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# *STRATEGIES FOR PROCESSING PUMPKIN SEED OIL: DRIYNG, EXTRACTION, AND ENCAPSULATION*

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Thesis presented to the Post-Graduate Program in Food Science and Technology of the Federal University of Ceará (UFC), as a partial requirement to obtain a Doctor's degree in Food Science and Technology. Area of concentration: Science and Technology of Products of Plant Origin.

Advisor: Profª. Drª. Lucicléia Barros de Vasconcelos (UFC).

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*To all the researchers who came before me and paved the way for women to occupy academic spaces.*
*To the memory of my grandfather, José Irineu Leal.*

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*“Onde quer que haja mulheres e homens, há sempre o que fazer, há sempre o que ensinar, há sempre o que aprender” Paulo Freire*

## ABSTRACT

This comprehensive study investigates the optimization of the drying process of pumpkin seeds using ultrasound technology. It evaluates the efficacy of different encapsulating agents in protecting and releasing oil extracted from these seeds, focusing on their antioxidant properties, total phenolics, and instrumental color characteristics. The research highlights the underutilized potential of pumpkin seeds, emphasizing their exceptional nutritional value and exploring methods to extend their shelf life and improve their applicability in food products. Convective drying, despite inducing nutrient loss, is identified as a valuable technique for utilizing these residues. At the same time, oil extraction from pumpkin seeds is proposed as a promising approach due to its high nutritional value. The drying process was optimized through a $2^3 + 3$ central points experimental design, using Statistica 7.0 software, and the effectiveness of ultrasound pretreatment in improving drying was evaluated despite observing vitamin C degradation. The analysis included the evaluation of parameters such as moisture content, water activity, drying time, pH, acidity, vitamin C concentration, protein content, color, antioxidant activity, and the profile of phenolic compounds, with the Page model demonstrating a better fit to the experimental data. Furthermore, the study examines the efficacy of different encapsulating agents - modified maltodextrin (Capsul - C), conventional maltodextrin (M), and a combination of both (CM) - in the protection and release of pumpkin seed oil, using Principal Component Analysis (PCA) to understand the correlations among the studied parameters. An in vitro gastrointestinal tract model was developed to simulate the oral, gastric, and intestinal phases, allowing for a detailed assessment of the microcapsules' release behavior. The results revealed significant differences in antioxidant capacity and oil release among the samples encapsulated with different agents, underlining the influence of the type of encapsulant on the oil's protection and release. Statistical analysis indicated that the lowest release rates occurred in the salivary fluid phase, suggesting that the microcapsule wall composition effectively protects against early degradation.



**Keywords:** ultrasound; experimental design; phenolic compounds profile; encapsulation; controlled release in the gastrointestinal tract; encapsulating agents; modified maltodextrin.

## RESUMO

Este estudo abrangente investiga a otimização do processo de secagem de sementes de abóbora utilizando tecnologia de ultrassom e avalia a eficácia de diferentes agentes encapsulantes na proteção e liberação do óleo extraído dessas sementes, com foco em suas propriedades antioxidantes, fenólicos totais e características de cor instrumental. A pesquisa destaca o potencial subutilizado das sementes de abóbora, ressaltando seu valor nutricional excepcional e explorando métodos para prolongar a vida útil e melhorar a aplicabilidade desses subprodutos em produtos alimentícios. A secagem convectiva, apesar de induzir perda de nutrientes, é identificada como uma técnica valiosa para a utilização desses resíduos, enquanto a extração de óleo de sementes de abóbora é proposta como uma abordagem promissora devido ao seu alto valor nutricional. A otimização do processo de secagem foi realizada através de um desenho experimental $2^3$ + 3 pontos centrais, utilizando o software Statistica 7.0, e a eficácia do pré-tratamento por ultrassom na melhoria da secagem foi avaliada, apesar de observar a degradação da vitamina C. A análise incluiu a avaliação de parâmetros como conteúdo de umidade, atividade de água, tempo de secagem, pH, acidez, concentração de vitamina C, conteúdo proteico, cor, atividade antioxidante e perfil de compostos fenólicos, com o modelo de Page demonstrando melhor ajuste aos dados experimentais. Além disso, o estudo examina a eficácia de diferentes agentes encapsulantes - maltodextrina modificada (Capsul - C), maltodextrina convencional (M) e uma combinação de ambos (CM) - na proteção e liberação do óleo de semente de abóbora, utilizando uma análise de Componentes Principais (PCA) para entender as correlações entre os parâmetros estudados. Um modelo in vitro do trato gastrointestinal foi desenvolvido para simular as fases oral, gástrica e intestinal, permitindo uma avaliação detalhada do comportamento de liberação das microcápsulas. Os resultados revelaram diferenças significativas na capacidade antioxidante e na liberação do óleo entre as amostras encapsuladas com diferentes agentes, sublinhando a influência do tipo de encapsulante na proteção e liberação do óleo. A análise estatística indicou que as taxas de liberação mais baixas ocorreram na fase do fluido salivar, sugerindo que a composição da parede da microcápsula oferece proteção eficaz contra a degradação precoce.



**Palavras-chave:** ultrassom; planejamento experimental; perfil de compostos fenólicos, encapsulação, liberação controlada no trato gastrointestinal, agentes encapsulantes, maltodextrina modificada.


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## ABBREVIATION LIST

| | |
|---|---|
| ABRACEN | Brazilian Association of Central Supply |
| ABTS | 2,2′-Azino-bis (3-ethylbenzothiazoline-6-sulfonic acid) |
| ANVISA | Agência Nacional de Vigilância Sanitária |
| AHA | American Heart Association. Pumpkin seeds pack a healthy punch |
| AOAC | Association of Official Analytical Chemists |
| $a_w$ | Water activity |
| Bi | Biot number |
| Def | Effective mass diffusivity |
| DF | Degree of freedom |
| DPPH | 2,2-diphenyl-1-picryl-hydrazyl-hydrate |
| DT | Drying time |
| FAO | Food and Agriculture Organization of the United Nations |
| Fcal | F calculated |
| FLW | Food Loss and Waste |
| FL | Food Loss |
| FRAP | Ferric Reducing Antioxidant Power |
| Ftab | F tabulated |
| FW | Food Waste |
| GC-MS | Gas chromatography/mass spectral analysis |
| LAFRUTH | Tropical Fruit Laboratory |
| ONU | United Nations Organization |
| PCA | Principal component analysis |
| PPO | Polyphenoloxidase enzyme. |
| PSO | Pumpkin seed oil |
| QM | Medium square |
| RMSD | Mean square deviation |
| $R^2$ | Coefficient of determination |
| US | Ultrasound technology |
| UT | Ultrasound time |
| UA | Ultrasound amplitude |

| | |
|---|---|
| RP-HPLC/DAD | Reversed phase - high-performance liquid chromatography / photoDiode Array Detector |
| SDG | Sustainable Development Goals |
| SQ | Sum of squares |
| TTA | Total Titratable Acidity |
| $\chi^2$ | Chi-square function |

**CONTENT**

## 1. INTRODUCTION

Seeds represent approximately 10% of the total weight of pumpkins and have garnered significant attention in the context of waste utilization due to their high nutritional value and their health-protective activities, including antioxidant, anti-inflammatory, hypoglycemic, and hypolipemic properties (Vinayashree & Vasu, 2021). Given the quantity of pumpkins produced, it can be estimated that approximately 912.2 thousand tons of seeds are treated as waste worldwide within a year, thus making a substantial contribution to environmental pollution (Ferreira et al., 2021).

In this context, the food industry must seek alternatives to promote agro-industrial waste utilization, add value to these by-products, and reduce the environmental issues associated with improper waste disposal (Barros et al., 2023).

One of the major challenges in preserving pumpkin seeds is their high moisture content, which makes them susceptible to chemical, microbial, and enzymatic reactions that detrimentally affect product quality and reduce its shelf life (Santos et al., 2020). Drying is an effective conservation technology that curbs the growth of microorganisms, reduces moisture-mediated degradation reactions, and decreases mass during transportation (Chao et al., 2022).

Despite being an age-old process, many aspects of food drying can still be improved. For instance, an ongoing demand exists to reduce processing time, energy consumption, and costs while enhancing product quality. One way to enhance this process is by employing pretreatments that modify the structure or composition of the food, thereby altering water flow during drying (Miano et al., 2021). Recently, ultrasound technology (US) has commonly been used as a pretreatment method for drying technology. This approach preserves the original quality characteristics of food products, reduces drying temperatures, and minimizes energy requirements. However, there are effects associated with the propagation of ultrasound waves in a liquid medium, such as acoustic cavitation, microjets, and inertial flow, which may cause damage to cell walls and the creation of microchannels (Feng et al., 2019). Therefore, a comprehensive study is required to examine the influence of ultrasound on food processing.

One of the alternatives for utilizing pumpkin seeds is through oil extraction. While pumpkin seed oil is not widely produced in Brazil, it is extensively manufactured in countries such as Slovenia, Hungary, Croatia, and southern Austria. This edible oil is characterized by its composition of fatty acids, including palmitic, stearic, oleic, and linoleic acids (Cuco et al., 2019). In addition to its fatty acid composition, the oil contains various bioactive compounds,

such as phytosterols, phenolics, antioxidants, tocopherols, and low levels of carotenoids (Potočnik et al., 2018).

Although pumpkin is widely consumed in Brazil, there is a scarcity of studies exploring the use of pumpkin seed oil in food applications. Based on this context, the present research aims to assess pumpkin seeds' nutritional and technological potential, promoting their comprehensive utilization.

The thesis is organized into appendices that detail different aspects of the study. The appendices include Appendix A, which presents the title: TECHNOLOGICAL AND NUTRITIONAL POTENTIAL ASSOCIATED WITH THE FULL USE OF PUMPKIN:A REVIEW. Appendix B presents the detailed methodology for the extraction and encapsulation of pumpkin seed oil; Appendix C contains experimental data on the seeds' drying kinetics; Appendix D shows the results of the physicochemical and nutritional analyses of the samples; and Appendix E discusses the application of the encapsulated oil in food products. This structure allows for a comprehensive understanding of the methods used and the results obtained, facilitating the replication and practical application of the findings.

# 2. LITERATURE REVIEW

## 2.1 Agro-Industrial Waste

According to the FAO (2015), nearly 40-50% of fruits and vegetables produced worldwide each year go unconsumed due to post-harvest losses. In response to these concerns, international experts have suggested the need to enhance preservation techniques that provide environmental, economic, and social benefits. This has led the food industry to take a greater interest in valorizing agricultural by-products as potential sources of food ingredients to mitigate this issue and add value to waste (Atencio et al., 2021).

Currently, agro-industrial waste has been employed to obtain and apply bioactive and phytochemical compounds. In addition to the food sector, these compounds find applications in the cosmetic and pharmaceutical industries. Given the substantial availability of these waste materials as a result of extensive food processing in agriculturally based economies, large quantities of various types of waste, such as seeds, peels, and pulp, are generated (Rosa et al., 2020).

Agro-industrial waste and food by-products are generated in significant quantities within the food industry. However, these by-products are often undervalued and discarded, resulting in environmental damage. Despite the considerable waste produced by agro-industries, there is enormous potential for utilizing by-products originating from the processing of plant-based foods, which exhibit high concentrations of bioactive compounds in their composition (Yepes-Betancur et al., 2020).

The industry's interest in making use of waste generated during food processing has grown significantly in recent years due to the nutritional composition found in many of these residues (Aranha et al., 2017). Non-conventional parts (such as peels, stalks, and seeds) often contain higher quantities of macronutrients (carbohydrates, proteins, and dietary fibers) and micronutrients (vitamins, minerals, and bioactive compounds) when compared to their respective pulps (Barroso et al., 2019). In this regard, the utilization of agricultural residues aims to maximize available resources, resulting in the production of new foods with high energy and commercial value, while also offering an environmentally friendly solution to waste reduction.

Given the significant environmental issues associated with waste generation and the nutritional characteristics of plant by-products, there has been an intensified focus on these residues. According to Torres-León et al. (2018), bioactive compounds commonly found in

plant by-products have gained importance due to their ability to promote health benefits, such as reducing the incidence of degenerative diseases like cancer and diabetes, and mitigating risk factors for cardiovascular diseases. Many of these bioactive compounds are phytochemicals, including antibiotics, alkaloids, pigments, and phenolic compounds.

## 2.2 Pumpkin

Pumpkin belongs to the Cucurbitaceae family, which is widely cultivated and consumed worldwide. According to the Food and Agriculture Organization (FAO, 2020), global pumpkin production increased by approximately 110.64% between 1994 and 2018, reaching over 27.6 million tons in 2018. The widespread popularity of pumpkin can be attributed to its sensory and nutritional characteristics, which encompass various components such as pro-vitamin A and carotenoids.

Furthermore, pumpkin pulp exhibits remarkable versatility and can be employed in the production of sweets, jams, and purées. Nevertheless, the processing of pumpkin generates significant quantities of peels and seeds, often referred to as by-products. Despite their frequent disposal, these by-products possess functional properties and can be utilized as nutritional supplements or food ingredients (FAO, 2018; Lima et al., 2021).

## 2.3 Pumpkin Seeds

Pumpkin seeds contain a substantial amount of proteins, fibers, lipids, minerals, and other nutrients such as starch. However, their utilization by humans is relatively low and less explored, primarily due to the presence of various antinutritional factors, including trypsin inhibitors, saponins, and tannins. These antinutritional factors diminish the bioavailability of nutrients, particularly proteins (Ferreira et al., 2021).

Nutritional compounds contribute to the reduction of essential nutrient bioavailability, resulting in a discrepancy between the nutritional values in the food and those that can be absorbed by the body. The main antinutritional factors present in grains and seeds are phytates, protease inhibitors, tannins, and non-starch polysaccharides. Phytic acid strongly binds to mineral cations such as iron, calcium, zinc, magnesium, and manganese. These chelating properties lead to the formation of phytate-mineral complexes, subsequently altering their solubility, functionality, absorption, bioavailability, and digestibility (Madsen and Brinch Pedersen, 2016; Mohammadi et al., 2020).

Modern consumer lifestyles have led to an increase in degenerative disorders or chronic diseases, such as diabetes and heart diseases, necessitating changes in dietary habits. The consumption of functional foods can be an alternative to mitigate these disorders or contribute to their prevention. Functional foods are natural or processed foods that contain known or unknown bioactive compounds in defined, effective, and non-toxic quantities, providing clinically proven health benefits for the prevention, management, or treatment of chronic diseases. Due to their nutrient content, pumpkin seeds can be considered functional foods (Luna-Guevara et al., 2017).

Antioxidant compounds, typically found in plant-based foods, have the ability to inhibit or delay degradative reactions caused by free radicals. However, these compounds are sensitive and can be affected by processing methods or even pre-treatments (Infante et al., 2021).

## 2.4 Pumpkin Seed Oil and Protein

In recent years, pumpkin seed oil has been widely used as cooking oil in some West African and Middle Eastern countries, for salad dressing, and in the production of margarine, gaining attention due to its high nutritional value. These seeds contain a significant amount of oil that is rich in unsaturated fatty acids and bioactive substances, including α-tocopherol, γ-tocopherol, phytosterols, β-carotene, squalene, and other compounds (Can-Cauich et al., 2019).

The chemical composition and yield of vegetable oil are influenced by the extraction method, pre-treatments, the plant part used, and fragmentation, which can increase the extraction yield of compounds of interest due to the larger contact surface area (Governici et al., 2020).

Consumption of pumpkin seed oil offers numerous health benefits and can be used as an herbal remedy and high-quality vegetable oil. The extraction of oil from pumpkin seeds results in a by-product known as defatted pumpkin seed flour, which is rich in proteins. Studies have shown that the proteins present in pumpkin seeds have physiological functions, such as inhibiting bacterial growth, reducing inflammation, and blood sugar levels (Wang et al., 2021). The intake of pumpkin seed oil is also associated with the prevention of cardiovascular diseases, prostate growth, and complications in postmenopausal women (Wong et al., 2019).

Many studies have also demonstrated the potential of by-products such as bran, defatted flours, and leaves of cereals, legumes, and oilseeds (wheat, barley, soy, peanuts,

canola, peanuts, flaxseed, grape seeds, sesame seeds, cottonseeds, pumpkin seeds, and sunflower) as a source of plant-based protein for human consumption. The utilization of plant protein extracted from any of these parts requires in-depth study to assess their functional properties and essential and non-essential amino acid composition (Kumar et al., 2022).

### 2.5 Convective Drying

Most post-harvest losses of agricultural products occur primarily due to inappropriate or inefficient preservation techniques. Among various preservation techniques used in agro-industrial processing, drying is of paramount importance as it integrates several food-processing systems and is one of the oldest and most commonly used methods (Surendhar et al., 2019).

Fresh pumpkin seeds are highly perishable due to their high moisture content, necessitating preservation methods after pulp separation. As an alternative, drying can be employed as a unit operation to reduce moisture content, preventing processes like germination, microbial, and physicochemical degradation, thereby maintaining the initial quality of the seeds during storage (Santana et al., 2022).

