysis was done. The material was prepared and categorized based on its soluble solids content (°Bx), which fell within the ranges 7-8, 10-11 and 14-15 °Bx. Fresh fruits were crushed using a grinder to achieve a homogeneous texture and smaller particle size. Approximately 2 grams of crushed material were prepared and placed in a headspace recipient with 15 mL of NaCl saturated solution (13% w/w) to minimize variability in ionic strength, improve sensitivity and promote the transfer of the analytes to the headspace (Buttery et al., 1987). Subsequently, the samples were analyzed with the optimized method and volatiles of fresh fruits were assigned using MassHunter software. Finally, the area of the main peaks was assigned.

6.4 Drying process in *Physalis peruviana* **fruits**

In order to transform *Physalis peruviana* into a product for direct consumption or suitable raw material for blending and formulating new products, the utilization of drying techniques was proposed to ensure physicochemical properties and microbiological preservation. During the drying process of *Physalis peruviana* fruits, conventionally used dehydration techniques were evaluated, including convective drying, fluidized bed drying, and freeze-drying, it is acknowledged that freeze-drying is an efficient technique but challenging to scale up to an industrial level due to its energy consumption and equipment cost. Therefore, convective drying and fluidized bed techniques were chosen for their accessibility and scalability, respectively.

6.4.1 Initial moisture content

Overripe fruits and those with any signs of mechanical damage were removed, once the material was selected, each fruit was cut into four pieces and grouped to gather a sample of 40 grams. The material was placed in an oven (Binder model ED53) for drying, which was set at a temperature of $110^{\circ}C \pm 5^{\circ}C$ for 8 hours or until it's mass did not vary by more than 1% according to NTC 1495. The dried fruit was weighed to calculate the initial moisture according to the equation 6.4.1:

$$
\% Moisture content = \frac{fresh\ material - dry\ material}{fresh\ material} * 100 \quad (6.4.1)
$$

6.4.2 Convective drying kinetic

Overripe fruits and those with any signs of mechanical damage were removed, *Physalis peruviana* fresh fruit selected were cut into four pieces. Subsequently, nine groups of cut fruits were formed and weighed (40 grams) and submitted to a drying process using a convective dryer (Thermolab C480) at 60°C oven temperature; air flow was not measured by equipment's electrical functionality. Total drying time was 48 hours, samples were taken at 0, 1, 2, 3, 5, 8, 10, 24 and 48 hours. The moisture content for each sample was determined according to methodology 6.4.1. On the other hand, the previous methodology was repeated, but in this case, a drying ramp was performed, starting at 60°C, and after two hours, the temperature was changed to 40°C until completing the 48 hours of the established processing time.

6.4.3 Freeze drying kinetic

Overripe fruits and those with any signs of mechanical damage were removed, *Physalis peruviana* fresh fruit were cut into four pieces. Subsequently, nine groups of cut fruits were formed and weighed (40 grams). They were stored at -20°C for 24 hours and submitted to a drying process using a freeze dryer (Biobase BK-FD12P), which operated at 100 Pa with a chamber temperature of -78.5°C. Total drying time was 48 hours, samples were taken at 0, 1, 2, 3, 5, 8, 10, 24 and 48 hours. The moisture content for each sample was determined according to methodology 6.4.1.

6.4.4 Fluidized bed drying kinetic

Overripe fruits and those with any signs of mechanical damage were removed, a sample of 1000 grams of *Physalis peruviana* was classified and cut into two pieces and dried in a fluidized bed dryer (Actum model SLF20L), it operated at 60 °C chamber temperature, agitation was off and a ventilation speed of 38 m/s. Total drying time was 5 hours and the moisture content was determined at 0, 1, 2, 3, 4 and 5 hours.

6.5. Analysis of volatile compounds in dried fruits and extracts

Once the drying kinetics were established for the three drying techniques studied, the equilibrium moisture content was determined for each one, this refers to the time at which the moisture remains constant; the total drying time was individually established based on the kinetic curve. Before being dried, fresh fruits were classified strictly within a range of soluble solids content between 12-13°Bx. The dried material obtained was analyzed using HS-SPME-GC/MS with the optimized method and results were analyzed by comparing the presence of compounds with respect to those obtained from fresh material and how they vary among the different drying methods used. All samples were tested in triplicate.

Dried fruits obtained by fluidized bed dryer were submitted to an ethanolic extraction using a high intensity ultrasound, volatiles of those extracts were also analyzed before and after to produce a dry ingredient through rotary evaporation and lyophilization.

6.6 Obtaining of a prototype of the flavoring ingredient from an extract of *Physalis peruviana*

6.6.1 High intensity ultrasound extraction of dried fruits

Extracts were obtained from a dried material previously dehydrated by a fluidized bed dryer. After obtaining the dried material, high-intensity ultrasound assisted extraction process was developed in an LSP-500 ultrasonic liquid processor (Sonomechanics, New York) equipped with an ultrasonic generator of 500 W, an air-cooled piezoelectric transducer (ACT-500), a full-wave Barbell Horn™ (FBH, 21mm tip diameter) and a reactor chamber (304 stainless steel). For continuous flow mode (300 mL/min) a Masterflex L/S digital peristaltic pump system with an Easy-Load® II pump head, a 1 L glass-jacketed beaker and a Polystat cooling circulating bath were coupled to the ultrasonic system. All experiments were performed at a constant frequency for 10 minutes with a batch size of 3500 mL of solvent, 10 % w/v of sample (35 g of dried powdered Cape gooseberry), a cooling temperature of 5° C and a stirring of 5600 rpm in the glass jacketed beaker. The impact of the ethanol concentration (v/v) and probe's amplitude in microns peak to peak (μpp), regarding the extraction of volatile compounds was evaluated under a single factorial design; the factors were ethanol concentration (70 and 80%) and probe's amplitude (13.899 and 46.330 μpp).

6.6.2 Final processing of the extract for the obtaining of the prototype of an ingredient.

The extracts were centrifuged during 10 minutes at 7000 rpm, supernatants were recovered and rotary evaporated at a pressure of 175 mbar with a heating bath at 40°C until the condensation flow of the solvent ceased. The final extracts was stored at -80°C for 24 hours and then subjected to a freeze-drying process to obtain dried extracts considered as final version of a prototype of the ingredient.

6.7 Sensory profile by multidimensional approach

Two sensory analyses were conducted for the identification and selection of descriptors and the establishment of their sensory profile using multidimensional approximation under compliance with NTC 3932 for both fresh material and lyophilized extract. The first material consisted was 2000 grams of fresh fruit according to point 6.2 and classified between 12-13 °Brix. The second material comprised was 200 grams of lyophilized extract or prototype obtained in point 6.6.2. Additionally, the material underwent tests for mesophilic aerobic counts (<10 CFU/g), molds and yeasts (<10 CFU/g), *E. coli* (<1 CFU/g), and *Salmonella spp* (absence /25g).

6.8 Statistical and data analysis

Area of volatile components was obtained with Qualitative Analysis of MassHunter Acquisition data B.07.00 and all the experiments were carried out at least in triplicate; areas and other values are expressed as the mean \pm standard deviation. The analysis of nonlinear regression (Newton, Gaussian, Henderson & Pabis and Fourier) were carried out using Matlab® version R2019a with its extension Curve fitting. Analysis of variance (ANOVA) was carried out for each variable to test the statistical significance using a p-value at 5% level. Data analysis with one, two factors and PCA were performed by Rstudio version 4.3.2. Pareto chart, surface optimization and effect of principal components graph were made in Statgraphics Centurion version XVI.I, Venn diagram, heat map and bar chart were made in Graph Pad Prism® version 8.