Convective drying is a unit operation often carried out at the industrial level in the production of various food products. However, conventional drying methods (based on prolonged exposure to hot air) can result in product deterioration and reduced quality, while requiring a substantial amount of energy during the process, leading to significant economic and environmental impacts (Carvalho et al., 2021).

According to Quequeto et al. (2021), drying represents a significant fraction of industrial energy usage, ranging from 27% to 70%, depending on the type of processed product. Therefore, its rational use is essential. The high-energy demand is associated with the heat required to remove moisture from grains, the heat transfer capacity between the drying air and the product, and energy losses associated with most industrial dryers.

The drying process involves a combination of conduction mechanisms (due to contact with the dryer's surface) and convection (due to the circulation of hot air around the sample), resulting in a higher mass transfer coefficient. These processes are characterized by complex interactions of heat and mass transfer (Von Gersdorff et al., 2020; Rajoriya et al., 2021).

## 2.6 Drying Kinetics

Despite the numerous advantages associated with food drying, the process induces several changes in the product, primarily observed in terms of texture, taste, aroma, color, and nutritional quality. This is because some substances are degraded when exposed to high temperatures. Therefore, it becomes essential to conduct studies on drying processes and systems to determine the conditions that yield a product of higher quality (Santos et al., 2019).

Furthermore, research related to drying kinetics, mathematical modeling, and effective diffusion is crucial for modeling the processes and determining parameters that can be applied to scale-up and equipment design. It also allows for predicting moisture content over the course of the process (Santana et al., 2022).

Drying kinetics consider only the dimensionless moisture ratio to ensure that differences in the initial moisture content of the samples do not interfere with the drying curves. Internal resistance to moisture transport decreases with an increase in drying temperature due to increased water molecule mobility within the food. External resistance decreases due to the increased water pressure gradient between phases. Furthermore, a high drying temperature provides the energy to overcome the latent heat of phase change that occurs during moisture evaporation (Corrêa et al., 2017).

## 2.7 Ultrasound

The lengthy periods of the dehydration process, high-energy consumption, and poor quality of the final product obtained by conventional hot air drying have contributed to the recent popularity of new non-thermal and eco-friendly treatments. Convective drying assisted by additional processes (Hybrid Drying), particularly pre-treatment with ultrasound, can be used to overcome the problems associated with convection drying (Bagheri and Dinani, 2019). For example, the ultrasonic process enhances the preservation of food quality attributes by reducing the time required for the completion of the thermal treatment (Xu et al., 2021).

Despite initial tests of applying ultrasound in food drying operations beginning in the 1950s by Boucher and Greguss, this technology is considered modern. The ultrasound process is present in various sectors and is constantly evolving in the food industry. Ultrasonic waves are generated by piezoelectric transducers that convert electrical energy into mechanical vibrations, defined as high-frequency mechanical sound waves beyond the natural human

hearing limit (20-100 MHz), causing changes in a substance (Ogutu and Mu, 2017; Xu et al., 2021).

The application of the ultrasound process induces fluid cavitation within the product due to an energetic phenomenon that disrupts the structure, forming new channels and pores inside food products. This phenomenon improves mass and heat transfer through mechanisms such as microjets, acoustic agitation, and inertial flow. However, the intensity of the effects provided by ultrasound depends on the conditions under which the product is processed and the medium through which ultrasound waves pass, with water being the most commonly applied medium for transmitting ultrasound waves in products (Miano et al., 2021).

According to Bagheri and Dinani (2019), the ultrasound process is defined as the propagation of sound waves in a medium with frequencies ranging from 20 kHz to 100 MHz, which are beyond the audible frequency range for humans.

The application of ultrasonic in solid-liquid systems generates various phenomena, such as the sponge effect (alternating expansions and compressions in the solid sample) and cavitation (increased temperature and pressure, shear rates, formation of free radicals, moisture reduction, and weakening of cell membranes), leading to the formation of micro channels (Bagheri and Dinani, 2019).

Cavitation (implosion of bubbles) in a liquid occurs when ultrasound waves are of high power and low frequency (frequencies of 20 to 40 kHz) and is considered the primary phenomenon of high-power ultrasound (Bagheri and Dinani, 2019). Ultrasonic waves passing through a solid-liquid system create alternating periods of positive and negative pressure, creating a zone of mechanical stress characterized by sequential increases and decreases in local pressure, leading to the boiling of the liquid and the formation of numerous bubbles using the surrounding air (Cao et al., 2019). These cavitation bubbles remain stable until their internal pressure exceeds their surface tension, at which point the cavity (microbubbles) approaches a vacuum state, leading to the progressive growth and violent implosion of bubbles with an increase in local temperature (5000 K) and pressure (1000 atm). The collapse of cavitation bubbles in food weakens and ruptures cell membranes, facilitating moisture release from the product and affecting the physical and chemical parameters of food (Xu et al., 2021).

Another consequence of this phenomenon is the generation of shear, moisture micro extraction, and the formation of free radicals due to the splitting of water molecules during these implosions. Additionally, the cavitation mechanism is efficient in removing highly bound water molecules (Tüfekçi & Özkal, 2017; Xu et al., 2021).

The sponge effect consists of cycles of mechanical compression and decompression of samples when subjected to ultrasonic waves (Andrés et al., 2021). Generally, the forces involved in this mechanical stress are greater than the surface tension that holds water molecules within the food matrix, thereby reducing the diffusion boundary layer and the initial moisture content (Xu et al., 2021). Additionally, the food structure directly influences the occurrence of the sponge effect, as foods with thin, soft, and porous tissues are more susceptible to this phenomenon compared to thicker and denser biological products (Zhang & Abatzoglou, 2020).

The microscopic channels (micropores) are formed as a result of cavitation and the sponge effect. Microjets produced by the atomization of bubbles inside and outside the sample, as well as their mechanical contraction and stretching, result in the formation of microscopic paths used to maximize mass and heat transfer during the drying process (XU et al., 2021).

According to Mohammadi et al. (2020), there are numerous studies that demonstrate the beneficial effects (enzyme inactivation, reduction of antinutritional compounds, retention of compounds, and reduction in drying time) of ultrasound in the drying process of plant-based products such as garlic (Feng et al., 2019), pumpkin (Rojas et al., 2020), zucchini (Bagheri and Dinani, 2019), jackfruit (Wu et al., 2021), and sunflower seeds (Dibagar et al., 2020).

# 3.OBJECTIVES

## 3.1 General Objectives

To assess the nutritional and technological potential of pumpkin seeds, promoting their comprehensive utilization.

## 3.2 Specific Objectives

• Perform the optimization of the drying and ultrasound process of pumpkin seeds through experimental design with the following independent variables: drying air temperature, amplitude, and ultrasound time.

• Determine, among the mathematical models of Page, Henderson & Pabis, and Midilli, which one best fits the experimental data obtained.

• Determine effective diffusivity, Biot number, mass convective coefficient, and spatial distribution of moisture during the process.

• Characterize pumpkin seed flours in terms of: moisture, water activity, drying time, pH, acidity, vitamin C, proteins, lipids, color, bioactive properties, phenolic profile, and fatty acid profile.

• Identify the influence of ultrasound and drying variables on the quality of the flours.

# FINAL CONSIDERATIONS

This thesis addressed several key questions:

a) Is it possible to define an optimized experiment that considers both economic and nutritional aspects? b) How does the ultrasound process contribute to the optimization of the pumpkin seed drying process, and in what way? c) Can heat and mass transfer during the process be predicted through mathematical modeling? d) Which mathematical model provides the most reliable predictions? e) What is the nutritional composition of pumpkin seeds? f) Can the influence of ultrasound amplitude and time on the nutritional properties of the flours be identified?

The research aimed to provide answers to these questions, shedding light on the feasibility, effectiveness, and nutritional impact of ultrasound-assisted drying of pumpkin seeds. The study demonstrates that using ultrasound as a pre-treatment in the convective drying of pumpkin seeds is an effective approach for producing flour and maximizing the utilization of this byproduct.

The optimized conditions involve subjecting the seeds to 15 minutes of ultrasound at a 70% amplitude, followed by convective drying at 70°C, resulting in reduced water content and improved product quality. Additionally, the study indicates that mathematical models, such as the Page model and the diffusion model, provide accurate predictions for the drying behavior of pumpkin seeds, facilitating the optimization of the drying process.

Overall, this research reveals the potential of ultrasound-assisted drying for enhancing the utilization of pumpkin seeds, offering benefits in terms of improved drying efficiency, product quality, and storage. The findings contribute to the understanding of how this innovative approach can be applied in the food industry, and ongoing research aims to explore its impact on various quality parameters and oil extraction from pumpkin seeds.

As suggestions for future research, it is recommended to evaluate the antimicrobial capacity of pumpkin seed oil and to encapsulate the oil. Furthermore, it is suggested to apply the encapsulated oil as a raw material in other food products and characterize the nutritional aspects of the encapsulated material. These investigations can provide valuable insights into the potential use of pumpkin seed oil as a natural antimicrobial agent and its application in food products, further expanding our knowledge of the properties and benefits of this food resource.

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# APPENDIX A - PAPER 1- TECHNOLOGICAL AND NUTRITIONAL POTENTIAL ASSOCIATED WITH THE FULL USE OF PUMPKIN: A REVIEW

## ABSTRACT

This review article addresses the importance of interdisciplinary approaches to achieve the Sustainable Development Goals (SDGs) established by the United Nations (UN), highlighting the interdependence of SDGs 02, 04, 09, 12, and 17. Furthermore, an in-depth investigation was conducted on the vast potential of fully exploiting pumpkins, emphasizing their positive impact on the SDGs, particularly SDG 2 (Zero Hunger and Sustainable Agriculture). We explore studies of all parts of the pumpkin (skin, flesh, and seeds), highlighting their key nutritional characteristics and technological potential to promote waste utilization and the development of new products. Additionally, a comprehensive analysis of previous research related to pumpkins was conducted, with special emphasis on the use of byproducts, major challenges, and advantages. This study concludes that pumpkins, as a widely cultivated and consumed food resource, play a vital role due to their low production cost. Their byproducts, including the skin and seeds, have significant nutritional and technological value. In addition to being sources of proteins, essential amino acids, and fatty acids, these byproducts find various applications in the food industry, ranging from the production of natural colorants to the manufacturing of pectin-rich biodegradable films. In synthesis, the full use of pumpkin high potential for application in the food industry, for the development of new products and inputs with attractive nutritional, functional and bioactive characteristics.

**Keywords:** Sustainability; Agro-industrial waste; Sustainable development goals; Zero hunger.

## 1. Introduction

Consumers are increasingly seeking natural and healthier foods. The consumption of plant-based foods has been rising, exemplified by the increased cultivation of pumpkins in Asia, Europe, and South America (Wang et al., 2021).

Pumpkin (*Cucurbita maxima*), a member of the *Cucurbitaceae* family, is widely consumed worldwide, annual production exceeding 22 million tons; it is mainly used for fresh consumption and as a raw material for the production of purees, juices, jellies, and alcoholic beverages (Pacheco et al., 2023). There has been a significant increase in global pumpkin production due to its low production cost and attractive nutritional characteristics, including minerals, vitamins, dietary fibers, and carotenoids (Shikder et al., 2023). However, the increased production of these products has led to a rise in waste generation, such as seeds and peels (Wang et al., 2021).

Given the pressing need to address environmental issues caused by waste generation, and plant byproducts' nutritional attributes, these residues have been subject to more intensive research (Torres-León et al., 2018). Furthermore, the utilization of agro-industrial waste can contribute to eradicating hunger and ensuring food security, aligning with the United Nations Sustainable Development Goals (Arshad et al., 2022).

One of the major challenges in the food industry is promoting the use of technologies to reduce post-harvest losses of plant-based products while developing products with high nutritional quality and good stability during storage (Cartagena et al., 2024).

In light of the Sustainable Development Goals (SDGs) (a set of 17 global objectives established by the United Nations in 2015 as part of the 2030 Agenda for Sustainable Development) 02 (Zero Hunger), 09 (Industry, Innovation, and Infrastructure), 17 (Partnerships for the Goals), 12 (Responsible Consumption and Production), and 04 (Quality Education), it is crucial to recognize the interdependence of SDGs and the importance of multidisciplinary actions to ensure food security. This review presents strategies to fully exploit pumpkins and provides an overview of the applied technologies and how they can contribute to the SDGs.

## 2. Sustainable Development

The United Nations adopted the Sustainable Development Goals (SDGs) in 2015, as a global call to action to eradicate poverty, protect the environment, and ensure that everyone can live in peace and prosperity by 2030 (Arshad et al., 2022).

These challenges have become even greater in the aftermath of the COVID-19 pandemic, which has led to increased malnutrition rates, particularly in more vulnerable communities (De Albuquerque et al., 2022). Factors such as loss of productivity, increased production costs, and oil prices linked to climate change (Kakaei et al., 2022) have contributed to this situation.

The economic and health crisis in Brazil has pushed many families into extreme poverty, significantly affecting their access to food in terms of quantity and quality. The VigiSAN Survey, conducted by the PenSSAN Network in late 2020, revealed a drastic worsening of food insecurity nationwide. Over 55% of Brazilian households experience some food insecurity, a 54% increase since 2018. Severe food insecurity affected 9% of households, equivalent to 19 million Brazilians (FAO, 2023).

Ribeiro et al. (2023), when assessing adult food consumption trends in Ceará, from 2015 to 2020, found a decrease in the number of adults consuming three meals a day. However, they also noted an increase in the consumption of fruits and vegetables.

Despite the availability of food preservation techniques against spoilage, losses and waste continue to exacerbate the issue of hunger. In a world where 690 million people are hungry, approximately 8.9% of the global population, the United Nations Food Waste Index report shows that about 931 million tons of food are wasted yearly (Karwowska, 2021).

## 3. Sustainable Development Goals

ONU member states, including Brazil, have committed to achieving sustainable development goals by 2030 (Dangles and Struelens, 2023). Eliminating hunger, achieving food security, improving nutrition, and promoting sustainable agriculture stand out among the commitments made. However, realizing these sustainable development goals is still far from being achieved (Salles-Costa et al., 2022).

Analyzing Figure 1, the interdependence of the five sustainable development goals benefited from fully utilizing agro-industrial waste becomes evident.


**Figure 1 -** Sustainable development goals related to the utilization of agro-industrial waste.

**Source:** Adapted from STF (2023).

According to Rodrigues and Fernandes (2023), sustainable development goals can only be achieved through broad improvements in health, education, and working conditions to ensure environmental preservation and sustainable economic growth (Rodrigues and Fernandes, 2023). In this regard, the SDGs are interconnected and can only be achieved through multidisciplinary actions.

Goal 2 (Zero Hunger and Sustainable Agriculture) encompasses the utilization of byproducts, sustainable agriculture, ensuring access to nutritious food, and food security for all. On the other hand, Goal 12 (Responsible Consumption and Production) primarily aims to halve global per capita food waste by reducing food losses throughout the production and supply chain, including post-harvest losses. Thus, we can demonstrate the similarity and complementarity of these goals (STF, 2023), aligning with the research purpose presented here.

According to Amicarelli and Bux (2020), especially concerning zero hunger (SDG 2), responsible consumption and production (SDG 12), it is impossible to ensure that the global population will have access to sufficient, safe, and nutritious food without rebalancing the food system, making it fairer, healthier, and more environmentally sustainable. This highlights the importance of developing new products using agro-industrial waste as a raw material to enhance the nutritional content of food matrices.

The quality and impact on consumer health of food matrices are determined by

various chemical compounds, such as fibers, proteins, fats/oils, vitamins, minerals, water, polyphenols, antioxidant capacity, and food additives, as well as how these components combine in meals and diets (Santos et al., 2021). Epidemiological evidence of diet-related diseases has increased awareness of the need to improve the nutritional quality of foods consumed (Aljahani et al., 2022). To meet the demands of consumers seeking protein-rich, fiber-rich, and bioactive agent-rich foods, the food industry has been incorporating ingredients from various fruits, vegetables, and legumes into its products.

According to FAO (2021), Food Insecurity occurs when people lack secure access to sufficient safe and nutritious food for normal growth and development and an active and healthy life. Therefore, it is clear that promoting access to food for everyone and ensuring proper nutrition is essential for improving people's quality of life.

Goal 4 (Quality Education) has the premise of ensuring inclusive, quality, and equitable education and promoting lifelong learning opportunities for all, including sustainable development and sustainable lifestyles, human rights, gender equality, the promotion of a culture of peace and non-violence, global citizenship, and appreciation of cultural diversity and the contribution of culture to sustainable development (STF, 2023). Education is the starting point for promoting sustainable development, as it provides learning opportunities for the academic community and the general public, especially when there is a well-established relationship between research groups and active producers in the state.

Goal 9 (Industry, Innovation, and Infrastructure) pertains to building resilient infrastructure, promoting inclusive and sustainable industrialization, and fostering innovation (STF, 2023). All the aforementioned goals can only be achieved through the practice of Goal 17 (Partnerships for the Goals). This goal aims to implement an interdisciplinary approach with multi-sectoral partnerships to promote the sharing of knowledge, expertise, technology, and financial resources. Moreover, it seeks to effectively stimulate and facilitate public, public-private, and civil society partnerships, based on the experiences gained through the resource mobilization strategies of these collaborations to promote the application of new technologies.

## 4. Addressing the Global Challenge of Food Loss and Waste

Despite increased corporate and public awareness of Food Loss and Waste (FLW), the FAO reports that approximately one-third of globally produced food, equivalent to about 1.3 billion tons of edible food, is still lost or wasted (Baysal and Ülkü, 2022; Ishangulyyev, Kim, and Lee, 2019). There is no common agreement on the definition of Food Loss (FL) and Food Waste (FW). However, the FAO (2019) defines FL as the losses in the food supply chain from harvest to (but not including) the retail level, while FW refers to losses that occur in the final stages of the chain, such as distribution, sale, and consumption.

According to the Brazilian Association of Central Supply - ABRACEN (2022), Brazil faces losses amounting to approximately 30% of food after harvest, making it one of the top ten countries with the highest rates of food waste. Therefore, simply increasing food production to meet the needs of a growing population would be ineffective unless measures for preventing and reducing Food Loss and Waste (FLW) are simultaneously addressed.

Most post-harvest losses of agricultural products occur mainly due to inadequate or inefficient conservation techniques (Surendhar et al., 2019). According to Costa et al. (2022), reducing food waste is crucial to enable a more sustainable food economy and, consequently, a sustainable society.

## 5. Full Utilization of Pumpkin

With the continuous development of the economy, the optimization of dietary structure, and the increase in per capita disposable income, significant changes have occurred in food consumption. One of the most noticeable changes is the growing consumer preference for more nutritious, healthy, and practical foods (Singh and Krishnaswamy, 2022).

Various research studies have shown that food is increasingly enriched with high-nutrient ingredients, such as proteins and fibers from sources like beans or whey protein. These trends lead the food market, while fruits and vegetables receive less attention in terms of research and development (Fan et al., 2023).

Pumpkin belongs to the Cucurbitaceae family and is widely cultivated and consumed worldwide. According to the Food and Agriculture Organization (FAO, 2020), global pumpkin production increased by about 110.64% between 1994 and 2018, with over 27.6 million tons produced in 2018. The popularity of pumpkin is attributed to its sensory and nutritional characteristics, include various components such as provitamin A and carotenoids.

In addition, pumpkin pulp is highly versatile and can produce sweets, jams, and purees (Lima et al., 2021).

According to Shikder et al. (2023), another reason for the popularity of pumpkin is its low production cost. Being produced in large quantities in Brazil and forming part of the Brazilian diet, particularly in the Northeast region, pumpkin has various attractive features for its full utilization. However, the pulp, seeds, and peel are highly perishable in their natural state due to their high water content. Therefore, technologies are required to reduce the water content, preventing seed germination and microbial and physicochemical degradation, favoring quality maintenance for a longer period (Santana et al., 2022).

Agro-industrial waste has been used to obtain and apply bioactive and phytochemical compounds. These compounds are used in the cosmetics and pharmaceutical industries. Due to the high availability of this waste from extensive food processing in agriculture-based economies, large quantities of different types of waste, such as seeds, peels, and pulp, are generated (Rosa et al., 2020).

The food industry generates agro-industrial waste and food byproducts in large quantities. However, these byproducts are commonly undervalued and discarded, causing environmental damage. Despite waste generated by agro-industry, there is significant potential for utilizing byproducts from the processing of plant-based foods, which have high nutritional quality with a significant availability of bioactive compounds in their composition (Yepes-Betancur et al., 2020).

Non-conventional parts (peels, stems, seeds), often have higher quantities of macronutrients (carbohydrates, proteins, and dietary fibers) and micronutrients (vitamins, minerals, and bioactive compounds) than their respective pulps (Aranha et al., 2017). In this sense, the utilization of agricultural waste aims to maximize available resources, producing new, high-energy, and commercially valuable foods, representing an alternative for reduction of an environmental problem (Barroso et al., 2019).

Studies also demonstrate the potential of byproducts such as bran, defatted flours, and leaves from cereals, legumes, and oilseeds (wheat, barley, soy, peanuts, canola, peanuts, flaxseed, grape seed, sesame seed, cotton seed, and pumpkin seed) as sources of protein for human consumption (Kumar et al., 2022).

With these new demands, the food industry has shown greater interest in valuing agricultural byproducts as potential food ingredients, promoting waste reduction and adding value to the waste (Atencio et al., 2021).

### 6.1 Nutritional and Technological Potential of Pumpkin Pulp

Pumpkin pulp is rich in micronutrients, including bioactive compounds, especially polyphenols and carotenoids. The concentration of these components varies depending on the species, ripeness stage, soil nutrition, and climatic conditions (Yang et al., 2022). These compounds are fundamental in maintaining cellular health and act as antioxidants against free radicals, making them recommended for a healthy diet (Ali et al., 2019).

Advanced processing of pumpkin has gained prominence due to its extensive production. Most of the processing is done for the manufacture of purees, sweets, juices, and dehydrated pumpkins (Wang et al., 2023). The pulp can be used as raw material for developing plant-based foods, and ingredients in processed products such as snacks and sauces.

García-Parra et al. (2016) assessed the effects of high pressure on the thermal processing of pumpkin puree, focusing on bioactive compounds such as carotenoids, polyphenols, antioxidant activity, and the polyphenoloxidase (PPO) enzyme. They found that purees treated with high pressure achieved higher microorganism inactivation, and the applied treatments significantly reduced PPO activity.

Ebrahimi et al. (2022) found that bread formulations containing 20% pumpkin puree showed superior technological and nutritional characteristics compared to the control formulation, especially in parameters such as hardness, porosity, total color difference, phytic acid content, and overall acceptability. Ahmed et al. (2023) fortified yogurt with β-carotene using pumpkin pulp.

Aydin et al. (2022) developed pumpkin chips by drying pumpkin pulp with added aromatic spices, resulting in a high total phenolic content, primarily composed of phenolic acids. Therefore, thanks to their beneficial health properties, these pumpkin-based snacks represent an excellent snack alternative suitable for all age groups.

### 6.2 Nutritional and Technological Potential of the Peel

Like pulp, byproducts such as peels and seeds also have properties nutritional and technological interest. Nutritionally, they contain significant amounts of proteins, essential amino acids, and fatty acids (Hussain et al., 2022).

Studies related to the technological application of pumpkin peels. Lima et al. (2021) investigated the extraction and encapsulation of carotenoids for natural dye production. Sebdani and Abbasi (2023) used ultrasound technology to extract carotenoids from the peels and pulp of pumpkins for natural pigment production and application in the food industry.

Lalnunthari et al. (2019) found that pumpkin peels are rich in pectin and can produce biodegradable films. Pumpkin peels and seeds have the potential to be converted into useful products with higher added value.

Pumpkin peel, due to its high pectin content, can be used as a gelling or thickening agent. Its incorporation into cereal flours for the development of baked products such as bread, cakes, and cookies help maintain the desired flavor, sweetness, and deep yellow color. It can also be added to soups, sauces, and extruded products to improve product quality (Garg et al., 2023; Roongruangsri and Bronlund, 2015; Cumarasamy et al., 2002).

Mishra and Sharma (2019) noted that pumpkin peel contains considerable protein, fiber, ascorbic acid, and calcium. Therefore, the use of pumpkin peel, along with other flours, can lead to the production of nutritionally enhanced food products.

### 6.3 Nutritional and Technological Potential of Seeds

Pumpkin seeds (*Cucurbita sp.)* have high utilization potential as they are considered a complete food, rich in macronutrients such as lipids (31.5-51.0%) and protein (24.0-36.5%), as well as micronutrients like minerals, phenolic compounds, carotenoids, phytosterols, and tocopherols (Sá et al., 2023). Barros et al. (2023), in their study of pumpkin seed potential, found that the predominant fatty acids were linoleic, oleic, stearic, palmitic, and nonadecanoic acids.

Despite their significant nutritional value, human consumption of pumpkin seeds is relatively low. It is estimated that approximately 912.2 thousand tons of seeds are treated as waste worldwide in a year, contributing significantly to environmental pollution (Ferreira et al., 2021). Therefore, one of the main focuses of the food industry has been the holistic utilization of plant-origin products (Singh & Kumar, 2023).

#### 6.3.1 Pumpkin Seed Oil

Pumpkin seed oil (PSO) can serve as a new source of edible oil to meet the growing demand for functional oils in the food industry due to its substantial content of essential

bioactive compounds (Singh & Kumar, 2023). PSO, known for its thick viscosity and potent nutraceutical effects, has already found applications in the pharmaceutical industry (Singh & Kumar, 2023) but remains relatively unexplored in the agri-food sector.

Pumpkin seed oil has long been used as a cooking oil in some West African and Middle Eastern countries, for salad dressing, and in the production of margarine, gaining attention due to its high nutritional value. The seeds are rich in oil containing unsaturated fatty acids and various bioactive substances such as α-tocopherol, γ-tocopherol, phytosterols, β-carotene, squalene, and other compounds (Can-Cauich et al., 2019).

The chemical composition and yield of vegetable oils are influenced by the extraction method, pre-treatments, plant part used, and fragmentation, which can enhance the extraction yield of interest compounds due to increased surface contact area (Goverinici et al., 2020). Research in vegetable oil extraction is crucial for optimizing processes, increasing production efficiency, and improving product quality. Such investigations benefit the industry, and the economy, and promote sustainable practices.

Pumpkin seed powder and oil can be incorporated into various foods to enhance their nutritional value, quality properties, and lipid stability while storing of meat products like burgers. There is also potential for adding microencapsulated pumpkin seed oil to emulsions, such as mayonnaise (Rojas et al., 2019). In another application, Syam et al. (2020) used pumpkin seed flour to produce biscuits in another application.

### 6.3.2 Defatted Flour

The mechanical pressing process of pumpkin seeds yields pumpkin seed oil, leaving behind a byproduct known as defatted cake, which represents the fat-free portion of pumpkin seeds and contains a high protein content (up to 65%) (Bučko et al., 2016). This defatted cake is a byproduct of vegetable oil processing and serves as a considerable source of plant protein. Pumpkin seeds, as a whole, are also regarded as a valuable protein source (Vinayashree & Vasu, 2021).

Research has demonstrated that the proteins in pumpkin seeds exhibit various physiological functions, such as inhibiting bacterial growth, and reducing inflammation and blood sugar levels (Rojas et al., 2019; Wang et al., 2021). Consequently, it can be inferred that pumpkin seed protein may be used as a functional food ingredient.

The utilization of vegetable protein extracted from any of its parts requires an in-depth study to observe its functional properties and the composition of essential and non-

essential amino acids (Kumar et al., 2022). Kotecka-Majchzak et al. (2020) state that pumpkin seed proteins consist of globulin and albumin fractions.

The globulin fraction exhibits satisfactory coagulation activity, which is necessary for cheese maturation, and it may serve as a substitute for commercial animal rennet (Kotecka-Majchrzak et al., 2020). Furthermore, pumpkin seed protein isolates possess functional properties comparable to legume seed proteins, such as emulsification, foam formation, and gelation abilities (Yang et al., 2019).

Du et al. (2022) studied the effects of ultrasound on isolated pumpkin seed protein and found that the process increased solubility, foam-forming capacity, and hydrophobicity. Additionally, they observed a reduction in particle size due to the ultrasonic treatment.

### 6.3.3 Encapsulated Pumpkin Seed Oil

Microencapsulation is a widely used method for improving liquid samples' thermal stability, oxidative stability, and shelf life by converting them into more stable powders. This process preserves bioactive compounds and volatile oils. Numerous techniques have been developed for oil microencapsulation, including coacervation, emulsification, polymerization, spray drying, freeze drying, and melt extrusion. Among these approaches, spray drying is often used for oil microencapsulation (Karrar et al., 2020).

Oil microencapsulation processes are sequential, involving the formation of an emulsion that is later dehydrated to form microparticles. There is a correlation between emulsion stability and encapsulation efficiency. Therefore, controlling emulsification process parameters is crucial (Benito-Román, Sanz, & Beltrán, 2020).

Enhancing microencapsulation efficiency is a critical objective in microencapsulation technology. This factor reflects the presence of the oily phase on the surface of finished powder particles, indicating a particular wall material's ability to prevent leakage of the inner oily phase due to leaching (Shamaei et al., 2017).

In general, oils are prone to oxidative reactions that lead to deterioration and subsequently reduce quality. Oil encapsulation, typically performed through spray drying, is commonly used to protect sensitive compounds of interest within the food matrix (Luna-Guevara et al., 2017).

As proposed in this project, the encapsulation of pumpkin seed oil (in powder form) and the application of the oil in its liquid form present opportunities for innovative, versatile,

and practical utilization of this oil in various food product formulations. It also represents a potential option for enriching food products according to their intrinsic composition.

### 6.3.4 Application of Pumpkin Seed Oil in Active Packaging

Interest in developing biofilms or coatings (biodegradable films) is growing, mainly driven by the demand for high-quality foods and environmental concerns. The creation of biofilms and coatings with the potential for use as fruit coatings aims to extend the shelf life of highly perishable products. These coatings incorporate natural raw materials on the fruit surface, creating a modified internal atmosphere that reduces degradation reactions, minimizes moisture loss, texture and color changes, and acts as a barrier against spoilage agents (Peralta-Ruiz et al., 2020).

In addition to the mentioned benefits and interests, there has been a growing trend in recent years toward developing and using materials that minimize of synthetic materials. Edible coatings and films derived from natural polymers offer an excellent alternative that aligns with environmental concerns since these natural biopolymers are made from renewable and biodegradable raw materials (Mohamed et al., 2020).

The use of coatings in food, besides preserving the fruit, provides new alternatives for the use of film-forming raw materials derived from agricultural products and by-products of the processing industry, adding value and creating new markets for these raw materials (Jafarzadeh et al., 2021).

Edible films or coatings produced from biopolymers are used as alternative packaging materials. An edible coating is defined as a thin layer of edible material applied to the surface of a food product to create a semi-permeable barrier to gases and volatile compounds. These coatings are formulated to extend the shelf life of fresh products, reduce the respiration rate, delay senescence, and maintain texture and color (Tkaczewska, 2020).

The primary compositions of edible films include polysaccharides, proteins, and lipids. Starch, being a natural high molecular weight polysaccharide, offers advantages such as low cost, low degradability, good biocompatibility, and easy conversion into thermoplastic materials. It can also be used as a carrier for bioactive substances to enhance packaging functionality and holds significant potential for the development of food packaging materials (Menzel, 2020).

However, native starch is insoluble in water at room temperature and tends to retrograde. Additionally, starch-based films are brittle and exhibit low water resistance and

barrier properties, limiting their use as food packaging materials (Wongphan & Harnkarnsujarit, 2020).

Amin et al. (2020) identified antioxidants, antibacterial (against 8 strains of *E. coli* and *Shigella sonnei*), and anti-inflammatory properties in pumpkin seed oil extracts. Therefore, pumpkin seed oil may be a promising source of ingredients for the pharmaceutical, food, and active packaging industries, exploring its antimicrobial and antioxidant capabilities.

Szafranska et al. (2022) developed processed cheese sauces using pumpkin and kale dietary fibers and found that these fibers improved the stability of the samples and provided an excellent alternative for replacing typical hydrocolloids used in sauce production. Besides enhancing technological characteristics, they also exhibited health-promoting properties.

Therefore, in-depth studies on the comprehensive utilization of food and developing new products are essential to promoting sustainable development.

## CONCLUSIONS

In Brazil, the economic and health crisis has led to extreme poverty, affecting access to food. However, it has also resulted in increased consumption of fruits and vegetables. Despite food preservation techniques, food losses and waste continue to exacerbate global hunger, with billions of tons of food wasted annually. Pumpkins can significantly contribute to sustainable development goals, particularly zero hunger (SDG 2) and reducing food waste (SDG 12). Achieving these goals requires quality education (SDG 4), interdisciplinary partnerships (SDG 17), and shared infrastructure and innovation (SDG 9). Pumpkin pulp is rich in micronutrients like polyphenols and carotenoids and can be used in various products such as bread, yogurt, sauces, and chips. Pumpkin peels are nutritionally valuable and have technological applications, including carotenoid extraction for natural pigments, pectin-rich biodegradable films, and use as thickening agents in food products. Pumpkin seeds are nutrient-rich but underutilized in human consumption. They have antifungal and antimicrobial properties, and their oil has nutritional and nutraceutical value, useful in the pharmaceutical and agri-food industries. Pumpkin seed oil can enhance food nutritional value and stability and be used in active packaging to reduce waste. Defatted flour from pumpkin seed oil extraction is protein-rich and can serve as a functional food ingredient with beneficial physiological functions, such as inhibiting bacterial growth and reducing inflammation.