7 Results and discussion

7.1 Characterization of *Physalis peruviana*

According to NTC 4580 the fruits of *Physalis peruviana* can be classified by counting, diameter or by other commercial practices. An inicial characterization of frutis was made, diameter, weight, color, total soluble solids (°Brix) and volatile composition were measured to search relationships between non-volatile and volatile composition. Concerning export, *Physalis peruviana* is categorized into three distinct classes: Extra, I and II, for this investigation an "extra" category was used, which is defined asintact whole fruit with smooth and lustrous rind, showcasing its distinctive color free from blemishes of signs of damage. According to table 13 the fruits of *Physalis peruviana* had an average caliber definded as C according to NTC 4580, these values are similar to those previously reported by Yıldız et al., 2015. However, it is important to note that the source of the fruits is the local market from the municipality of La Unión, Antioquia, which means that there exists a degree of uncertainty regarding the specific ecotype of the fruits (Florez et al., 2000).

Table 13	
Average diameter of fruits.	

 $n = 50$

The NTC 4580 establishes independence between fruit color and size or caliber. Therefore, in this study an examination of the relationship between weight and solids soluble content was conducted through an analysis of variance (p-value <0.01), it was clear that there was neither a direct correlation between weight and soluble solids content, table 14.

Table 14 Variance analysis of the association between °Brix vs Weigh.

	Df	Sum Sq	Mean Sq	F Value	$Pr(\geq F)$
Weight		15,618	3,904	2,386	0.054
Residuals	145	237,315	1,637		
$\sqrt{2}$					

p value <0.01

 $n = 150$

Maturity at harvest is the most important factor that determines storage life and final fruit quality (Kader, 1999) and it can be visually assessed by observing the change in its external color (ICONTEC, 1999). This standard introduces seven categories that establish the relation between color, solids soluble content, stage of maturity and titratable acidity. Based on table 15, the strategy used in this work to assign color did not yield colors that match those presented in the standard document (NTC4580), this may be because standard classification was made considering a description of the entire fruit and in this investigation the color was only measured in the equatorial zone. The parameter identified as "color view" is represented by RGB scale that was obtained from CIEL*a*b* scale (using Matlab).

Table 15 Relationship between classification parameters evaluated on fresh fruits of P. peruviana

Classification		Brix ranges Brix measured	L^*	a*	h^*	Color view
$\overline{0}$	<10	9.60 ± 0.28		65.02 ± 1.32 -1.95 ± 11.75	47.91 ± 0.89	
	$10 - 11$	10.53 ± 0.31		60.87 ± 4.31 11.73 ± 2.14	40.35 ± 3.68	
$\overline{2}$	$11 - 12$	11.60 ± 0.31	60.60 ± 4.28 11.38 ± 2.57		41.32 ± 3.59	
3	$12 - 13$	12.53 ± 0.31		59.51 ± 4.01 12.83 ± 2.99	40.15 ± 4.31	
$\overline{4}$	$13 - 14$	13.46 ± 0.29		$58.27 + 4.05$ $14.49 + 2.25$	41.07 ± 4.08	
-5	14-15	14.45 ± 0.28		58.53 ± 2.70 14.38 ± 2.16	40.50 ± 3.56	
6	>15	15.22 ± 0.21		57.21 ± 2.29 14.02 ± 1.55	39.44 ± 3.20	

n = 150

Sugars, acids, total solids, pH, color, pigments, and others characteristics are considered as non-volatile substances that drive fruit eating quality which, with the proper balance, indicate the ripeness level (Arshamian et al., 2017; Stingone et al., 2017). In line with the fact that the fruits used in this research were obtained from local markets, it was evident that the number of fruits classified as 0 would be minimal. Additionally, this can also be observed in the "color view" column as the tone associated with classification 0 was noticeably different from the other tones, furthermore the range of values obtained in the colorimetric space were consistent with the reported ones (Restrepo et al., 2009; Yilmaztekin, 2014b). Principal component analysis (Abdi & Williams, 2010) was conducted to explore the potential relationship between a non-volatile component such as solids soluble content (${}^{\circ}$ Brix) and color expressed as L^{*}, a^{*} and b^{*} values; data were normalized indicating that a component can capture the 42.149% of the accumulated variance, two components 77.72% and three 92.186%; table 16. A biplot for two components were selected, figure 6, linear combinations for ${}^{\circ}B$ rix and L*, a* and b* values were 0.3796, - 0.6763, 0.0771 and 0.6264 for component 1 and 0.5655, 0.0675, 0.7384 and 0.3608 for component 2, respectively. For this case, component 1 highlights a correlation between coordinate a* and °Brix within the same plane, involving a meaningful linear connection between the two variables, it is worth noting that this relationship does not extend to L* and b* coordinates.

Acknowledging the existence of a correlation between solids soluble content (°Brix) and color coordinate a*, a least significant differences test (LSD) was made, table 17. This test with a p-value < 0.01 enables the identification of three possible groups a, b and c. Likewise, these groups could be suggested to be associated with fruit maturity.

LSD test: coorainate a* of color ana sollas soluble content					
$^{\circ}Brix$	Mean	Group	Maturity		
<10	-1.945	a	Unripe		
$10 - 11$	11.731	b	Ripe		
$11 - 12$	11.038	b	Ripe		
$12 - 13$	12.827	b	Ripe		
$13 - 14$	14.489	c	Overripe		
$14 - 15$	14.376	c	Overripe		
>15	14.016	$\mathbf c$	Overripe		

Table 107 LSD test: coordinate a of color and solids soluble content*

7.2 Volatile composition of fresh *Physalis peruviana*

Flavors are sensory impressions that could be experienced with the consumption of foods and beverages, they are formed by several chemical substances such as volatiles which, in this specific case, are responsible for fruit characters. However, it is the interaction of volatile with nonvolatile compounds that contributes to the final sensory perception of food. Sweetness and sourness in fruit are due to the presence of sugars and acids. Phenolic compounds, anthocyanins, alkaloids and amino acids may elicit other sensory responses such as astringency, bitterness and umami. The presence of long chain polysaccharides such as pectin and other cell wall polysaccharides can affect the mouth fell of a fruit, all these non-volatile compounds interact with aroma volatile compounds, the intact fruit and the mouth upon chewing, which determines the overall sensation of aroma and taste that comprise flavor (Arshamian et al., 2017) given in turn by the stimulation of the human odorant receptors (Block, 2018).

Initially*, Physlais peruviana* fruits were evaluated by HS-SPME-GC/MS after discarding those fruit that were immature or had physical damaged, which highlighted the need for a more rigorous classification of the plant material to be used in subsequent processes. It was crucial to ensure control over confounding variables that may lead to errors or biases from the classification stage onwards. Therefore, classification was based on Brix degrees, ensuring a narrower range of maturity, the selection criterion for the material to be analyzed considers fruits with Brix degrees below 10 as immature and those with values above 14 as showing deterioration or over-ripening. Consequently, fruits within intermediate ranges (12-13 °Brix) were chosen for evaluation to preserve the integrity and properties of the fruits.

Volatile compounds present in fresh *Physalis peruviana* fruit were classified in a range of 12-13 °Brix and evaluated by HS-SPME-GC/MS, result was presented in the chromatogram illustrated in the figure 7. A total of 46 peaks were pointed out and assigned using a NIST database, table 18; most of them have been previously reported and components with match factor lower than 500 were not selected. The presence of some of the components were confirmed using standards of (-)-α-Pinene, (-)-β-Pinene and Eucalyptol.

Figure 7

P1: Ethyl acetate, P2:Butanol, P3: Hexanal, P4: Ethyl butanoate, P5: Butyl ethanoate, P6: (E)-2-Hexenal, P7: Hexanol, P8: 1R-α-Pinene, P9: Camphene, P10: Isobutyl butyrate, P11: Benzaldehyde, P12: (-)-β-Pinene, P13: Ether benzyl methyl, P14: β-Pinene, P15: Butyl butanoate, P16: Ethyl hexanoate, P17: Terpinolene, P18: Hexyl acetate, P19: o-Cymene, P20: β-Terpinyl acetate, P21: Eucalyptol, P22: β-cis-Ocimene, P23: γ-Terpinene, P24:Octanol, P25: α-Terpineol, P26: Carveol, P27: Methyl octanoate, P28: 4-Terpineol, P29: p-Cymen-8-ol, P30: Butyl hexanoate, P31: Ethyl octanoate, P32: β-Cyclocitral, P33: γ-Undecalactone, P34: Propyl octanoate, P35: Ethyl nonanoate, P36: Methyl decanoate, P37: Isobutyl octanoate, P38: Butyl octanoate, P39: Ethyl decanoate, P40: Geranyl acetone, P41: β-Ionone, P42: Propyl decanoate, P43: Cadinene, P44: Ethyl dodecanoate, P45: Myristic acid and P46: Hexadecanoic acid.