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# APPENDIX B - PAPER 2- (SUBMITTED TO INTERNATIONAL JOURNAL OF FOOD SCIENCE & TECHNOLOGY)

# OPTIMIZATION OF THE PUMPKIN SEED DRYING PROCESS: INFLUENCE OF ULTRASOUND AND DRYING TEMPERATURE ON KINETIC MONITORING AND MASS TRANSFER

**Abstract**

Pumpkin seeds are often discarded, but convection drying is a commonly used alternative to add value and preserve fruit and vegetable seeds. This unit operation increases the product's shelf life by reducing its moisture content. In this study, it has been evaluated the potential of and optimization of pre-treatment with ultrasound and convective drying of pumpkin seeds. An experimental design $2^3$ with three central points was performed to evaluate the effect of drying temperature (50, 60, and 70 °C), ultrasound amplitude (30, 50, and 70%) and ultrasound time (5, 10 and 15 min) on drying time and mass transfer parameters. Furthermore, empirical and diffusive models were fitted to the experimental data to describe the drying kinetics, and the mass transfer process. Optimized drying conditions were obtained for seeds pre-treated by ultrasound for 15 min with an amplitude of 70% and submitted to convective drying at 70 °C, which provided the shortest drying time (210 min) and the highest value of effective diffusivity of mass ($7.22 \times 10^{-10}$ $m^2 s^{-1}$) and convective mass transfer coefficient ($12.39 \times 10^{-8}$ $m\ s^{-1}$). Page's empirical model and the diffusion models were satisfactory fit for all conditions studied, presenting $R^2 > 0.99$ and low mean square deviation values. Thus, the findings of this study will contribute to the optimization of pumpkin seed drying and the pre-treatment with ultrasound, in addition to minimizing the impacts of the drying process, can certainly be suggested as an industrial application.

**Keywords:** Mathematical modeling, Response surface methodology, Waste recovery, Drying pre-treatments.

## 1. Introduction

The annual world production of pumpkin exceeds 22.9 million tons, which is used to produce purees, juices, jellies, and alcoholic beverages (Pacheco et al., 2023). During its processing, tons of by-products, such as seeds and bark, are generated (Ortiz—Jerez et al., 2022). Pumpkin seeds discarded as domestic and industrial by-products have a variety of nutritional properties, mainly due to the presence of bioactive compounds such as proteins, oils, sterols, polyamines, and antioxidants (Charaya et al., 2023).

Additionally, the seeds are grown and consumed frequently for their health-promoting properties, including anti-cancer, anti-inflammatory, anti-aging, immunity boosting, and detoxifying (Singh et al., 2020). For Singh & Kumar (2022) the use of industrial by-product aims to maximize the available resources, culminating in the production of new foods with high energy and commercial value, also representing an alternative for reducing an environmental problem, arousing the interest of the industry in the use of these residues (Aranha et al., 2017).

Fresh pumpkin seeds are highly susceptible to deterioration due to their high-water content and water activity, which lead to changes in appearance, texture and loss of bioactive compounds, impairing their storage, transport and sale (Chao & Fan, 2022), requiring processes or operations to extend its useful life. According to Shammi et al. (2022), among the post-harvest operations, convection drying has been the most used to preserve the original characteristics, being considered a hygienic and low-cost technique (Santana et al., 2022). However, these conventional drying methods can reduce the nutritional quality of the final product; in addition, they require a large amount of energy, consequently generating a high economic issue (Carvalho et al., 2021).

The long periods of the dehydration process, the high-energy consumption and the low quality of the final product obtained by conventional drying with hot air have contributed to the popularity of new non-thermal and ecological treatments (Santos et al., 2023). Additional processes (Hybrid Drying) might contribute to convective drying; in particular, ultrasound pretreatment can be used to reduce problems associated with convection drying (Bagheri & Dinani, 2019).

Ultrasound pretreatment has become one of the most common mechanical pretreatments, and has shown encouraging results in increasing the drying properties of plant products (Santos et al., 2022). According to Pandiselvam et al. (2023), when ultrasonic waves are applied, the material undergoes a sequence of rapid compressions and expansions, like a sponge being repeatedly squeezed and released, which aids in the transfer of water within the

substance. Microchannels are formed through the sponge effect, which helps transfer intracellular water to the surface, improving heat and mass transmission during drying (Santos et al., 2022).

The use of ultrasound as an alternative to mitigate nutritional losses in heat-treated foods has gained popularity due to increased consumer interest in nutritious and ecologically sustainable food products (Santos et al., 2023). In this context, emerging technologies have stood out in the most diverse industrial sectors to develop functional foods (Araújo et al., 2020). Thus, this study aimed to develop an optimized drying process for pumpkin seeds by evaluating the influence of ultrasound and temperature on mass transfer and drying kinetics along the drying process and the efficiency of these treatments.

## 2. Materials and methods

### 2.1 Plant material and sample preparation

Pumpkin seeds (*Cucurbita maxima*) were supplied by a minimally processed vegetable products company located in the city of Fortaleza, Ceará, Brazil. The seeds were taken to the Tropical Fruit Laboratory (LAFRUTH**)** at the Federal University of Ceará. The seeds were washed with distilled water, sanitized in a sodium hypochlorite solution (200 ppm) for 15 minutes, and pulp and peel adhered were manually removed. Then, the samples were stored at -20 °C.

### 2.2 Experimental planning

An experimental design $2^3$ with 3 repetitions at the central point was performed to analyze the effect of ultrasound pretreatment conditions (time: 5, 10 and 15 min; and amplitude: 30, 50 and 70%) and drying temperature (50, 60 and 70 °C) on drying time and the mass transfer parameters of pumpkin seeds. The experimental design resulted in a matrix with 11 experiments, as shown in Table 1.

**Table 1-** Planning matrix for the ultrasound process and convective drying of pumpkin seed with its independent variables (coded and real values).

| Experiments | Independent variables | | |
|---|---|---|---|
| | Ultrasound time (min) | Amplitude (%) | Drying temperature (°C) |
| E1 | -1 (5) | -1 (30) | -1 (50) |
| E2 | 1 (15) | -1 (30) | -1 (50) |
| E3 | -1 (5) | 1 (70) | -1 (50) |
| E4 | 1 (15) | 1 (70) | -1 (50) |
| E5 | -1 (5) | -1 (30) | 1 (70) |
| E6 | 1 (15) | -1 (30) | 1 (70) |
| E7 | -1 (5) | 1 (70) | 1 (70) |
| E8 | 1 (15) | 1 (70) | 1 (70) |
| E9 (C) | 0 (10) | 0 (50) | 0 (60) |
| E10 (C) | 0 (10) | 0 (50) | 0 (60) |
| E11 (C) | 0 (10) | 0 (50) | 0 (60) |

Note: C=Center point

Statistica® version 7.0 (StatSoft Inc., Tulsa, OK, USA) performed the statistical analysis. The results were subjected to analysis of variance (ANOVA) and Pareto chart analyses, and non-significant ($P > 0.05$) components were removed from the model.

### 2.3 Application of pre-treatment with ultrasound

The ultrasound process was performed using a ultrasonic processor (UP400S, Hielscher Ultrasonics, Teltow, Germany), with an H22 titanium sonotrode centrally immersed in a beaker with distilled water and pumpkin seeds, in a proportion of 1 g of sample to 10 mL of water, working with a fixed cycle (1.0) with amplitude and time varying according to the experimental design described in Table 1. After the ultrasound, the excess of water was removed on the material using a paper towel.

### 2.4 Drying experiments

The drying process was performed in triplicate for samples without ultrasound pre-treatment (control), and then it was submitted to the drying process at temperatures of 50, 60, and 70°C. The other drying experiments were performed after ultrasound pretreatment, varying the drying temperature, amplitude, and ultrasound time, according to the factorial design described in Table 1. The experiments were carried out using a convective dryer (TECHNAL, model TE-394/I) with an air velocity of 1.5 m $s^{-1}$. The drying process was carried out until it reached the mass transfer equilibrium. The data was recorded for all cases, and the moisture ratio of the drying process was calculated by Method No 1934 (AOAC, 2016).

**Table 1-** Planning matrix for the ultrasound process and convective drying of pumpkin seed with its independent variables (coded and real values).

| Experiments | Independent variables | | |
|---|---|---|---|
| | Ultrasound time (min) | Amplitude (%) | Drying temperature (°C) |
| E1 | -1 (5) | -1 (30) | -1 (50) |
| E2 | 1 (15) | -1 (30) | -1 (50) |
| E3 | -1 (5) | 1 (70) | -1 (50) |
| E4 | 1 (15) | 1 (70) | -1 (50) |
| E5 | -1 (5) | -1 (30) | 1 (70) |
| E6 | 1 (15) | -1 (30) | 1 (70) |
| E7 | -1 (5) | 1 (70) | 1 (70) |
| E8 | 1 (15) | 1 (70) | 1 (70) |
| E9 (C) | 0 (10) | 0 (50) | 0 (60) |
| E10 (C) | 0 (10) | 0 (50) | 0 (60) |
| E11 (C) | 0 (10) | 0 (50) | 0 (60) |

Note: C=Center point

## 2.5 Mathematical modeling of drying

### 2.5.1 Empirical models

Several authors have used empirical equations to model the drying process. Among the most suggested models, we have Page (Equation 1) (Page, 1949), Midili (Equation 2) (Midili et al., 2002) and Henderson & Pabis (Equation 3) (Henderson & Pabis, 1961)

$$RX = \exp(-k \times t^n) \quad (1)$$

$$RX = a \times \exp(-k \times t^n) + b \times t \quad (2)$$

$$RX = a \times \exp(-k \times t) \quad (3)$$

Where: RX is the moisture content ratio; K, a, b and n are dimensionless coefficients; t is time (min).

The model's fittings to the experimental data were analyzed by Statistica® version 7.0 (StatSoft Inc., Tulsa, OK, USA), using the non-linear regression analysis, by the Quasi-Newton method. The models were selected by taking as a parameter the magnitude of the coefficient of determination ($R^2$) and the mean square deviation (RMSD)

### 2.5.2 Drying rate

Equation 4 is obtained as a derivative of Page's model, and it represents the drying rates for pumpkin seeds during the processing time. This equation allows, through two curves, the evaluation of drying temperatures in control experiments, dehydrated at temperatures of 50, 60, and 70°C, and analyze the effect of ultrasound variables on the drying rates of experiments E1, E2 and E3, both submitted to drying temperature at 50 °C. The graphs of drying rates were obtained using the Labfit software (available as freeware from: http://www.labfit.net).

$$\frac{dRX}{dt} = -K \times n \times t^{n-1} \times e^{-K \times t^n} \quad (4)$$

Where: $\frac{dRX}{dt}$ is derived from the moisture content ratio; K, and n are dimensionless coefficients; t is time (min).

### 2.5.3 Diffusion model: slab infinite geometry

Pumpkin seeds have a much larger diameter compared to their thickness, as seen in Figure 1B, so its geometry might be considered an infinite wall (Figure 1A), as studied by Silva

(2009) and Patankar (1980), through the finite volume method and the balance in the volume control (described in Figure 1C).

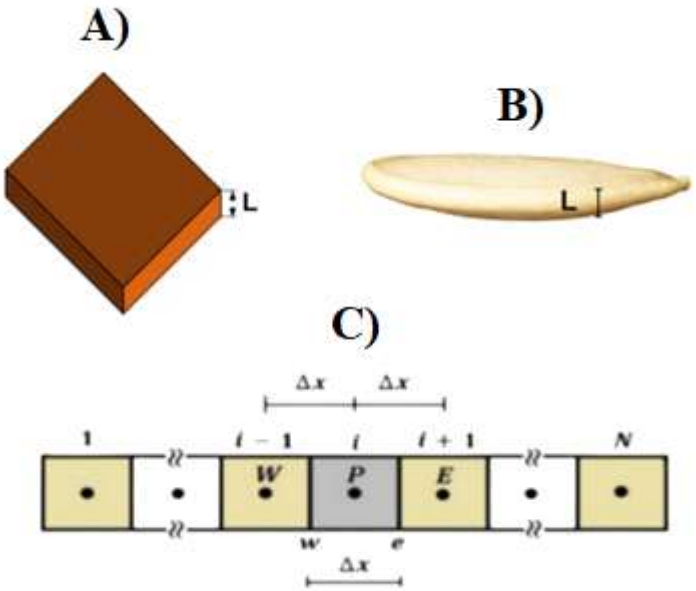


**Figure 1-** Infinite wall geometry (A), side view of the pumpkin seed (B) and balance in control volumes (C).

Source: Adapted from Silva (2009)

For the third-type boundary conditions, the analytical solution of the diffusion equation is given by the series presented in Equations 7, 8, and 9 (Luikov et al., 1968), in which only the first 16 terms of the infinite series were used. Consequently, the third-type boundary condition was applied. According to Silva et al. (2015), this condition allows a better fit to the experimental data.

$$X^*(t) = \sum_{n=1}^{16} B_n \exp(-\mu_n^2 \frac{Def}{(L/2)^2} t) \quad (5)$$

$$B_n = \frac{2Bi^2}{\mu_n^2 (Bi^2 + Bi + \mu_n^2)} \quad (6)$$

$$Bi = \frac{h(L/2)}{Def} \quad (7)$$

Where, L is the thickness of the infinite slab; $D_{ef}$ is the effective mass diffusivity; h is the convective mass transfer coefficient; t is the drying time; Bi is the Biot number; $B_n$ coefficient; $\mu_n$ are the roots of the transcendental.

Equation 5 was fitted to the experimental data using the optimization proposed by Silva et al. (2010).

### 2.5.4 Numerical solution for diffusion model: distribution of water content

The moisture distribution at a given moment might be determined using the parameters estimated for the diffusive model according to Equation 8 (Santos *et al.*, 2020).

$$X^*(x,t) = \sum_{n=1}^{16} A_n \cos\left(\frac{\mu_n}{L/2}x\right) \exp\left[-\frac{\mu_n^2}{(L/2)^2} D_{ef} t\right] \quad (8)$$

where, $A_n$ is given by:

$$A_n = \frac{4\sin\mu_n}{2\mu_n + \sin(2\mu_n)} \quad (9)$$

## 3. Results and discussion

### 3.1 Kinetic follow-up and adjustment of empirical models

Aiming to better fit the empirical model to the evolution of the moisture content of pumpkin seeds during drying, some correlation parameters were estimated to evaluate its performance: the R2 and RMSD. Table 2 shows the estimated values of the parameters for Page, Henderson & Pabis, and Midilli models for drying pumpkin seeds (control and pre-treated with ultrasound), as well as the R2 and the RMSD.

**Table 2 -** Adjustment parameters of the Page, Henderson & Pabis and Midilli models to the pumpkin seeds drying experimental data (control and pre-treated experiments).

| **Experiments** | | **Parameters** | | | | | |
|---|---|---|---|---|---|---|---|
| | | **a** | **k** | **n** | **b** | **$R^2$** | **RMSD** |
| | | | **Page** | | | | |
| Control samples | 50 °C | - | 0.013 | 1.010 | - | 0.999 | 0.027 |
| | 60 °C | - | 0.020 | 1.021 | - | 0.998 | 0.002 |
| | 70 °C | - | 0.029 | 0.971 | - | 0.998 | 0.010 |
| With | E1 | - | 0.024 | 1.014 | - | 0.999 | 0.004 |
| | E2 | - | 0.026 | 1.086 | - | 0.999 | 0.007 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| ultrasound | E3 | - | 0.029 | 1.107 | - | 0.998 | 0.001 |
| | E4 | - | 0.030 | 1.241 | - | 0.999 | 0.024 |
| | E5 | - | 0.035 | 1.300 | - | 0.999 | 0.005 |
| | E6 | - | 0.031 | 0.990 | - | 0.997 | 0.010 |
| | E7 | - | 0.035 | 1.172 | - | 0.997 | 0.011 |
| | E8 | - | 0.036 | 1.323 | - | 0.998 | 0.014 |
| | E9 | - | 0.034 | 1.024 | - | 0.999 | 0.004 |
| | E10 | - | 0.034 | 1.018 | - | 0.997 | 0.014 |
| | E11 | - | 0.035 | 1.168 | - | 0.998 | 0.014 |
| | | | **Henderson & Pabis** | | | | |
| Control samples | 50 °C | 1.005 | 0.013 | - | - | 0.989 | 0.024 |
| | 60 °C | 0.993 | 0.021 | - | - | 0.998 | 0.001 |
| | 70 °C | 0.994 | 0.026 | - | - | 0.998 | 0.017 |
| With ultrasound | E1 | 1.013 | 0.023 | - | - | 0.998 | 0.011 |
| | E2 | 1.017 | 0.024 | - | - | 0.998 | 0.017 |
| | E3 | 1.030 | 0.027 | - | - | 0.997 | 0.007 |
| | E4 | 1.055 | 0.027 | - | - | 0.994 | 0.026 |
| | E5 | 1.053 | 0.033 | - | - | 0.991 | 0.015 |
| | E6 | 0.978 | 0.028 | - | - | 0.994 | 0.026 |
| | E7 | 1.030 | 0.034 | - | - | 0.993 | 0.024 |
| | E8 | 1.072 | 0.034 | - | - | 0.991 | 0.021 |
| | E9 | 1.000 | 0.030 | - | - | 0.999 | 0.005 |
| | E10 | 1.029 | 0.030 | - | - | 0.993 | 0.024 |
| | E11 | 1.029 | 0.032 | - | - | 0.995 | 0.026 |
| | | | **Midilli** | | | | |
| Control samples | 50 °C | 1.002 | 0.012 | 1.0198 | $1\times10^{-4}$ | 0.999 | 0.005 |
| | 60 °C | 0.975 | 0.016 | 1.072 | 0 | 0.998 | 0.001 |
| | 70 °C | 1.003 | 0.030 | 0.971 | 0 | 0.998 | 0.002 |
| With ultrasound | E1 | 0.983 | 0.020 | 1.113 | $1\times10^{-4}$ | 0.999 | 0.002 |
| | E2 | 0.987 | 0.025 | 1.111 | $1\times10^{-4}$ | 0.999 | 0.001 |
| | E3 | 1.004 | 0.030 | 1.105 | $1\times10^{-4}$ | 0.989 | 0.011 |
| | E4 | 0.995 | 0.031 | 1.258 | $1\times10^{-4}$ | 0.999 | 0.001 |
| | E5 | 0.983 | 0.033 | 1.352 | 0 | 0.998 | 0.003 |
| | E6 | 1.014 | 0.031 | 0.977 | $1\times10^{-4}$ | 0.997 | 0.002 |
| | E7 | 0.978 | 0.034 | 1.223 | $1\times10^{-4}$ | 0.997 | 0.030 |
| | E8 | 1.005 | 0.035 | 1.328 | $1\times10^{-4}$ | 0.999 | 0.002 |
| | E9 | 0.988 | 0.031 | 1.0452 | $1\times10^{-4}$ | 0.999 | 0.001 |
| | E10 | 0.968 | 0.032 | 1.267 | $1\times10^{-4}$ | 0.998 | 0.001 |
| | E11 | 0.971 | 0.032 | 1.242 | $1\times10^{-4}$ | 0.998 | 0.002 |

Note: $R^2$ is the coefficient of determination; RMSD is the root-mean square deviation; “a”, “k”, “n” and “b” are the parameters of mathematical models.

The models used represented the drying process well with $R^2$ being higher than 0.9 and the RMSD lower than 0.0271. The Page and Midilli models presented $R^2$ with values greater than 0.99 in all experiments, in addition to presenting low values in the RMSD.

According to Alves et al. (2019), the higher the $R^2$, the greater the reliability of the mathematical model in describing the drying process. It can still be stated that $R^2$ values greater than 0.95 indicate that the model can accurately predict the product's behavior, regarding reducing water content during the drying process. However, this parameter must be evaluated in association with a statistical parameter, such as the RMSD. Given the simplicity of the Page model and its satisfactory fit to the experimental data, it is suggested that this is the most suitable model for describing the drying process of pumpkin seeds. Xu et al. (2020), performed the adjustment of the Page, Midilli, Logarítimo, Henderson & Pabis and Newton models to the experimental data of the drying of cider slices, found that the Page and Midilli models presented better adjustments when compared to the other analyzed models.

Table 2 also provides the results for the parameters of the tested models. Notably, the drying rate constants (k) displayed a consistent upward trend as the drying air temperature increased, a pattern observed across all the mathematical models applied. In the context of our study, the "k" value represents the drying rate constant, which signifies how quickly moisture is removed during the drying process. A higher "k" value suggests a faster drying rate, which could be beneficial efficiency (Santos et al., 2020). As for the impact of ultrasound pre-treatment on the "k" value, our observations indicate that ultrasound has a positive effect. Specifically, we noticed that the "k" value increased in experiments with longer ultrasound exposure and greater amplitude, as exemplified in Experiment 8. This suggests that ultrasound pre-treatment enhances the drying process by potentially breaking down barriers to moisture removal, resulting in a more efficient drying rate.

The Page model's coefficient "n" varied slightly between 0.97 and 1.24. However, there was no correlation between the values of this coefficient and the independent variables (drying air temperatures, ultrasound time, and ultrasound amplitude). Santos et al. (2020) state that the "n" coefficient signifies the gradient between the air's vapor pressures and the material's vapor pressure. It represents the internal resistance of the product to drying. In theory, increasing the drying air temperature should increase this coefficient, ultimately leading to a higher water removal rate from the product. Despite this, the authors did not verify the influence of temperature on the values obtained for the "n" parameter during the convective drying process of red rice. This behavior can be explained because factors such as drying air speed, initial water content, final water content, relative air humidity, temperature, pressure, and product composition directly influence the drying kinetics of the products (Moreira et al., 2018a).

The “a” parameter, represented only in the Henderson & Pabis and Midilii models, does not present a direct correlation between the values obtained for the parameters and the variables (drying temperature, ultrasound time, and amplitude). According to Leite et al. (2022), “a” and “b” parameters are constants of proportionality between the drying rates and the humidity rate and do not always present a correlation with the increase in temperature. SANTOS et al. (2018) also did not observe a clear trend in the behavior of the “a”, “b”, and “n” coefficients of the models adjusted to the experimental data of passion fruit seed drying, so these coefficients are commonly treated as empirical variables. The “b” parameter exhibited varying magnitudes, ranging from 0 to 0.001. In the control experiments, only the sample subjected to a temperature of 50°C displayed a value of 0.001, suggesting a decrease in “b” with increasing temperature. However, only experiment 5 presented a value equal to 0, while the others presented a value of 0.001, with no correlation between “b” parameter and the independent variables.

Figure 2 presents the drying kinetics curves obtained by fitting the Page model to the experimental data of pumpkin seeds drying.

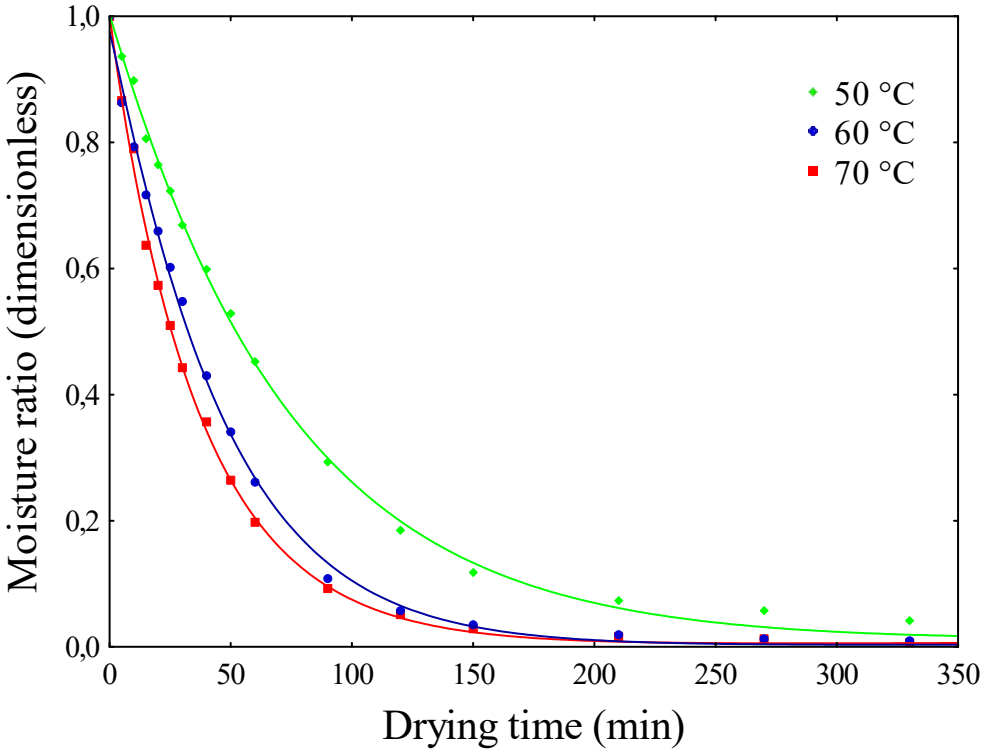


**Figure 2 A**- Drying curves of pumpkin seeds obtained by the Page model to control experiments 50, 60 and 70 °C temperatures.

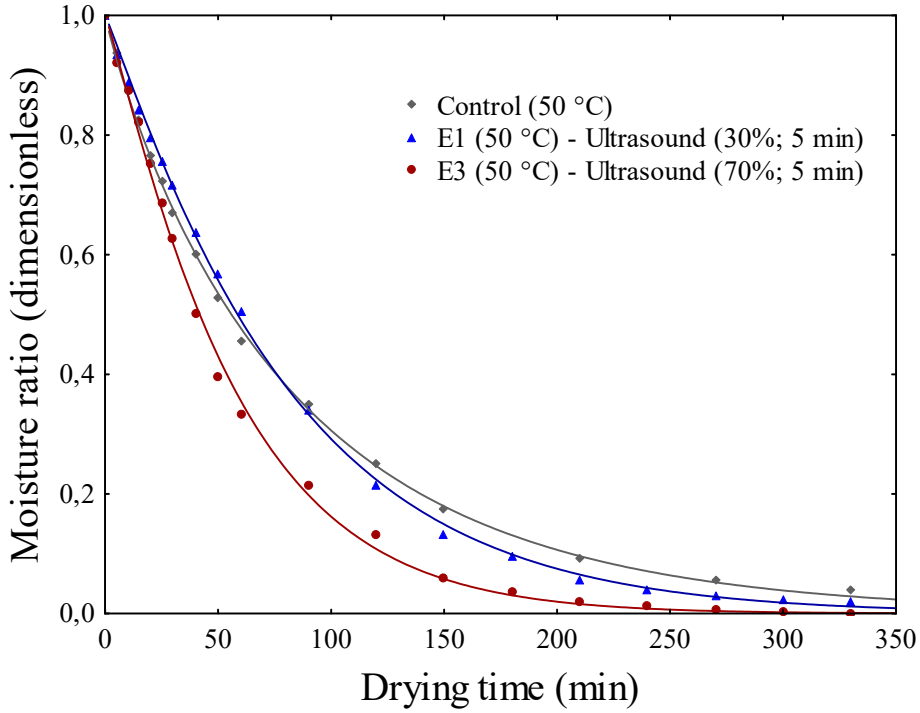


**Figure 2 B** - Drying curves of pumpkin seeds obtained by the Page model to control experiment, E1 and E3, at temperature 50 °C.

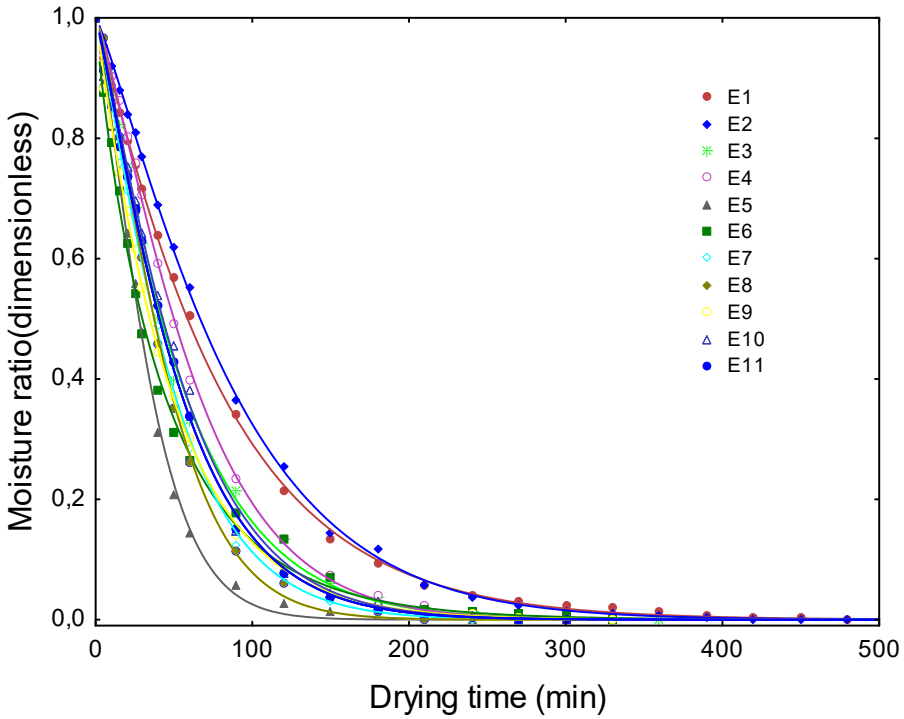


**Figure 2 C**- Drying curves of pumpkin seeds obtained by the Page model to experiments, E1, E2, E3, E4, E5, E6, E7, E8, E9, E10, and E11.

Figure 2A presents the control experiments (without the previous ultrasound treatment) with different drying air temperatures (50, 60, and 70 °C) so that the influence of the increase in the drying air temperature on the drying air graphs can be verified. Figure 2B shows the control experiment (without previous ultrasound treatment).

Experiments E1 and E3, both using a drying temperature of 50 °C, were selected to promote the analysis of the influence of ultrasound amplitude on the drying kinetics of the samples. Figure 2C represents the drying kinetic curves of ultrasound treatments (determined by experimental planning).

When analyzing the drying curves of pumpkin seeds in the control experiments at different temperatures, we observed an interaction between the drying air temperature and drying time, which ranged from 450 to 600 minutes when the drying temperature varied from 50 to 70°C. Remarkably, the shortest drying time (i.e., 450 minutes) was achieved when the air temperature reached 70 °C. At the beginning of the process, the water loss is higher; this behavior is evidenced by the curve that presents a linear region at the beginning of the process, followed by polynomial behavior. Costa et al. (2021) also analyzed this trend in drying the seeds of Amaranthus cruentus “BRS Alegria”.

For the drying of melon seeds, Leite et al. (2022) found that increases in drying temperatures resulted in an enhancement in the rate of water evaporation from the seeds, but the curves presented were similar. The authors attributed these higher drying rates to the fact that a greater water pressure gradient is generated at higher temperatures, allowing faster evaporation of the liquid phase in a short time interval. In addition to the influence of temperature on the water pressure gradient, there are considerable changes in water viscosity. According to Almeida et al. (2021), the increase in temperature causes a decrease in the viscosity of the water, consequently reducing the resistance to flow and the diffusion of water in the product, which starts to flow more easily.

Figure 2B, represents the drying kinetics curves of the control experiments, E1 and E3. It was verified that the kinetics curve of the control experiment is like the E1 experiment in the initial moments of the process, but there was a more expressive distinction from the moment t = 200 min, since the experiment submitted to the ultrasound process remained with a high rate of water loss and reached the equilibrium condition in a faster way. This behavior occurs because of the action of ultrasonic waves, which cause a sequence of rapid compressions and expansions in the material, thus helping the transfer of water within the substance. Because of

this mechanical tension, microchannels are formed in the material, which assists in transfering intracellular water to the surface (Pandiselvan et al., 2023).

The curves of the milder treatments (E1 and E2) differed significantly from the others. For these treatments to reach equilibrium, a drying time of more than 500 minutes was required. Furthermore, the similarity of the other experiments can be verified, especially in the initial moments when there is a sudden reduction in the moisture ratio. In this way, it can be inferred that the changes caused by the ultrasound treatment become more evident at higher amplitudes, which causes a more pronounced reduction in the mix ratio.

It can be observed that the ultrasound process promoted a significant reduction in the drying time of the samples since the control sample (without the ultrasound treatment) presented a longer drying time (690 min) when compared to the other experiments. It can still be inferred that the experiment submitted to the ultrasound process with higher amplitude (70%) presented a shorter drying time (360 min), when compared to the other experiments, resulting in a reduction of approximately 50% of the time required in the drying process.

Corrêa et al. (2017) conducted studies on pineapple drying, pre-treated by ultrasound and osmotic dehydration, and observed that the ultrasound process promoted a considerable reduction in drying time (35%). These authors found that more porous materials tend to show a greater reduction in drying time by the application of ultrasound, such as apple (Rodriguez et al., 2017) and orange peel (Garcia-Perez et al., 2011), which showed reductions of 54% and 45%, respectively.

### 3.2 Evaluation of the drying rate

In Figure 3, we present the graphical representation of the drying rates of pumpkin seeds as determined by the Page model. Figure 3A shows the curves representing the drying rates at all temperatures applied (50, 60, and 70°C) in the samples that were not subjected to the ultrasound process, in order to verify the influence of air temperature on the drying rates.