In nature, esters are one of the largest and main group of volatile compounds identified in fruit aroma (Bruckner, 2008). Aliphatic esters contribute to the aroma of nearly all fruits and are responsible for a particular fruit aroma or for the smell of odorants, giving characteristic odors described as fruity, sweet, pungent, pineapple, banana, wine, green, wax, among others (Niu et al., 2019). Esters comprise the second most abundant volatile compounds in *Physalis peruviana* representing 38.52% (Yilmaztekin, 2014a)*.* In this investigation, Ethyl butanoate, Ethyl acetate, Butyl ethanoate, Isobutyl butyrate, Hexyl acetate, β-Terpinyl acetate, Methyl octanoate, Ethyl decanoate were esters found. Free and esterified primary alcohols occur widely in fruits and their odor is relatively weak (Surburg & Panten, 2006). Alcohols such 1-Hexanol, Butanol and Carveol were found too; they are reported as an important contributor to the aroma of "fresh" that are described as green and herbaceous notes. According to Yilmaztekin, 2014a alcohols are the most abundant volatile constituents in *Physalis peruviana* (43.8%) with a reported concentration between 268-300 μg/kg. Terpenes, linear or cyclic, could be present (Breitmaier, 2006; Masyita et al., 2022). According to Yilmaztekin, 2014a, terpenes and derivatives compounds in *Physalis peruviana* represented 7.31% of volatile compounds.

After a HS-SPME-GC/MS analysis of *P. peruviana* fruits, the type of components previously described were found and according to its match factor there were possible to set the confidence of the identification process, where just one of the assigned components had a low identification confidence suggesting that a deeper analysis should be done. Additionally to those components, the presence of lactones, aldehydes and fatty acids were suggested by the database.

Tentative compound	Match factor	Identification confidence	Retention time \pm SD (min)	Group Compound	Aroma descriptor (Arshamian et al., 2017)	Reference
Ethyl butanoate	791	Medium	$6.868\pm$ 0.019	Ester	Fruity, sweet, reminiscent pineapple or apple	(Majcher et al., 2020; Kupska & Jeleń, 2017; Yilmaztekin, 2014a)
Ethyl acetate	731	Medium	$2.484+$ 0.014	Ester	Sweet, fruity	(Yilmaztekin, 2014a)
Butyl ethanoate	821	Medium	$7.414+$ 0.015	Ester	Fruity like apple	(Gutiérrez et al., 2010; Kupska & Jelen, 2017; Yilmaztekin, 2014a)
Isobutyl butyrate	679	Low	$14.231 \pm$ 0.044	Ester	Fruity	No references
Hexyl acetate	741	Medium	$17.129 \pm$ 0.004	Ester	Fruity green note reminiscent of pear with floral facets	(Gutiérrez et al., 2010; Yilmaztekin, 2014b)

Table 18 *Tentative compounds found in fresh fruit Physalis peruviana classified between 12-13 °Brix and their odor description.*

Yilmaztekin, 2014a;

Gutiérrez et al., 2010)

SD: standard deviation

Additionally some peaks were identified as solvent components or column degradation products, that include Hexamethyl cyclotrisiloxane or Hexadecamethyl heptasiloxane, figure 8. The chromatogram exhibits co-elution in some peaks, hindering the complete identification of these compounds. Peaks related to the NaCl saturated solution used as solvent are shown, peak in the retention time of 28.673 minutes is (+)-Carvone (5 ppm) which was used as internal standard.

** (+)-Carvona as standard reference (5 ppm)*

Analyzing variation of volatile composition according to the state of ripeness, fruits were classified according to their soluble solids content in three different categories 7-8, 10-11 and 14- 15; although 46 components were initially identified, the analysis was made prioritizing 16 of them, figura 9.

Figure 9 Total area of volatile components in three category of fruits according to its °Brix.

*Significant difference: ****p <0.0001, *** p-value < 0.001, ** p-value< 0.01, * p-value<0.1, ns no statistical difference.*

Results showed that, independent of maturity stage, there are components in great abundance (Ethyl octanoate, Terpinolene, etc.) and another in smaller quantities (Camphene and β-Cyclocitral). Additionally, certain components such as Ethyl octanoate and Ethyl decanoate demonstrated a correlation between their area and °Bx, with the highest area at a 10-11 °Bx range followed by a subsequent reduction. Another noteworthy behavior involves an increase in area as the °Brix range increments how it was observed in the case of 2-Methyl butanol, opposite to β-Cyclocitral which decreased. The volatile profiles of fruits are intricate and multifaceted, with fluctuations influenced by factors like the cultivar, ripeness, environmental conditions before and after harvesting, the form of the fruit sample and the analytical methods employed for analysis, factors that could be responsible for the changes occurred in *Physalis peruviana* through its ripening process (Bruckner, 2008). Different investigations have evidenced those modifications in others fruits, for example, Ethyl decanoate, Ethyl butanoate, β-Ocimene, 4-Terpineol in Tomate (*Solanum betaceum*) have highest level in a middle stage of maturity (Quintero Ramírez et al., 2023). Another clear example is the levels of Ethyl hexanoate of feijoa fruit (*Acca sellowiana*) which at 13.58 ± 0.16 °Bx has the highest level (Song et al., 2023). Storage is other criteria to

consider the presence of volatile compounds, as is in the case of stored tangerines (*Citrus reticulata Blanco*) where the quantity of 4-Terpineol and α -Terpineol decreased after postharvest (Tietel et al., 2011). This was evidenced in other fruits such as orange and "Braeburn" apple where β-Cyclocitral is an indicator of flavor deterioration for orange juice. Other examples are 2-Methyl butanol and Ethyl butanoate that decreased after sixth storage day in "Braeburn" apples (Mpelasoka & Hossein Behboudian, 2002; Perez-Cacho & Rouseff, 2008).

Despite the initial classification of the analyzed samples, standard deviations were observed between the replicates, figure 10, this relative standard deviation is common in research involving HS-SPME-GC/MS as shown regardless of the type of SMPE fiber used as CAR-DVB-PDMS, CAR-PDMS, DVB-PDMS and PDMS (Minář et al., 2023; Quintero Ramírez et al., 2023; Song et al., 2023). In this case, it is worth considering that the use of heterogeneous material, composed of seeds, skin, and pulp may not ensure the consistent preservation of their proportions in each prepared sample. Likewise, it is considered that volatile composition fruits vary according to their genetics, preharvest factors, postharvest handling, temperature, storage atmosphere and chemical applications; as a result, this variability can introduce deviations when the samples are analyzed (Bruckner, 2008; Christensen et al., 2007).

Considering variations between replicates it was needed to evaluate some variables to optimize headspace-solid-phase microextraction (HS-SPME) conditions for the purpose of obtaining the best volatilization and extraction of the compounds of interest.

7.3. Optimization of volatile extraction conditions

The use of the HS-SPME-GC/MS technique enables comprehensive exploration across a spectrum of boiling points and polarity gradients in each sample (Roszkowska et al., 2019). The underlying principle of the SPME technique revolves around the partitioning of analytes between the complex sample matrix and the interaction with fiber coating. The successful SPME extraction materializes when the analyte attains a state of equilibrium, achieving a concentration distribution equilibrium between the sample matrix and the fiber, this equilibrium is influenced by the type of fiber material (PDMS liquids, DVB or CAR solids and DVB/CAR/PDMS pulps) (Carasek & Pawliszyn, 2006), extraction mode, separation detection mode, agitation method, sample volume, extraction time, pH, ionic strength, temperature, percentage of organic modifier, calibration method (Pawliszyn, 2000).

In this investigation, it was attempted to improve the HS-SPME-GC/MS method using a Box-Behnken experimental design, evaluating the total area obtained as a response variable in the chromatogram with the aim of obtaining the highest possible area that represented the total volatile components present in *Physalis peruviana*. In total, 13 sample injections were performed and the ANOVA results are detailed in table 19. According to the evaluated variables, just incubation temperature had a significant effect on the total area obtained.

The results of the experimental design indicated that response can be improved, figure 11, in this case, the optimal values of incubation temperature, extraction time and desorption time at which a higher total area of the chromatogram was obtained are 49.99 °C, 39.99 min and 8.73 min, respectively.

Figure 12 shows the Pareto chart summarizing the results obtained using the experimental design with fiber DVB/CAR/PDMS. Two factors evaluated were not significant with 95% of confidence and only incubation temperature was a relevant effect. These results were consistent with a thermodynamic perspective as Pawliszyn, 2012 showed, who consider that "Heating the sample not only increases the Henry's law constant but also induces convection in the headspace because density gradients are associated with temperature gradients present in the system, result in higher mass transport rates". It must be highlighted that based on this information these factors were used for the experimental design to obtain the best conditions for the extraction temperature and extraction time.

Figure 13 represents the main effects diagram among the evaluated variables. It was observed that, under the experimental design proposed, the extraction time and desorption time manage to reach a peak, but as shown in figure 12, these two variables and their interaction generate an effect on the response variable, but this effect was not significant. Regarding the evaluated incubation temperatures, it did produce a significant effect, but it did not reach their maximum point or a concavity change. A hypothesis could be generated that this point might be visualized at temperatures above 60°C since, as described in the methodology, the material used comes from fluidized bed drying at 60°C for 5 hours.

Figure 13 *Main effects plot of tested conditions by HS-SPME-GC/MS*

Furthermore, the optimized method or method with the improved variable (incubation temperature: 49.999°C, extraction time: 39.999 min and desorption time: 8.73 min was verified
considering the different parameters established by the United States Pharmacopeia (USP) in its general chapter <1225> Validation of compendial method. The parameters known as linearity (R^2) , repeatability (RMSE), limit of detection (LOD) and limited of quantification (LOQ) were evaluated using the standards of (-)-α-Pinene, (-)-β-Pinene and Eucalyptol, table 20. It observed that the method displays linearity at the range of concentration evaluated with these three standards; the Yaxis of the curve represents the area obtained in the chromatogram and the X-axis represents the concentration of the standard (ppm).

Standards	Equation	Linearity range [ppm]	R^2	RMSE	LOD [ppm]	LOO [ppm]
$(-)-\alpha$ -Pinene	$Y = 4 \cdot 10^7 \cdot X - 1 \cdot 10^7$	$0.414 - 6.624$	0.9973	$5.585 \cdot 10^6$	0.39	1.19
$(-)-\beta$ -Pinene	$Y = 2 \cdot 10^7 \cdot X - 1 \cdot 10^7$	$0.421 - 6.732$	0.9902	$1.205 \cdot 10^{7}$	0.67	2.03
Eucalyptol	$Y = 1 \cdot 10^7 \cdot X + 5 \cdot 10^6$	$0.218 - 5.893$	0.9994	$1.178 \cdot 10^6$	0.21	0.63

Table 20 Analytical characteristics used to evaluate validity of method.

Once the linear equations were established, three compounds were quantified in the different products (dried fruits, extract, prototype) obtained from *Physalis Peruviana*, table 21. Those compounds were identified in fresh material (fresh material classified in 12-13°Bx), fluidized bed dried material (material obtained after dry in a fluidized bed dryer at 60°C and 5 hours) and final ingredient (material obtained after extraction/rotary evaporation/freeze drying process).

<i>Quantification of three compounds followed infoughout the process</i>						
	Concentration $\pm SD$ [ppm]					
Compound	Fresh material	Fluidized bed dried material Extract		Final ingredient		
Eucalyptol	25.811 ± 0.203	25.172 ± 0.677	NF	NF		
$1R-\alpha$ -Pinene	7.540 ± 1.782	5.367 ± 0.701	0.634 ± 0.002 NF			
$(-)-\beta$ -Pinene	9.319 ± 1.127	7.933 ± 0.514	6.373 ± 0.032 NF			

Quantification of three compounds followed throughout the process

NF: not found.            

Table 21

The impact of the fluidized bed drying method on the three quantified volatile components was evident. Eucalyptol is the one that undergoes the least impact by the drying process but it did not remain until the end of the productive process. Additionally, it is observed that with the established extraction method the presence decresead considerably despite they posees solubility in ethanol and low solubility in water. Finally, like Eucalyptol, 1R-α-Pinene and (-)-β-Pinene were not detected in the prototype of ingredient, which may indicate that the development process of an ingredient under the evaluated conditions compromises the presence of volatile substances that may be involved in its final flavor and aroma.

7.4 Drying

7.4.1 Kinetic drying

Drying commonly describes the process of thermally removing moisture to yield a solid product. In many of the cases, the drying process only focuses on examining the removal of moisture from products (fruits, vegetables, grains, etc.) to reach a point of stability with the purpose of obtaining easy to handle free-flowing solids that could be preserved, stored and transported easily and reducing costs. During dehydration, not only the moisture content changes, but there are also losses or variations in the content of vitamins, natural pigments, flavor and texture leading to some degradations, browning and loss of properties (Mujumdar, 2014). In this research, three different drying techniques were applied to obtain the drying kinetic curve of *Physalis peruviana* fruits, figure 1. Also, the effect of the drying technique on volatile components was evaluated too.

During freeze the drying (FD) process it was evidenced that vegetable material must be cut into pieces since dehydration was not possible using intact fruits. In the kinetic curve, in relation to the first and second samples, the loss of moisture was not possible to estimate, maybe because the fruits were previously frozen and at the beginning the freeze drying process was unable to reduce the moisture content of the material, the material may have thawed. After four hours, the moisture content started to decrease, but despite this, at the end of the process (48 h) the moisture had not yet reached its equilibrium point (<20%), it was damp. At this point, the experiment was stopped because it was neither considered nor planned to obtain results beyond 48 hours. Moreover, when contemplating industrial scaling, a drying process requiring such an extensive amount of time will not be deemed technically feasible at an industrial level. In convective drying (CVD) (constant/ ramp temperature) and fluidized bed drying (FBD), it acquired a typical drying curve until reaching an equilibrium moisture content. In convective drying with ramp temperature, the effect of changing the temperature from 60 to 40°C was evident, slightly extending the time necessary for this material to reach its equilibrium moisture. Fluidized bed drying (FBD) takes five hours to dry fruits and it was not necessary to complete 48 h, the final moisture content was below 20%. The materials obtained by FBD, FD, CVD at constant temperature were analyzed in HS-SPME-GC/MS and compared between them.

On the other hand, it was proposed to analyze the effect of temperature on the volatile chemistry of the material exposed to 60°C with a drying ramp in the convective drying equipment. However, this could not be carried out since only one equipment was available (Thermolab C480), and due to the climacteric nature of the material using one batch was not able because of the possibility of guarantee that this material would conserve its properties between treatments with a 48 hour waiting period.

The drying kinetics were modeled under four different isothermal functions (Newton, Gaussian, Henderson & Pabis and Fourier), table 22, and it was observed that the three drying techniques present unique characteristics regarding the total drying time analyzed and the moisture loss at each of these moments. Fluidized bed drying was the one with the shortest time and at the same time, in each analysis, it did not show high deviations and their moisture content gradually decreased, this leads to the evaluated models that have an \mathbb{R}^2 above 0.95, and models like Gaussian and Fourier exhibited a low RMSE. On the other hand, mathematical models applied to freezedrying showed a moderate fit, but they did not achieve a higher value, it was possible that during

the initial six hours the sample were unstable, probably due to its thawing which prevents these functions from fitting to the real value. Finally, convective drying showed a typical drying curve but all the kinetic models used had the poorest fit compared to the other two drying techniques, this was due to none of the models being able to adapt to the curvature of the graph between the 3 and 8 hours marks and the final time of 48 hours, it seemed that these two factors prevent the models from fitting well. 