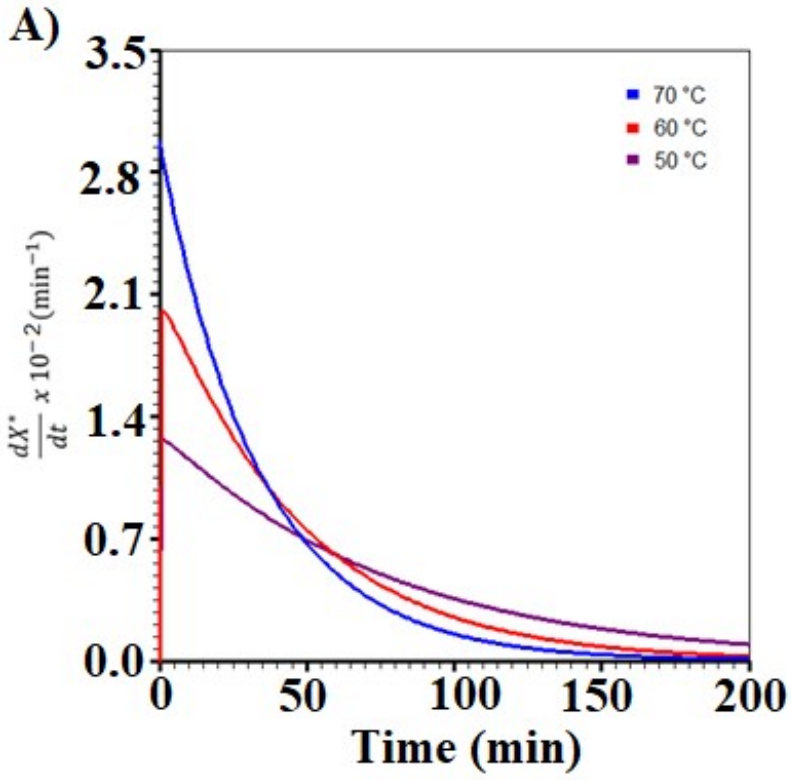


**Figure 3 A**- Drying rates of pumpkin seeds obtained by the Page model to control samples (50, 60, and 70°C).

Figure 3B shows the curves representing the graph obtained for the drying rates of pumpkin seeds from experiments E1, E2 and E3, dehydrated in convective drying at a temperature of 50 °C.

Experiments E1, E2, and E3 were selected to evidence the influence of ultrasound amplitude and time on the drying rates of the samples. Our hypotheses are that maintaining a constant drying temperature ensures that any differences observed in the drying kinetics are primarily attributed to the ultrasound treatment and amplitude, rather than temperature variations.

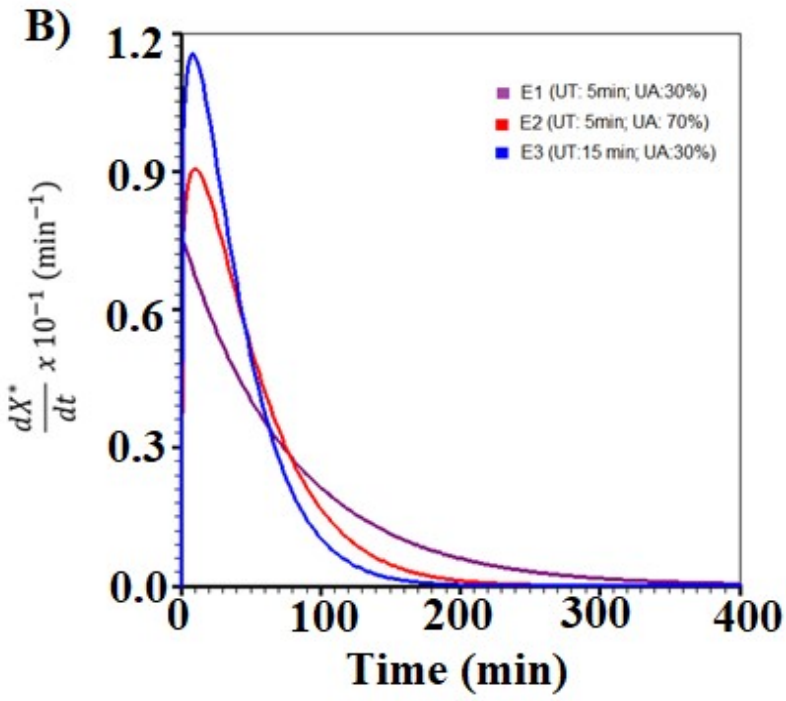


**Figure 3 B**- Graph of the drying rates of pumpkin seeds obtained by the Page model to samples submitted to the ultrasound process (E1, E2, and E3).

Evaluating the curves in Figure 3A, it can be observed that the seeds present increasing drying rates in the initial moments of the process (induction period). At the end of the induction period, the highest drying rate was obtained in the process, which ranged from $1.3x10^{-2}$ $min^{-1}$ (temperature of 50 °C) to $3.0x10^{-2}$ $min^{-1}$ (temperature of 70 °C), the drying rate was found to be proportional to the gradient of the water content in the product.

After reaching the highest drying rate possible, a period of decreasing rate begins, which occurs due to the reduction in the gradient of water content in the seeds. So, throughout the process, there is a decrease in the water content and a decrease in the gradients presented, consequently, a decrease in the drying rate (-dX*/dt) from the initial moments to the end of the process, when the drying rate reaches a null value, indicating an equilibrium condition has been reached (Leite et al., 2022).

Similar behavior was observed by Dhurve et al. (2022) in watermelon seeds drying and by Keneni et al. (2019) in the drying kinetics of Jatropha curcas L. seeds. The authors defined the drying rate as being proportional to the product's water content. Thus, higher values of the rates are observed in the initial moments of the process and decrease as the water content decreases. This fact can be explained because, in the initial moments of the process, there is a

steeper gradient between the water content of the material and the humidity of the air, thus providing higher rates of mass transfer.

Analyzing the curves that represent the drying rates, it can be observed that the drying rates in the initial moments of the samples submitted to ultrasound are higher than those not pre-treated with ultrasound. In Figure 3B, the influence of the variables applied in the ultrasound on the drying rates is observed; experiments E1, E2, and E3 were submitted to the same drying temperature. Experiment E3 presented a higher drying rate than the other experiments. This fact can be explained by the fact that sample 3 was submitted to a greater amplitude (70%), while samples E2 and E3 were submitted to a lower amplitude (30%). When comparing the E2 and E3 curves, it appears that the E2 presented a higher rate when compared to the E1, indicating that the drying time slightly influenced the drying rate of the samples, as the E1 was subjected to the ultrasound process for 5 min, while E2 to the ultrasound process for 15 min.

According to Cao et al. (2019), the ultrasound-induced shear stress destroyed part of the cell walls, thus allowing more free water inside the material, resulting in a higher dehydration rate after heat transfer. In addition, the correlation between drying time and pretreatment duration reveals that damage to the internal structure depends on ultrasound exposure, which, combined with high drying temperatures, resulted in an effective drying time. No period of constant drying rate was observed in any of the experiments (Figures 3a and 3b). According to Araújo et al. (2020), this behavior can occur when the product has a high lipid content in its composition. When there are no constant rate periods in the drying process, it can be inferred that diffusion is the dominant physical mechanism that drives the mass transfer from the material to the drying air.

### 3.3 Analytical solution of the diffusion model

The values obtained for the Biot number (Bi) presented magnitudes that ranged from 1 to 8.25, contrary to what was observed about effective mass diffusivity and with convective mass transfer coefficient, no correlation was observed between the Biot number and the independent variables of the drying and ultrasound process. Santos et al. (2020) observed similar results during the convective drying kinetics of red rice. The authors suggested that when relatively low Biot numbers (10) are coupled with high R2 values and low chi-square values, this combination may indicate that the diffusion model, applied with a third-type boundary condition, adequately characterizes the drying process of pumpkin seeds.

According to Shewale et al. (2019), the Biot number is a function of a latency factor. It indicates internal and external resistance to water diffusion in the product (if, 0.1 <Bi < 100). According to Moreira et al. (2018b), when the Biot number is sufficiently small (<0.1), it can be assumed that the moisture distribution is uniform in the product, at any time of the drying process. Mathematical models are applied to predict the behavior of the various phenomena that occur during the drying process, which can promote the reduction of operational costs and better quality of the final (Mesías et al., 2021).

The diffusion model was applied considering the boundary condition of the third type (convective). According to Aires et al. (2018), this boundary condition presents a better fit to the experimental data, since it showed higher values of the $R^2$ ($R^2$ >0.99) and low chi-square values ($\chi^2$). Table 3 shows the parameters obtained in the simulation of the drying process of the pumpkin seeds of the control samples and those that were submitted to the ultrasound process.

**Table 3 -** Parameters obtained through simulations using the third type analytical solution and boundary condition.

| Experiments | | DT (min) | *Def* ($m^2/s^1$) | h (m/s) | Bi | $R^2$ | $\chi^2$ |
|---|---|---|---|---|---|---|---|
| Control samples | 50 °C | 690 | $1.87\times10^{-10}$ | $1.11\times10^{-8}$ | 1.75 | 0.992 | 0.025 |
| | 60 °C | 570 | $2.01\times10^{-10}$ | $2.99\times10^{-8}$ | 1.00 | 0.994 | 0.012 |
| | 70 °C | 450 | $3.20\times10^{-10}$ | $6.24\times10^{-8}$ | 1.75 | 0.996 | 0.012 |
| With ultrasound | E1 | 510 | $2.21\times10^{-10}$ | $3.69\times10^{-8}$ | 1.50 | 0.993 | 0.015 |
| | E2 | 480 | $2.62\times10^{-10}$ | $4.97\times10^{-8}$ | 1.75 | 0.997 | 0.025 |
| | E3 | 360 | $3.45\times10^{-10}$ | $8.33\times10^{-8}$ | 3.00 | 0.995 | 0.038 |
| | E4 | 330 | $3.90\times10^{-10}$ | $8.64\times10^{-8}$ | 2.25 | 0.998 | 0.012 |
| | E5 | 270 | $6.22\times10^{-10}$ | $12.22\times10^{-8}$ | 1.25 | 0.998 | 0.012 |
| | E6 | 330 | $3.85\times10^{-10}$ | $8.82\times10^{-8}$ | 8.25 | 0.998 | 0.014 |
| | E7 | 240 | $7.10\times10^{-10}$ | $12.35\times10^{-8}$ | 3.50 | 0.993 | 0.061 |
| | E8 | 210 | $7.22\times10^{-10}$ | $12.39\times10^{-8}$ | 6.50 | 0.998 | 0.019 |
| | E9 | 330 | $4.53\times10^{-10}$ | $11.46\times10^{-8}$ | 1.00 | 0.997 | 0.025 |
| | E10 | 300 | $4.23\times10^{-10}$ | $11.25\times10^{-8}$ | 1.75 | 0.994 | 0.031 |
| | E11 | 300 | $4.59\times10^{-10}$ | $11.34\times10^{-8}$ | 1.50 | 0.992 | 0.011 |

Note: DT is the drying time; *Def* is the effective mass diffusivity; h is the convective mass transfer coefficient; Bi is the Biot number (dimensionless); $R^2$ is the coefficient of determination and $\chi^2$ is the chi-square function.

The samples that were not subjected to the ultrasound process showed effective diffusivity (*Def*) values that ranged from 1.87 to $3.20\times10^{-10}$ $m^2$ $s^{-1}$, the highest value was obtained when the samples were subjected to a higher temperature (70 °C), indicating a direct correlation between drying air temperature and effective diffusivity. Similarly, Ferreira et al. (2021) observed the influence of effective diffusivity temperature when drying pumpkin seeds and obtained effective diffusivity values ranging from 1.75 to $3.70\times10^{-10}$ $m^2$ $s^{-1}$ when the process temperature ranged from 50 to 70 °C.

It is observed that the increase in time and amplitude of ultrasound contributed significantly to the enhancement in the seed's diffusivity, thus increasing the drying efficiency. The highest diffusivity value was observed in experiment 8 ($7.22\times10^{-10}$ $m^2$ $s^{-1}$) which was subjected to the highest level of ultrasound amplitude (70%), longer time (15 min), and higher drying temperature (70 °C). A lower effective diffusivity value was observed in the control experiment with a drying temperature of 50 °C and presented a magnitude corresponding to $1.87\times10^{-10}$ $m^2$ $s^{-1}$.

Dibagar et al. (2020) evaluated the influence of ultrasound power on sunflower seeds and observed similar behavior; the values obtained for the Def of sunflower seeds varied between 1.7 and 7.1×10-10 m2 s-1. Specific values of effective diffusivity are scarce in the literature, especially for foods, since this parameter is influenced by several factors, such as the intrinsic composition of the product and process variables through which the product is subjected. The most common food diffusivity range observed in the literature is from 10-11 to 10-9 m2 s-1 (Almeida et al., 2021; Ferreira et al., 2021).

During the ultrasound process, the internal structure of the material flexes and expands due to the vibration caused by the high frequencies of ultrasound. In this way, the kinetic energies and emotions generated cause the formation of many bubbles that burst instantly; this process is called cavitation. The main effect of cavitation is related to the increased mobility of water flags that increase the diffusivity of the material and accelerate the water diffusion process (Xi et al., 2019).

Another parameter obtained through the analytical solution of the diffusion model was the mass transfer coefficient (h) (Tabela 3). The lowest hm value was 1.11×10-8 m s- 1, obtained in the dry control sample with the lowest drying air temperature (50 °C), and the highest value of this parameter was observed in the E8 sample (12.39×10-8 m s-1), which was

subjected to a long time and amplitude of ultrasound. Thus, it was observed that the pre-treated samples presented higher values of hm than the control samples. Rajoriya et al. (2021), in their drying studies, observed that an increase in the temperature applied in the process promoted an increase in the convective mass transfer coefficient (h), which is associated with an increase in effective diffusivity (Def). The increase in the values of these parameters generates higher rates in the drying process (water movement from the inner layer to the outer layer of the product), as shown in Figure 3.

The correlation between the mass transfer coefficient (h) and the effective diffusivity (Def), reported by Rajoriya et al. (2021), was also observed in the present study, as the samples that presented higher values of Def simultaneously presented high values of hm, which was directly proportional to the pre-treatment time, ultrasound amplitude and drying temperature. The ultrasound process causes pressure variations in the molecules that make up the product interface. It also causes the creation of microscopic channels that change the cellular structure of the samples (Tao et al., 2016),

Consequently, the boundary layer is reduced, and the mass transfer coefficient is increased due to the reduction of external resistance to moisture transfer (Santos et al., 2020). In the present study, the values obtained were equal to or greater than 1, which may indicate that the water content distribution does not occur completely homogeneously in the sample during the drying process.

### 3.4 Optimization of the pumpkin seed drying process

According to Alim et al. (2023), optimization is important in food engineering, as it is pivotal in enhancing process efficiency and increasing throughput.

Analyzing the Pareto graphs (Figure 4), it is possible to verify the magnitude of the influence of the independent variables on the dependent variable. Figure 4A shows that only the drying air temperature and the ultrasound amplitude showed statistical significance at a confidence level of 95% and decreasing effects concerning the dependent variable, with the drying air temperature being the variable that most interfered with the drying time of the experiments.

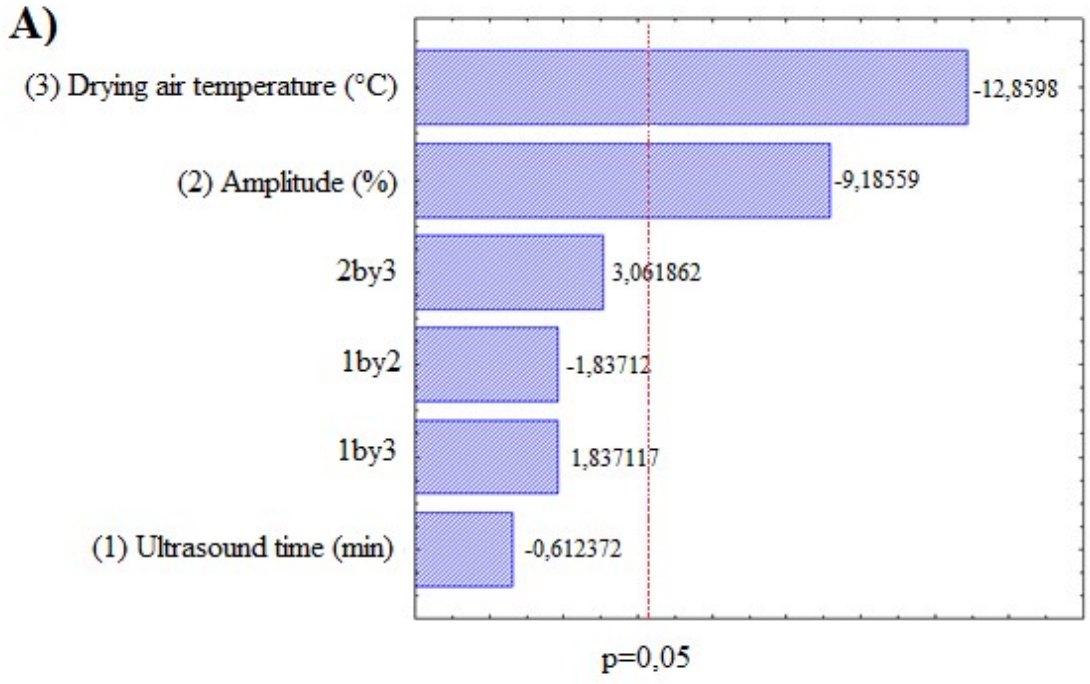


**Figure 4A -** Pareto graph for drying time.

The ultrasound time was not statistically significant in the drying time. In Figure 4B, only the variables drying temperature and ultrasound amplitude showed statistical significance and positive effects in relation to the effective diffusivity of the product.