Table 22 Thin layer drying kinetic models using fluidized bed dryer, convective dryer and freeze dryer applied to Physalis peruviana fruits.

	No Model	Formula	Fluidized bed drying		Convective drying		Freeze drying	
							R-square RMSE R-square RMSE R-square RMSE	
	Newton	$MR = a \cdot t + b$	0.967	29.19	0.385	130.1	0.903	41.9
\mathcal{L}	Gaussian	$MR = a \cdot exp(-(t-b)/c)^{2}$	0.999	4.462	0.759	13.34	0.946	33.86
2	Henderson and Pabis $MR = a \cdot exp(-k \cdot t)$		0.965	29.87	0.809	11	0.945	31.36
$\overline{4}$ DISCO	Fourier	$MR = a+b\cdot cos(c\cdot t) + d\cdot sin(c\cdot t)$	0.999	4.711	0.842	12.15	0.954	34.3

RMSE: root-mean square deviation

Additionally, analyzing the root-mean-square error (RMSE) and R^2 for the tested models, it could be said that the Henderson & Pabis model had a lower deviation between the observed and predicted values for the convective drying. Contrary to this, for fluidized bed drying the models Gaussian and Fourier had the lowest deviation. RMSE for the freeze drying process was the highest value. 

7.4.2 Identification of volatile components in dried material

A total of 65 compounds presents in the fresh material classified in a category of 12-13 soluble solids content were compared with fruits of the same batch that were dried using a convective dryer, freeze dryer and fluidized bed dryer (until their specific equilibrium point according to the dry technique), figure 15.

**Percentages were obtained from 65 components.*

Venn diagram allows correlating the presence of different components identified according to the analyzed material by HS-SPME-GC/MS. The analysis of fresh material led to observed 65 compounds, which were searched in dried material derived from the three types of drying techniques. Twenty of those compounds were not present in any of the three drying treatments, they included (E)-2-Hexenal, Benzaldehyde, Butanol, Butyl ethanoate, Cadinene, Ether benzyl methyl, Ethyl acetate, Ethyl nonanoate, Geranyl acetone, Hexadecanoic acid, Hexanal, Hexyl acetate, Isobutyl butyrate, Isobutyl octanoate, Myristic acid, Octanol, Propyl decanoate, Propyl octanoate, β-Terpinyl acetate and γ-Undecalactone, which represents 30.8% of the fresh *Physalis peruviana* volatile compounds. Regarding lyophilized material, 4 compounds were different from the others materials, they were identified as Butyl ethanoate, 1-Butanol, Isoamyl ethanoate and β-Cymene. Conversely, dried fruit obtained with the fluidized bed dryer just showed an exclusive component recognized as β-Myrcene. Differently that these two treatments, after convective drying 8 exclusive compounds were observed, in other words, they were not present in another drying treatments or in the fresh material, those compounds were: 1,3,8-p-Menthatriene, 3-Methyl butanal, Curcumene, Limonene, Terpineol, Verbenone, β-Linalol and β-trans-Ocimene. Contrasting the compounds present in each drying technique with those observed in fresh material,

it was evidenced that after the convective drying, dried fruits shares 32.2% presence of the volatiles compounds with fresh material, from fluidized bed drying 32.5% and lyophilization 35.4%. It was clear from the above that the loss of components related to the natural flavor of *Physalis peruviana* after a drying process might be unavoidable. However, the three drying techniques showed a similar number of components when compared to the fresh material, however, contributions of the compared compounds to the odor and flavor of the fruit were not considered at this point.

On the other hand, analyzing the components only in the drying techniques applied to a same batch was made, figure 16. Comparing the treatments, it was observed that the final dried materials shared 25 compounds which correspond to 55.6% of similarity, additionally it was observed that each drying technique generates compounds that were not shared among them, which could be said to mean that each method produces a final product with different chemical characteristics. For example, in convective drying, a higher presence of terpenes was noticeable, for lyophilization a higher presence of esters and for fluidized bed drying a mixture of terpenes, alcohols and esters ware observed

Figure 11

Venn diagram for volatile components identified in Physalis peruviana material obtained by convective drying, lyophilization and fluidized bed drying.

**Percentages were obtained from 45 components*

A heat map with the 25 shared compounds between drying techniques was made, figure 17, with a least significant difference test. This analysis allows to compare how the drying method affected the quantity of those shared compounds which was correlated with their respective area obtained by the software Qualitative analysis of Masshunter. This analysis showed that Ethyl octanoate was the most abundant analyte being more representative through lyophilization or fluidized bed drying techniques. (-)-β-Pinene, 4-Terpineol, Camphene, Caryophyllene, Ethyl decanoate, Ethyl dodecanoate, Eucalyptol, Hexane, Nonanal, o-Cymene and δ-Candinene were not affected by any treatment. Likewise, it was observed that fluidized bed drying and lyophilization were the techniques that had the greatest amount of area. Fluidized bed drying had the higher area in 1R-α-Pinene, Terpinolene, β-cis-Ocymene, β-Cyclocitral and β-pinene. Conversely, convective drying only resulted in a greater area for two compounds: Butyl octanoate and Ethyl decanoate.

	Convective	Fluidized bed	Lyophilisation	
$(-)$ - β -Pinene	$61c + 007$ ³	3.2c+007 ^a	$1.9c + 007$ ^a	
1R-a-Pinene	7.3e+007 ab	9.1e+007 a	4,2e+007 b	
4-Terpineol	8.3e+007 ³	7.7e+007 ^a	1.3e+008. ^a	1.61e+009
Butyl hexanoate	$9.0e + 008$ ¹¹	6.5e+007 b	$1.5e+0.08$ b	
Butyl octanoate	9456974.6b	2.8e+007 ab	5.3e+007 a	
Camphene	9534196.3 ⁸	$1.2e+007$ ^a	6673043.2 a	1.41e+009
Carvophyllene	$1.0c + 007$ ^a	5691029.1 ^B	$1.2e+007$ ^a	
Ethyl decanoate	8.0e+008 ^a	6.6e+008 ³	$7.4c + 0.08$ a	
Ethvl dodecanoate	5.7e+007 a	8.3e+007 a	$8.6e+007$ a	1.21e+009
Ethyl hexanoate	$1.5e + 0.08$	1.6c+008 ab	$3.9c + 0.08$ a	
Ethyl octanoate	$1,2e+008$	1.6e+009 B	$1.8e+0.09$ ⁿ	
Eucalyptol	$21c+00s$ ^a	$1.3e+0.08$ a	$1.4c + 0.08$ a	1.01e+009
Hexane	3.4e+008 ^a	$3.2e + 0.08$ a	$2.4e + 0.08$ a	
Methyl decanoate	$1.9e + 008$ ^B	5574980.4b	$2.2e+007$ b	8.10e+008
Methyl octanoate	1.3e+007 b	$1.0e + 0.08$ ^B	8.1e+007 a	
Nonanal	$2.0e + 0.08$ a	$2.7e + 0.08$ a	$2.9e + 0.08$ a	
o-Cymene	8.6c+007 a	$1.2c + 0.08$ a	$1.1c + 0.08$ ²	$6.10e+008$
p-Cymen-8-ol	$5.2e+007$	2.0e+007 ^c	7.8e+007 a	
Terpinolene	6.4c+00s b	9.6e+00x ³¹	$6.0c + 008$ b	
Thymol	$1.2e+0.08$	$4.4e+0083$	b $1.9e + 007$	4.10e+008
ß-cis-Ocimene	$23e+008$ ab	3.7e+008 ^a	$1.7c + 0.08$ b	
B-Cyclocitral	$1.1e+0.08$	9.9e+007 b	$1.5e+0.08$ 21	
B-Ionone	$13e + 007$	$1.2e + 007$	$2.6c + 007$ ^a	2.10e+008
B-Pinene	$6.0e + 0.08$ ¹¹	6,9e+008 3	$3,4c+008$ b	
δ-Candinene	5156203.8 ³	3200443.7 ⁸	3803415.0 ^a	$1.00e + 007$

*Values presented denote the area of components detected through Quality analysis using Masshunter. Different superscripts within the same row indicate significant differences based on the least significant difference test (LSD). significance level: ****p <0.0001, *** p-value < 0.001, ** p-value< 0.01, * p-value<0.1, ns no significant.*

Analyzing the compounds obtained with significant differences between each drying technique and their potential sensory contribution, convective drying could be said to exhibit pronounced notes of pineapple, sweet and fruity characteristics provided by Butyl hexanoate. On the other hand, the material obtained through fluidized bed drying will exhibit odors reminiscent of green pine or herbal, woody and floral notes provided by 1R-α-Pinene, Terpinolene, Thymol, βcis-Ocimene and β-Pinene. Finally, the material obtained by freeze-drying and its high predominant presence of esters will present fruity and floral notes contributed by Butyl octanoate, Ethyl hexanoate, Ethyl octanoate, Methyl decanoate and Methyl octanoate, notes of pine provided by p-Cymen-8-ol, citrusy and lemony notes contributed by β-Cyclocitral and woody notes by β-Ionone.  Considering the objectives and the results, the selection of the drying technique for continuation in the UAE process was based on drying kinetics. Priority was given to achieving the shortest drying time to reach fruits with a moisture content below 20%. Consequently, lyophilization proved to have low viability as it required more than 48 hours. In contrast, the drying kinetics obtained in convective drying and fluidized bed processes showed drying times of less than 12 and 6 hours, respectively. On the other hand, if energy consumption were considered, fluidized bed drying might require less energy since its drying time is shorter. Additionally, it offers processing capacity and can operate in batch and continuous modes. Furthermore, it could be utilized for future scaling and preserve volatile components of interest compared to fresh fruit. Based on the aforementioned reasons, this drying technique was selected to proceed with the ingredient extraction process.

7.5 Ultrasound assisted extraction

7.5.1 Dried material used in ultrasound assisted extraction

Once the drying technique was selected, new dried fruits were obtained through fluidized bed drying from a fresh batch and submitted to an extraction process afterwards. Fruits were monitored by HS-SPME-GC/MS to evaluate the presence of volatile components in both, fresh and dried material, figure 18. It was able to identify similarities and differences between fresh and dried fruits of this new batch, where dried material shares with fresh material 34 compounds, and differ in 12 of them. Fresh material showed the presence of Hexanal, Geranyl acetone, γ-Undecalactone,

Benzaldehyde, Myristic acid, (E)-2-Hexenal, Ethyl acetate, Butanol, Butyl ethanoate, Hexyl acetate, Hexadecanoic acid and β-cis-Ocimene. Meanwhile, 1-Cyclohexene-1-acetaldehyde, 2,6,6- Trimethyl-trans-2-caren-4-ol, 1,4-Dimethyl-δ-3-Tetrahydroacetophenone and p-Mentha-1,3,8 triene with match factor of 804, 840, 893 and 872 sequentially. Fluidized bed dried material, 12 compounds had not been related to the fresh volatile chemistry of *Physalis peruviana*. Nevertheless, both materials shared 58.6% of the compounds detected by HS-SPME-GC/MS which suggests that through this drying technique it was possible to preserve over half of *Physalis peruviana* volatile components.

Figure 13

Venn diagram for volatile components identified in fresh and dried material obtained by fluidized bed.

**Percentages were obtained from 58 components*

Some of the components shared between fresh material and dried material have, figure 19, changed after treatment, as in the case of Isobutyl butyrate, Ethyl nonanoate, γ-Terpinene that decreased their area and of Camphene, Isobutyl octanoate, (-)-β-Pinene, Eucalyptol, Ethyl butanoate, 1R-α-Pinene and Butyl butanoate that increased their area but not significant differences were observed. In the remaining compounds significant changes occurred, those changes include the increase of Terpinolene, ρ-Cymel-δ-ol and o-Cymene and the decrease of Cadinene, Propyl decanoate, Carveol, β-Ionone, Propyl octanoate, β-Cyclocitral, Methyl decanoate, Octanol, Methyl octanoate, 4-Terpineol, Butyl hexanoate, Benzyl methyl ether and Butyl octanoate.

Figure 14 Heat map for volatile components identified and correlated between fresh and dried material obtained by fluidized bed.

	Fresh material		Fluidized bed drying	
Isobutyl butyrate	$2.1e+007$	$\overline{\text{ns}}$	$3.7e + 007$	$6.01e + 009$
Cadinene	$4.9e + 007$	s.	$1.3e + 007$	
Terpinolene	$5.8c + 007$	ی ن	$1.5c + 008$	
Camphene	$5.8c + 007$	n _s	$6.4c + 007$	
Propyl decanoate	$5.9e + 007$	٠	5910000.0	
p-Cymen-8-ol	$7.9e + 007$	\ast	$1.3e + 008$	$5.01e + 009$
Carveol	$9.9c + 007$	۰	7.3e+007	
Ethyl nonanoate	$1.0e + 008$	R _s	$1.4e + 0.08$	
y-Terpinene	$1.3e + 008$	n _s	1.5e+008	
B-Ionone	$1.3e + 0.08$	出业	$1.0c + 007$	
Isobutyl octanoate	$1.3e + 008$	n _s	$1.0e + 008$	
Propyl octanoate	$1.3e+008$	×	6.5e+007	$4.01e+009$
B-Cyclocitral	$1.6e + 008$	***	6.9e+007	
$(-)-B-Pinene$	$1.7e + 008$	n's	1.4c+008	
Methyl decanoate	$1.9e + 008$		1.0e+008	
β-Terpinyl acetate	$2.0e + 008$	n _s	$2.1e+008$	
Octanol	$2.1e + 008$	۰	1.6e+008	
o-Cymene	$2.1e + 008$	ż	3.0e+008	$3.01e + 009$
Eucalyptol	$2.6e+008$	n s	$2.6e + 008$	
Ethyl butanoate	$2.9e + 008$	$\overline{\bf n}$	$2.3e + 0.08$	
$IR-a-Pinene$	$2.9e + 008$	n s	$2.0e + 008$	
Methyl octanoate	$3.5c + 0.08$	×.	$2.2c - 008$	
4-Terpineol	$3.6e + 008$	N 6	$2.1e + 008$	$2.01e+0.09$
Butvl butanoate	$3.8e + 008$	n _s	$2.9e + 008$	
Butyl hexanoate	$6.1e+008$	电电	3.3e+008	
Ether, benzyl methyl	$6.1c + 008$	***	$5.8c + 007$	
Butyl octanoate	$6.5e+008$	出来	$6.1e+007$	
Hexanol	$7.9e + 008$	***	$1.3e + 008$	
Ethyl dodecanoate	$8.2e + 008$	**	$3.1c + 008$	$1.01e + 009$
Ethyl hexanoate	8.6e+008	**	$5.1c + 008$	
B-Pinene	$1.2e + 009$	n ₅	$9.6e + 008$	
a-Terpineol	$1.8c + 009$	***	$1.1e + 008$	
Ethyl decanoate	$2.8c + 009$	●米	$1.4e + 009$	
Ethyl octanoate	6.0e+009		$3.7e + 009$	$1.00e + 007$

*Values presented denote the area of components detected through Quality analysis using Masshunter. Different superscripts within the same row indicate significant differences based on the least significant difference test (LSD). Significance level: ****p <0.0001, *** p-value < 0.001, ** p-value< 0.01, * p-value<0.1, ns no significant.*

Results showed that it was possible to establish operational conditions during a drying process that preserves the flavor preservation of components of interest. Now, It could be considered to modify variables such as the drying temperature to reduce the significant variation of compounds such as β-Cyclocitral and Ether benzyl methyl just as reported by Oliver-Simancas et al., 2020, who obtained a higher proportion of monoterpenes in mango peel using drying temperatures of 45°C.