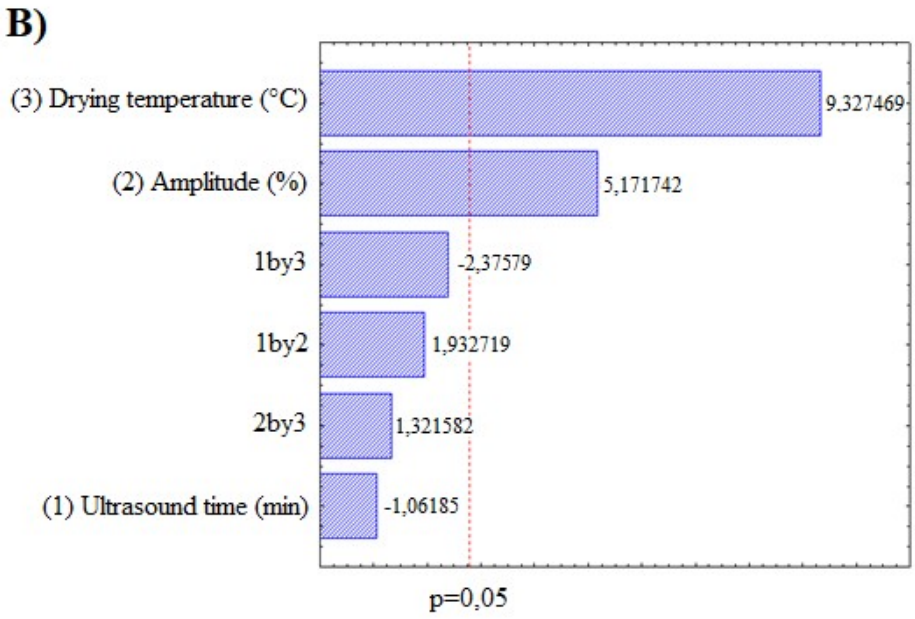


**Figure 4B -** Pareto graph for effective diffusivity.

The mass convective coefficient and Biot number did not show statistical significance at a 95% confidence level; therefore, the corresponding values obtained for the ANOVA Table are not expressed. Table 4 shows the parameters derived from the ANOVA test, considering drying time and effective diffusivity as dependent variables.

**Table 4 -** ANOVA table for drying time and effective diffusivity.

| **Factor** | **Source of variation** | **SQ** | **DF** | **QM** | **$F_{cal}$** | **$F_{tab}$** | **$F_{cal}/F_{tab}$** | **$R^2$ (%)** |
|---|---|---|---|---|---|---|---|---|
| Drying time | Regression | 79875 | 3 | 26625 | 28.45 | 6.59 | 4.31 | 99.02 |
| | Lack of fit | 3143.18 | 2 | 1571.59 | 5.24 | 19.25 | | |
| | Pure error | 600 | 2 | 300 | | | | |
| | Total SS | 83618.18 | 7 | | | | | |
| **Factor** | **Source of variation** | **SQ** | **DF** | **QM** | **$F_{cal}$** | **$F_{tab}$** | | **$R^2$ (%)** |
| *Def* | Regression | 26.98 | 6 | 8.99 | 42 | 6.16 | 6.81 | 92.30 |
| | Lack of fit | 0.78 | 2 | 0.39 | 10.51 | 19.25 | | |
| | Pure error | 0.07 | 2 | 0.03 | | | | |
| | Total SS | 27.84 | 10 | | | | | |

Note: SQ is the Sum of squares; DF is the degree of freedom; QM is Medium square; *Def* is the effective mass diffusivity; h is the convective mass transfer coefficient; $R^2$ is missing, Fcal is the F calculated by the analysis of variance and Ftab is the F tabulated by the analysis of variance.

The experimental data were fitted to a polynomial regression model for the drying time (Equation 12) and effective diffusivity (Equation 13) dependent variables. The polynomial model obtained for the drying time was statistically significant at a 95% confidence interval since the $F_{calculated}$ value (28.45) was higher than the $F_{tabulated}$ value (6.59). This result indicates that the polynomial model obtained for this parameter is statistically significant and satisfactorily represents the influence of the independent variables (amplitude, ultrasound time, and drying air temperature) on the dependent variable (drying time). On the other hand, the calculated F of the lack of fit was lower than the $F_{tabulated}$, indicating that the lack of fit of the model is not significant, thus indicating that the polynomial model presents a satisfactory fit to the experimental data. A good $R^2$ was also obtained ($R^2 = 0.99$).

The model obtained for the effective diffusivity also presented $F_{calculated}$ (42) higher than the $F_{tabled}$ (6.16), which indicates the statistical significance of the model. In addition to

presenting $F_{calculated}$ of the lack of fit inferior to the $F_{tabled}$, which indicates that the polynomial model presents a satisfactory fit to the experimental data.

$$DT = 33272 - 7875 \times UA - 5625 \times DT + 1875 \times UA \times DT + 11.25 \times 10 \times UA - 11.25 \times 10 \times DT - 375 \quad (12)$$

$$Def = -6.22 - 0.05 \times UA + 0.17 \times DT + 0.003 \times 10 \times UA - 0.007 \times DT + 0.001 \times UA \times DT + 2.73 \quad (13)$$

Where: DT is drying temperature, UA is ultrasound amplitude and D*ef* is effective diffusivity.

According to Box & Wetz, for regression to be significant and predictive, the value of the ratio between the $F_{calculated}$ and the $F_{tabulated}$ must be at least greater than 4.

Therefore, linear models obtained for drying time and effective diffusivity are predictive. Response surface plots for drying time and effective diffusivity are shown in Figure 5.

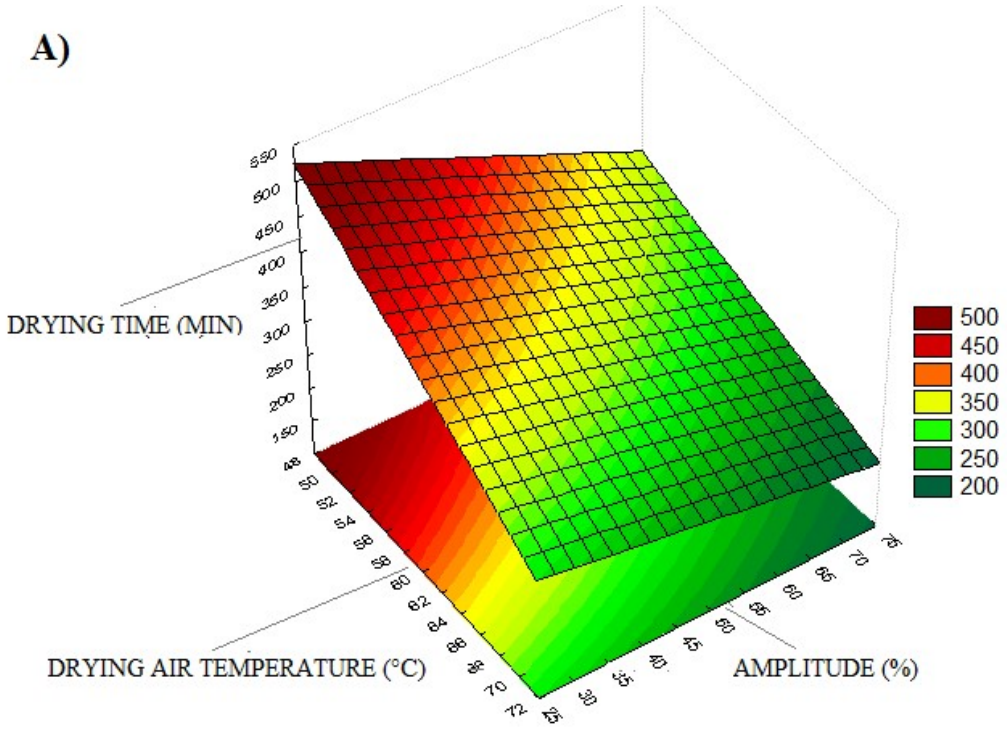


**Figure 5 A-** Response surface graph for drying time diffusivity with ultrasound time corresponding to 15 min.

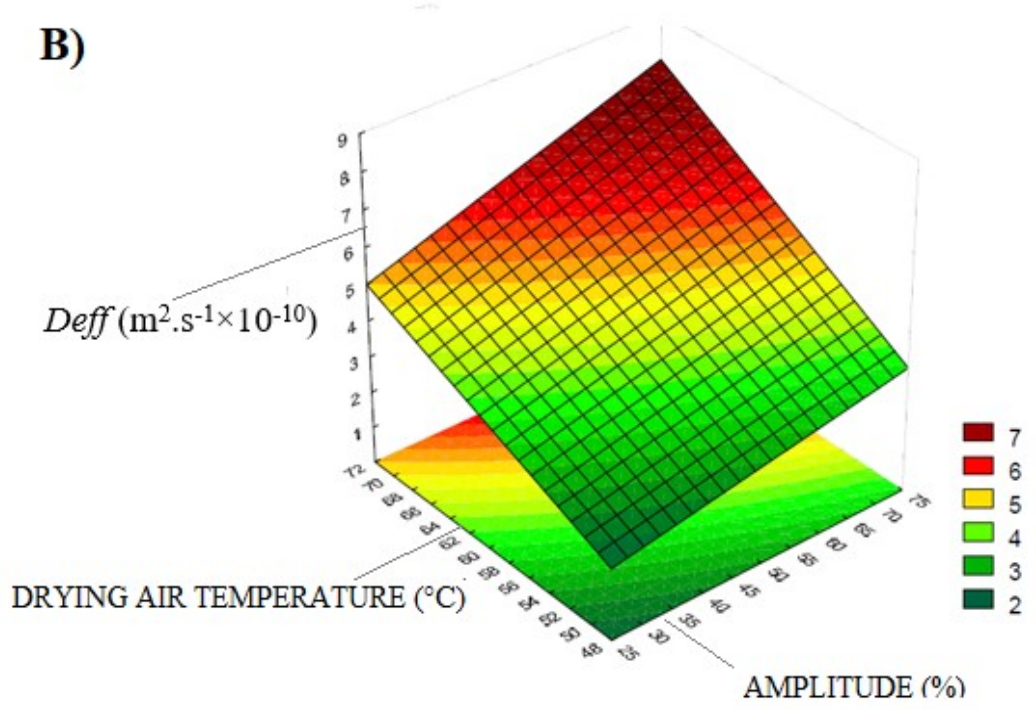


**Figure 5 B-** Response surface graph for effective diffusivity with ultrasound time corresponding to 15 min.

In Figure 5A, the response surfaces for the drying time are presented, which allows the observation of the influence of the drying temperature and the ultrasound amplitude on the drying time. The increase in ultrasound amplitude and temperature promoted a significant reduction in drying time, which was lower when the higher levels (+1) were applied to the independent variables. Experiment E8 allowed a greater reduction in the convective drying time, which corresponded to 210 min. The longest drying time was observed in the experiment that was not subjected to the ultrasound process, and the drying time was 50 °C. Rashid et al. (2019) also found that the ultrasound frequency promotes a significant reduction in the drying time of the samples, possibly due to the increase in the drying coefficient values and effective diffusivity. The response surface obtained for the effective diffusivity is shown in Figure 5B, through which it can be inferred that the increase in the drying temperature and ultrasound amplitude promoted an increase in the effective diffusivity of the product. The higher values of this parameter occurred at the highest drying air temperature (+1) and ultrasound amplitude (+1). Magalhães et al. (2017b) observed similar behavior in the ultrasound-assisted air-drying.

The optimized conditions for drying time (Figure 5A) and diffusivity (Figure 5B) use an amplitude of 70% and a drying temperature of 70ºC. This result converges with the data obtained in the research, indicating that the predictive model is highly reliable.

### 3.5 Numerical solution of the diffusion model: distribution of water content

The diffusion equation numerically solved through infinite volumes, with a completely implicit formulation, made it possible to observe the water content distribution in the samples observed in Figure 6.

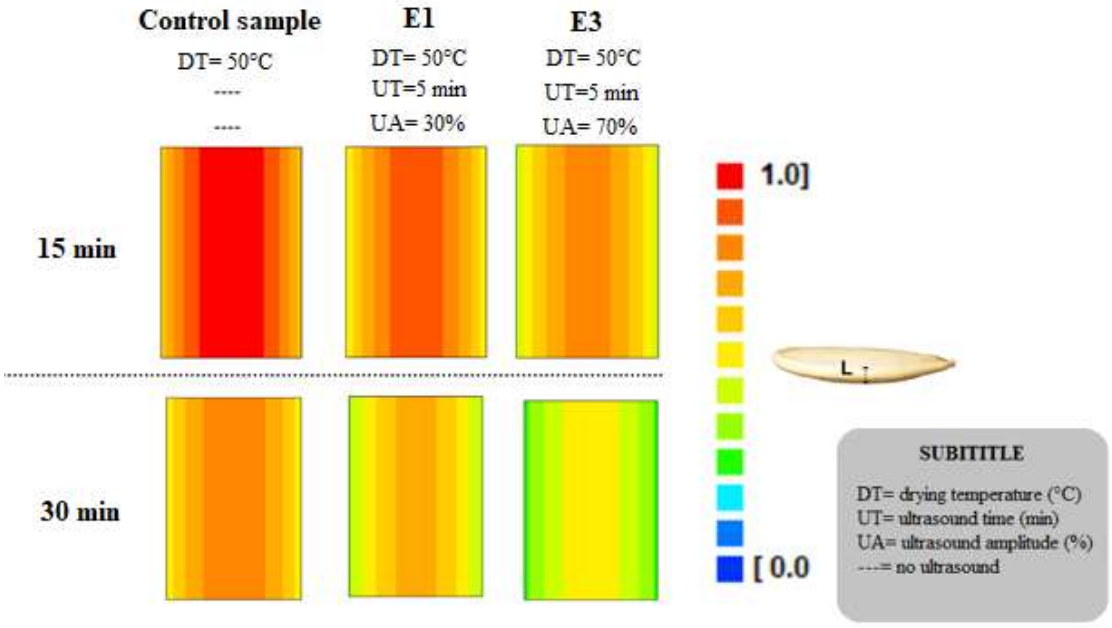


**Figure 6 -** Spatial distribution of the water content of the control experiment, E1, and E3 at a temperature of 50 ° C at times t = 15 and 30 min.

The samples (control, E1, and E3) were selected so that it was possible to observe the influence of the amplitude used in the ultrasound process on the water content distribution in the product at different drying times (15 and 30 min). From Figure 6, it was possible to analyze the moisture distribution during the convective drying process of pumpkin seeds along with its experimental domain (thickness of approximately 2.6 mm). Significant moisture gradients was observed, indicating that drying does not occur uniformly along with the seed. The reduction of the water content during the drying process initially occurred in the peripheral regions of the infinite wall, until reaching the central region.

It can still be observed that the control sample requires a longer time in the drying process to reach the equilibrium condition when compared to experiments E1 and E3. Among the samples submitted to the ultrasound process, it can be observed that the E3 sample presented a greater reduction in the humidity gradients, indicating that the ultrasound process promoted a greater uniformity in the loss of water in the product during the drying process. In this sense, it is suggested that the amplitude applied in the ultrasound process directly influences the moisture distribution. Tao et al. (2019), when evaluating the influence of the ultrasound process on the moisture distribution in the drying of white cabbage, and the authors observed that the samples that were subjected to the ultrasound process showed a more homogeneous moisture distribution, when compared to the samples that were not subjected to ultrasound. It was also verified that the increase in drying time promotes the reduction of moisture gradients.

## CONCLUSIONS

The study showed that using ultrasound in the drying process makes it feasible to use pumpkin seeds. This method is cost-effective and provides better storage conditions. The dried seeds can be consumed directly or used as raw material in other production processes. Optimized drying conditions were obtained for seeds pre-treated by ultrasound for 15 min with an amplitude of 70% and submitted to convective drying at 70 °C (experiment 8), which provided the shortest drying time (210 min) and the highest value effective mass diffusivity and convective mass transfer coefficient . Consequently, under these conditions, the highest drying rate of the material was observed. The Page and the diffusion models presented the best adjustment (R2 >0.99) compared to the others. Therefore, it indicates that these models should be used to predict the drying behavior of pumpkin seeds. In addition, it was seen that the drying of the seeds does not occur uniformly. Further investigations into the influence of these variables on pumpkin seed quality parameters and oil extraction are ongoing in our laboratory.