7.5.2 Ultrasound assisted extraction conditions

Ultrasound assisted extraction process incites the formation of bubbles which expand from their initial size to a critical point before collapsing violently into smaller bubbles. Compression and rarefaction cycle depends on the amplitude of the ultrasonic waves, higher the ultrasonic amplitude greater the number of cavities which in turn results in achieving a maximum extraction yield (Chemat et al., 2017; Oroian et al., 2020). On the other hand, the ethanol concentration directly affects the polarity of used solvent, while absorption of ultrasound depends on the dielectric constant of the solvent and increases with the water content in aqueous ethanol (Pavlić et al., 2019). As an example, ethanol and water had been used to extract antioxidants from *Zyzyphus lotus* fruits, pomegranate peel and rosemary; carotenoids from tomato pomace; natural colors from *Punica granatum;* phenols from *Myrciaria cauliflora* and *Vitis vinifera;* and flavor volatile compounds from *Mentha spicata* and tea (Chemat et al., 2017).

The area of the 30 components found in the final ingredients were analyzed by HS-SPME-GC/MS and a variance analysis was done, table 22. It was found that ethanol concentration affected the extraction in relation to the area of volatile components.

variance anarysis, emanor concentration and amputate used in OAE of I hysaus peruviana.					
	Sum Sq	Mean Sq	F Value	P (>F)	
A: Ethanol concentration	8.24 E18	38.24 E18	6.71	0.0321	
B: Amplitude	1.33 E15	1.33 E15	0.00	0.9745	
AB	3.77 E18	3.77 E18	3.07	0.1178	
Total error	9.82 E18	1.23 E18			
Total (corr)	2.18 E18				
$\alpha > 0.05$					

Table 22 Variance analysis: ethanol concentration and amplitude used in UAE of Physalis peruviana.

p < 0.05.

In figure 20, the effect of ethanol concentration on its ability to extract volatile components present in *Physalis peruviana* dried fruits was observed in a higher concentration that leads to greater extraction capacity. On the other hand, it was observed that the amplitude had almost a negligible effect, this result can be interpreted as if the amplitude had no significant effect (p-value <0.05), It was possible that cavitation did not occur, therefore, fragmentation, sonocapillary effect, erosion, sonoporation, detexturation and local shear stress neither. Chema at al., 2017 suggest that physical parameters affect the acoustic cavitation phenomenon and more specifically cavitation threshold because the initiation of cavitation in a liquid requires that the negative pressure during the rarefaction cycle must overcome the cohesive forces between compounds composing the liquid. In the same way, Santos et al, 2008 proposed a general rule "To achieve the cavitation threshold a minimum intensity/amplitude is required" and "the higher the amplitude the more the analyte is extracted" (Santos et al., 2008).

Figure 20 Main effects plot for area of components identified in the obtained extract.

Developing the ultrasound assisted extraction process through a response surface graph, figure 22. It was found that the variables that allow for a higher yield of area related to the volatile components present in dried *Physalis peruviana* were 80% ethanol v/v and 50% amplitude (46.330 μpp). It should be noted that this optimization was carried out considering the total area for 30 components obtained by HS-SPME-GC/MS, but It could not be claimed that all of them were related or refer to *Physalis peruviana* fresh or dried material*.*

Figure 151 Response surface by factorial design for ultrasound assisted extraction.

On the other hand, this section presents the evaluation of the effect of amplitude and ethanol concentration of UAE on volatile components in *Physalis peruviana* which was called "ingredient" after a rotary evaporation/lyophilization process. . The volatile composition of liquid extracts was estimated too to evaluate the effect of the rotary evaporation/lyophilization on the presence of these components since the final ingredient was obtained by the application of both processes, figure 23.

Figure 22

**Percentage was obtained from 97 components.*

Table 23 identified the exclusive or uncorrelated components for each stage of the developed process. Comparing fluidized bed drying technique with fresh material, it was evident that there were 9 compounds exclusive of heat treatment, although those compounds could be considered off-odor (Cadwallader, 2019) due to not being initially present. Compounds such as α-Terpinolene, Cymene and Curcumene could be associated with herbal and citrus odors in dried material. Likewise, this material underwent darkening which can be described by the Maillard reaction (Somjai et al., 2021). It was observed that the extracts obtained by UAE were affected by rotary evaporation/freezing/lyophilization to obtain a final ingredient, showing 23 compounds that

were not detected before. It could be hypothesized that during rotary evaporation, a hydrolysis process occurred leading to the modification of some metabolites. Alternatively, during storage at -80°C to generate a frozen extract for subsequent lyophilization, instability may have been induced. Another possibility was that the lyophilization process itself induces alterations among these compounds, as it not only sublimates water but also volatile components (Luo et al., 2023) (Luo et al., 2023). Finally, seven compounds were preserved throughout the entire process, and these were Butyl octanoate, Ethyl decanoate, Ethyl dodecanoate, Ethyl nonanoate, Ethyl octanoate, Propyl decanoate and Propyl octanoate, most of them esters.

Table 23

Fresh material (11) Fluidized bed drying (9)		Extract (23)		Final ingredient (10)
$(E)-2$ -Hexenal	$1,4$ -Dimethyl- δ -3-	1,11-Dodecadiene	Ethyl tetradecanoate	1-Menthol
Benzaldehyde	tetrahydroacetophenone	1,3,8-p-Menthatriene	Hexylcyclohexane	2,6,10- Trimethyltetradecane
Butanol	1-Cyclohexene-1- acetaldehyde, 2,6,6-	1-Dodecene	Isobutyl laurate	Butylhydroxytoluene
Butyl ethanoate	trimethyl-	1-Hexadecanol 2-Methyl-1-	Methylundecane	Damascenone
Ethyl acetate	Curcumene	hexadecanol	Nonadecanol	Ethyl isovalerate
Geranyl acetone	p-Mentha-1,3,8-triene	3-Carene	Octylcyclohexane	Hexadecene
Hexadecanoic acid	α -Terpinolene	3-Methylundecane	Tetradecane	Octadecan
Hexanal	β -Caryophyllene	5-Methylpentadecane	lα-Curcumene	sec-Butyl octanoate
Hexyl acetate	B-trans-Ocimene	5-Methyltridecane	α-Tetradecene	Tetradecyl octanoate
Myristic acid	Ocimene	Copaene	β -Cymene	α -Methylionol
γ -Undecalactone	trans-2-Caren-4-ol	Decane	β-Guaiene	
		Dodecane		

Volatile components throughout treatments of Physalis peruviana fruits.

7.5.3 Volatile components in freeze dried ingredient (final ingredient)

The preservation of physical and chemical properties is essential for the development of a flavoring agent, however, subjecting a plant material to specific heating or cooling conditions will inevitably affect its properties (Mujumdar, 2014). In this case, after obtaining the volatile composition of fresh *Physalis peruviana* fruit, fresh fruits were subjected to fluidized bed drying, extraction process with UAE, rotaevaporation, freezing and liophilizatio to obtaining an ingredient. In this case, fluidized dry material, freeze-dried ingredient and fresh material were compared, figure 24.

Figure 23

Venn diagrams: A) Volatile components identified in dried material obtained by fluidized bed and freeze-dried ingredient or final ingredient. B) Volatile components identified in fresh material and freeze-dried ingredient.

Table 23 compared the compounds present in freeze-dried ingredient with those present in fluidized bed dried material, it was observed that they shared 24.6% of the components, which indicates that the final ingredient had a low proportion of the expected metabolites. Indeed, 1- Menthol, Butyl dodecanoate, Hexadecane, Octadecane and so on were not present in the initial material which suggest the development of a flavor and aroma that could be categorized as fruity, sweet, herbal and minty; however, it was not chemically directly related to the chemical characteristics of the initial material. On the other hand, when compare the freeze-dried ingredient with the fresh material an 81% loss of flavor was generated, compounds as β-Terpinyl acetate, Isobutyl octanoate, Butanol, Octanol, γ-Undecalactone, Ethyl butanoate, Isobutyl butyrate, (-)-β-Pinene, 1R-α-Pinene, Hexanal, Benzaldehyde, Butyl ethanoate, Cadinene, Myristic acid, Methyl

octanoate, Terpinolene, (E)-2-Hexenal, Butyl butanoate and so on, were not present in the final material. Different areas of preserved compounds were estimated and compared, figure 24. Nevertheless, compounds that were initially present in great abundance, such as Ethyl octanoate, Ethyl decanoate and α-Terpineol statistically decreased their area. Moreover, Ethyl dodecanoate, Ethyl nonanoate and Propyl decanoate there were no significant changes which indicated that the descriptor aroma depicted as tropical fruity, floral, woody, green vegetable might be preserved.

*Values presented denote the area of components detected through quality analysis using Masshunter. Different superscripts within the same row indicate significant differences based on the least significant difference (LSD) test at a significance level: ****p <0.0001, *** p-value < 0.001, ** p-value< 0.01, * p-value<0.1, ns no significant.*

7.6 Sensory profile

7.6.1 Sensory profile of *Physalis peruviana* **fruit**

Multidimensional proximity sensory profiling is a qualitative method used to identify and select descriptors that help design or identify the characteristic sensory profile of a specific product. Initially, descriptors that characterize the product are defined, and subsequently, the intensity of each descriptor is quantified. Through analysis, a set of relevant descriptors is identified and selected, providing maximum information about the sensory attributes of the product under study. This establishes a sensory profile, with descriptor intensities evaluated by trained judges on a scale from 0 to 5. Overall quality is assessed on a scale from 1 to 3, where 3 represents high quality and 1 represents low quality. These descriptors can be used to compare a product with others of the same type in the market, expressing differences in terms of sensory perception across attributes such as appearance (A), odor (O), flavor (S), texture (T), and overall quality (SS). In this specific case, the analysis involved 6 trained panelists, comprising both men and women aged between 25 and 60 years. Panelists are trained according to the guidelines of GTC 280 (formerly ISO 8586), NTC 4129 (formerly ISO 8586-1), NTC 4130 (formerly ISO 8586-2) for sensory evaluation, olfactory training under NTC 4503 (ISO 5496), gustatory training under NTC 3915 (ISO 3972), texture training according to NTC 4489 (ISO 11036), and specific methodology under NTC 3932 (ISO 11035). The tests were conducted in controlled facilities adhering to GTC 226 (ISO 8589), involving judges with approximately 10 to 20 years of experience, overseen by the panel leader.

According to figure 25, the sensory profile observed were for both fresh fruits of *Physalis peruviana* classified within a range of 12-13 brix, and the flavoring ingredient obtained under the proposed process. In the fresh fruit, there was a predominance of fresh and fruity aromas with medium notes related to acidic taste, highlighting a shiny appearance and homogeneous color. Specific comments from the panelists noted, "The cape gooseberry is slightly overripe but it does not affect the overall sensory quality of the product; it is balanced and fresh. The fruit has a pronounced floral and herbal aroma with a good balance of notes. Bitter taste is perceived in the calyx, along with an astringent sensation, both persistent". On the other hand, for the sensory profile of the ingredient, high notes of fruity and fresh smell were achieved, in addition to possessing a bitter, fruity, and acidic taste, along with the presence of alcoholic notes. Additionally, the specific comments from the panelists were: "a refreshing sensation is perceived. Defined fruity flavor of goldenberry. Alcoholic notes, good balance of notes. The bitter note becomes more pronounced and persistent. Floating particles in appearance. High astringency. Fruity notes persist in the mouth, and it feels like the temperature in the mouth increases slightly. Spicy sensation in the throat. Citric acid profile"

Figure 25

A) *Multidimensional proximity sensory profile of fresh Cape Gooseberrie fruit with 12-13° Brix. B) Multidimensional sensory profile by proximity in diluted hydroalcoholic fruit extract (5.03%).*

Finally, in figure 26 and table 24, the two analyzed materials were compared, showing a significant difference $(p<0.05)$ in the attributes of acidic, fermented, fresh, and fruity smell. These attributes received higher ratings in the fresh goldenberry sample, except for the fermented smell attribute, which received a higher rating in the diluted goldenberry extract sample. There is a significant difference $(p<0.05)$ in the attributes of sweet, fermented, fresh, fruity taste, and the somatosensory sensation of astringency. These attributes received higher ratings in the fresh

goldenberry sample, except for the fermented taste and astringent sensation attributes, which received higher ratings in the diluted goldenberry extract sample.

Figure 26 *Sensory profile of smell, taste, and overall quality in a sample of fresh goldenberry vs. a sample of diluted hydroalcoholic extract (5.03%)*

** Student t-test for independent groups*

9 Conclusions

It was possible to assess different techniques (fluidized bed, convective and freeze drying) for the preservation process of *Physalis peruviana* seeking to guarantee the conservation of the organoleptic properties and the volatile chemical composition that are determinant for the generation of high value fruit-base products or ingredients such as dried fruits or extracts. In addition, it was able to evaluate the impact of cold extraction of dried fruits of *P. peruviana* by high intensity ultrasound under established conditions, however, it was evident that using an hydroalcoholic mixture showed a loss of compounds of interest, which could significantly impact on flavor, aroma, and color. Consequently, although high intensity ultrasound assisted extraction has been documented for its applicability in different plant matrices, it is needed to optimize the extraction parameters (other food grade solvents, time of extraction, amplitude, etc.) to improve the obtaining and concentration of components of interest through the cavitation generated. On the other hand, in search for a prototype flavoring ingredient from *P. peruviana* with organoleptic characteristics and volatile chemical composition of interest, the extracts were submitted to different process determined mainly by the presence of ethanol, but additional thermal treatments such as rotary evaporation impacts importantly on volatile profile of the final explored prototypes.

As estates above, there is evidence to support that the tested conditions for the integration of a drying technique with high-intensity ultrasound-assisted extraction to obtain a flavoring ingredient did not preserve the initial volatile compositions. Nevertheless, more experiments should be carried out to stablish if those preserved volatile compounds are determinant markers of flavor and aroma in *Physalis peruviana* fruits. The fulfilment of this vision requires that a trained sensory panel determine if the obtained prototype possesses organoleptic characteristics proper of *P. peruviana* fruits.

- 1. The presence of volatile components in *Physalis peruviana* showed to be related to its ripeness state, however, it would be appropriate to evaluate different sources or providers, with the aim of knowing how volatile profile differs from one provider to another.
- 2. While identifying volatile compounds based on soluble solids content range worked well to classified fruits, it is recommended to explore alternative classification strategies to avoid the loss of vegetal material inherent to the process used in this work.
- 3. The SPME methodology is divided into three usage forms: headspace, direct immersion, and in-tube SPME. In this research, HS-SPME was used but both, direct immersion and in-tube SPME, could be implemented in order to enhace the identification of components present in this material.
- 4. Drying is a preservation technique, which inappropiately implemented could significantly impact the volatile chemistry of materials. It is recommended to assess additional temperature ranges (constant or in a ramp way) or techniques (microwave drying, infrared drying or the refractory window) to discover how they affect the volatile chemistry of *Physalis peruviana*.
- **5.** Different variables affect high intensity ultrasound assisted extraction process, such as amplitude, intensity, type of solvent, temperature, extraction time, external pressure, direct and indirect ultrasonic application to induce cavitation, which enhances mass transfer rates in the extraction process. It is recommended to continue searching for and modifying these variables, with the aim of reaching the critical point at which cavitation occurs in the extraction of components of interest like those related to flavor and aroma of fruits.
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