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# APPENDIX C - PAPER 3- (PUBLISHED TO WASTE AND BIOMASS VALORIZATION)

# PHYSICAL-CHEMICAL, FUNCTIONAL AND ANTIOXIDANT PROPERTIES OF DEHYDRATED PUMPKIN SEEDS: EFFECTS OF ULTRASOUND TIME AND AMPLITUDE AND DRYING TEMPERATURE


## ABSTRACT

This study aimed to investigate the influence of ultrasound pretreatment conditions and drying temperature on the quality parameters of pumpkin seeds. Various parameters, including drying time, water content, water activity, pH, total titratable acidity, proteins and lipid content, vitamin C, antioxidant activity and instrumental color, were evaluated. An experimental design ($2^3$ + 3 central points) was employed, with independent variables of ultrasound time (5, 10 and 15 min), ultrasound amplitude (30, 50 and 70 %), and drying temperature (50, 60 and 70 °C). The water content and water activity showed lower values in samples submitted to higher ultrasound amplitudes and drying temperatures. The pH of the samples ranged from 6.90 to 7.04, and the total titratable acidity ranged from 1.83 to 2.39. Milder ultrasound experiments promoted more remarkable preservation of vitamin C than the others, presenting values ranging from 15.76 to 26.02 mg/100g. The increase in drying air temperature caused more significant protein denaturation. The antioxidant potential was not affected by the application of the ultrasound process. Among the compounds evaluated in the phenolic profile, Procyanidin B2 and Gallic Acid components were predominantly present. The most abundant fatty acids in the samples were oleic and linoleic. The results provide insights into the optimization of the drying process and the quality attributes of pumpkin seeds.


**KEYWORDS:** Pumpkin seed, Response surface methodology, Multivariate analyses, Phenolic profile, Fatty acids.

## 1. INTRODUCTION

Consumers are looking for more natural foods with more ingredients. For this reason, the intake of plant foods has been increasing; an example is a pumpkin (*Cucurbita moschata*), whose cultivation expanded in Asia, Europe, and South America. On the other hand, the increase in production is a consequence of the increase in the generation of items such as seeds and husks (Wang et al., 2021).

Seeds represent about 10% of the total weight of pumpkins and have been highlighted in the industrial by-product due to their high nutritional value, in addition to performing health protection activities, such as antioxidant, anti-inflammatory, hypoglycemic, and lipid-lowering activities (Vinayashree & Vasu, 2021). Considering the global production of pumpkins and the proportion represented by their seeds, it can be estimated that approximately 912.2 thousand tons of seeds are generated as an industrial by-product each year (Ferreira et al., 2021). However, if these by-products are not properly reused or disposed of, they can contribute significantly to environmental pollution (Santos et al., 2023).

One of the advantages associated with the consumption of pumpkin seeds is that, unlike some oilseeds, cases of allergic reactions caused by their ingestion are rare (Kotecka-Majchrzak et al., 2020). In addition, pumpkin seeds contain a substantial amount of proteins, fibers, lipids and minerals, and other nutrients such as starch, carotenoids, and bioactive compounds, like tocopherols, sterols, α-carotene, and lutein, as well as vitamins. The most considerable fraction of pumpkin seed protein contains globulin and albumin, in less abundant fractions are prolamins and glutelins (Ferreira et al., 2021).

Despite this tremendous nutritional potential, the use of pumpkin seeds in human food is relatively low, being necessary to promote studies to observe the potential of the use of pumpkin seeds and the implications that this use can promote to the environment and people's eating habits (Rojas et al., 2019; Ferreira et al., 2021).

One of the biggest challenges in conserving pumpkin seeds occurs due to their high water content, which allows chemical, microbial, and enzymatic reactions, causing damage to the quality of the product and reducing its shelf life (Chao et al., 2022). Furthermore, bioactive compounds decrease during transport and storage as bacterial and fungal contamination risk increases. Convection drying in an air-circulated oven is an effective, low-cost, versatile and easy to operate (Chikpah et al., 2022). This technology combines conduction and convection mechanisms, promoting a more significant reduction in the water content and water activity of the products, causing the growth of microorganisms, reducing the occurrence of degradation

reactions and to prevent the decomposition of phytoconstituents and nutritious compounds, consequently allowing an increase in the shelf life of these products (Ramarao et al., 2022).

Despite the various advantages associated with applying convective drying to products of plant origin, there are some changes in the composition of the food (nutrient losses) and significant changes in its structure, which negatively influence the sensory parameters (color, flavor, texture) (Miano et al., 2021). Pre-treatment such as osmotic dehydration, ultrasound, high hydrostatic pressure, pulsed electric fields, and immersion in ethanol is commonly used to reduce drying time and improve the final product's quality (Kroehnke et al., 2021).

Process-assisted convective drying (Hybrid drying), in particular ultrasonic pre-treatment, can be used to overcome the additional problems of convection drying (Bagheri and Dinani, 2019). For example, the ultrasound (US) process enhances the preservation of food quality attributes by reducing the time required to complete the heat treatment (XU et al., 2021). This study aims to evaluate the effect of ultrasound time and amplitude and drying temperature on the physical-chemical, functional and antioxidant properties of pumpkin seeds. Therefore, this study aims to provide relevant information to the food industry, promoting the sustainable use of pumpkin seeds and encouraging the diversification of high-value-added food products.

## 2. MATERIALS AND METHODS

### 2.1 Plant material and sample preparation

Pumpkin seeds (*Cucurbita maxima*) were supplied by a minimally processed vegetable products company located in Fortaleza, Ceará, Brazil, In January 2022. The seeds were taken to the tropical fruit laboratory, in the Federal University of Ceará, for washing in distilled water, sanitizing in a sodium hypochlorite solution at 200 ppm for 15 minutes, and removing the unselected materials. After the washing and sanitizing process, a paper towel was used to remove excess water from the surface of the seeds. Afterward, the samples were stored at -20 °C until the experiments were carried out.

### 2.2 Experimental planning

To investigate the impact of ultrasound pretreatment conditions and drying temperature on various quality parameters of pumpkin seeds (including instrumental color, drying time, water content, water activity, pH, total titratable acidity, proteins, vitamin C, and lipid content), an experimental design was employed. The independent variables, including

ultrasound time (5, 10, and 15 min), ultrasound amplitude (30, 50, and 70%), and drying temperature (50, 60, and 70 °C), were utilized to construct a $2^3$ experimental design with three repetitions at the central point. This resulted in a total of 11 experiments, as presented in Table 1.

**Table 1-** Planning matrix for the ultrasound process and convective drying of pumpkin seed with its independent variables (coded and real values).

| Experiments | Independent variables | | |
|---|---|---|---|
| | Ultrasound time (min) | Amplitude (%) | Drying temperature (°C) |
| E1 | -1 (5) | -1 (30) | -1 (50) |
| E2 | 1 (15) | -1 (30) | -1 (50) |
| E3 | -1 (5) | 1 (70) | -1 (50) |
| E4 | 1 (15) | 1 (70) | -1 (50) |
| E5 | -1 (5) | -1 (30) | 1 (70) |
| E6 | 1 (15) | -1 (30) | 1 (70) |
| E7 | -1 (5) | 1 (70) | 1 (70) |
| E8 | 1 (15) | 1 (70) | 1 (70) |
| E9 (C) | 0 (10) | 0 (50) | 0 (60) |
| E10 (C) | 0 (10) | 0 (50) | 0 (60) |
| E11 (C) | 0 (10) | 0 (50) | 0 (60) |

Note: C=Center point

### 2.3 Ultrasound and drying process

The ultrasonic process was carried out in a 400 W ultrasonic processor (UP400S, Hielscher Ultrasonics, Teltow, Germany), with an H22 titanium sonotrode centrally immersed in a beaker with distilled water and pumpkin seeds. The ratio was 1 g of sample to 10 mL of water, working with a fixed cycle (1.0) and amplitude varying according to the experimental design described in Table 1. After the ultrasound process, the excess of water was removed on the material by using a paper towel.

In the convective drying process, the pumpkin seeds were weighed in a petri dish (insert the amount) and evenly distributed forming a thin layer, being subjected to drying at

temperatures of 50, 60 and 70 °C, using an air circulation oven (TECHNAL, model TE-394/I) with a fixed speed of 1.5 m $s^{-1}$. For comparative purposes, a group of seeds that were not subjected to pre-treatment with ultrasound were also subjected to drying at the same temperatures and were named control samples. For all cases, the drying process was performed in triplicate and continued until the constant (equilibrium) mass reading was recorded. After drying, the seeds were grounded in an analytical mill (IKA A11® basic mill) and sieved in 18 mesh (1.0 mm) to obtain flours with homogeneous granulometry.

### 2.4 Sample characterization

#### 2.4.1 Water content and water activity (aw)

The water content was determined by drying the samples to constant weight in an oven at 105 °C according to the methodology proposed by AOAC (2016) and the water activity ($a_w$) was determined by sample direct reading, using the Aqualab equipment, model 4TE (Hygrometer, WA, USA), at temperature 25 °C.

#### 2.4.2 pH and total titratable acidity (TTA)

The potentiometric method determined the pH by using a calibrated meter (Q400MT, Quimis, Brazil) with pH 4.0 and 7.0 buffer solutions and total titratable acidity (TTA) was evaluated according to AOAC, (2016).

#### 2.4.3 Protein and lipids

Protein content was established by the Kjeldahl method, with the sample digestion in $H_2SO_4$, the release of ammonia by adding NaOH, and titration of ammonia with HCl (AOAC, 2016). The lipid content of the flours was determined using the Soxhlet system for 8 hours. Hexane was used as a solvent and was subsequently removed through a rotary evaporator, according to the AOAC methodology (AOAC, 2016).

#### 2.4.4 Vitamin C

Tillman's method determined the ascorbic acid (Vitamin C) content (AOAC, 2016).

#### 2.4.5 Instrumental color

Instrumental color was determined by the ColorQuest-XE colorimeter (HunterLab, Virginia, USA) according to the average of the readings of the CIE lab coordinates, being L*, a*, b*, Chroma (c*), and Hue Angle (h *).

### 2.5 Antioxidant activity

The antioxidant activity of pumpkin seed flours was determined in triplicate using the ABTS and DPPH free radical scavenging methods, according to Re et al. (1999) and Kim, Guo, and Packer (2002), respectively. The determination of antioxidants by FRAP was performed according to the methodology described by Rufino et al. (2006). The results were compared with a standard curve of ferrous sulfate at 100–2000 μmol $L^{-1}$, and expressed in mmol of $Fe^{2+}$ per liter of sample. The Trolox analytical standard was used to construct the analytical curve, and the results were expressed as Trolox equivalent per liter of extract (mmol TEAC $L^{-1}$).

### 2.6 Extraction and Quantification of the Individual Phenolic Compounds by RP-HPLC/DAD

According to the results obtained, experiments with the highest nutritional quality in terms of protein, lipid, and vitamin C content were selected for the analysis and quantification of individual phenolic compounds. Among the experiments carried out, as a control sample (without ultrasound), E1 (5 min of ultrasound, 30% amplitude) and E3 (5 min of ultrasound, 70% amplitude) were dehydrated at the drying temperature corresponding to 50° C. Both were selected to determine the ultrasound process's influence on the samples' phenolic compounds profile.

Pumpkin phenolic compounds were extracted by the solid-liquid product method, following a method described by Burin et al. (2014). Two organic phases were combined and evaporated on a rotary evaporator with temperature control (35 ± 1°C), the remaining coupling from the red coupling system was dissolved in 2 mL of 50% v/v methanol. Phenolic compounds were identified and quantified using the Agilent 260 Infinity LC coupling systemThe column used was Zorbax Eclipse Plus RP-C18 (100 × 4.6 mm, 3.5 μm) and the pre-column Zorbax C18 12.6 × 4.6 mm, 5 μm (Zorbax, USA). The temperature was 35 °C, and the injection volume was 20 μL. The solvent flow was 0.8 $mLmin^{-1}$.

The new gradient used in the separation was 0–5 min: 5% B; 5-14 min: 23% B; 14-30 min: 50% B; 30–33 min: 80% B, where solvent A was a 0.1 M phosphoric acid solution (pH

= 2.0) and solvent B was acidified with methanol with 0.5% $H_3PO_4$. The identification and quantification of the compounds were performed by comparison with external standards of gallic acid, syringic acid, p-coumaric acid, chlorogenic acid, trans-caftaric acid, caffeic acid, hesperidin, naringenin, procyanidin B1, catechin, epicatechin and procyanidin B2 from Sigma Aldrich (St. Louis, MO, USA) (DUTRA et al., 2018; PADILHA et al., 2017). Epigallocatechin, epicatechin gallate, procyanidin A2 quercetin 3-glucoside, rutin (quercetin 3-rutinoside), kaempferol 3-glycoside, myricetin from Extrasynthesis (Genay, France). Trans-resveratrol and cis-resveratrol were obtained from the Cayman Chemical Company (Michigan, USA).

### 2.7 Lipid content and determination of fatty acids

The lipid content of the selected flours was determined according to the AOAC methodology (AOAC, 2016). The oil was extracted from pumpkin seed flours using Hexane for 8 hours in a Soxhlet system. The solvent was evaporated using a rotary evaporator, and the extracted oil was stored at 20°C until further use.

According to the results obtained for the protein and lipid contents, the control samples (without ultrasound), E1 (5 min of ultrasound, 30% amplitude), and E3 (5 min of ultrasound, 70% amplitude) were selected. To analyze the ultrasound process's influence on the samples' fatty acid composition, both E1 and E3 were dehydrated at the drying air temperature of 50ºC.

Before fatty acid analysis, fatty acid methyl esters were prepared from the extracted oils described above (He et al., 2016), with some modifications. Briefly, a 100 mg oil sample was mixed with 2 mL of Hexane, vortexed for 1 min, then 2 mL of 2mol/L KOH in methanol and 2 mL of methanol were added, and the mixture was stirred with vortex for 5 min. After adding 2 mL of distilled water, the mixture was stirred, and the top layer was collected and subjected to treatment with anhydrous $Na_2SO_4$ to remove residual water. Fatty acid methyl esters were analyzed by a gas chromatography apparatus equipped with a flame ionization detector and a capillary column (Ghafoor et al., 2019).

### 2.8 Gas chromatography/mass spectral analysis (GC-MS)

Chemical analysis of the trans-methylated constituents was performed with a Shimadzu Instrument (QP-2010 Ultra) using the following conditions column: Rtx-5MS (Cross bond 5%, diphenyl / 95% dimethyl polysiloxane) with 30m x 0.25 mm x 0.25 μm df; carrier gas: He (24.2mL/min, in constant linear velocity mode); the injector temperature was 250°C,

in mode division (1:100), and the detector temperature was 250°C. The column temperature was programmed at 140ºC for 10 minutes, then 140-250°C at 7°C/min for 10min, and 250ºC for 10 minutes; mass spectrum: electron impact 70 eV. The sample was injected in a volume of 1μL. Compounds were identified by their retention times relative to known compounds in GC and by comparing their mass spectra with those in the computer database (NIST) and published literature (Adams, 2001; Adams, 2007)

### 2.9 Statistic analyses

#### 2.9.1 Univariate analysis

All tests were performed in triplicate, the results were evaluated by analysis of variance (ANOVA) and average comparison by Tukey's test at 5% probability, using the Statistica software version 7.0 (Tulsa, USA, 2004) was used to build the experimental design, the response surface, and the Pareto plot ($P < 0.05$).

#### 2.9.2 Multivariate analyses

To evaluate the behavior of the phenolic profile and Fatty acids profile of the pumpkin seeds dried, the application of multivariate statistics was carried out using principal component analysis (PCA) with pretreatment of the data for normalization and scaling. The program SPSS Inc., version 17.0 (Chicago, IL, USA) was utilized.

## 3. RESULTS AND DISCUSSION

### 3.1 Evaluation of processing conditions on the quality parameters of pumpkin seeds

The pumpkin seed flours obtained through the drying process at different temperatures (with or without ultrasound) were characterized, and the results obtained about water content, $a_w$, pH, and TTA, were expressed in Table 2.

**Table 2** - Results of analysis of water content, water activity ($a_w$), pH, total titratable acidity (TTA), protein, vitamin C and lipids of pumpkin seed flours, with their respective standard deviations.

| Experiments | | Water content (%) | $a_w$ | pH | TTA | Proteins (g/100g) | Vitamin C (mg/100g) | Lipids (g/100g) | Colorimetry | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | L* | a* | b* |
| **Fresh sample** | | 38.59±0.08 | 0.974±0.03 | 6.92±0.01 | 2.39±0.05 | 37.84±0.02 | 35,12±0.02 | 22.34±0.05 | - | - | - |
| **Control samples** | **50°C** | 5.09±0.14 $^{a}$ | 0.41±0.01$^{a}$ | 7.07$^{b}$ | 2.01±0.01$^{ab}$ | 35.71±0.01 $^{a}$ | 8.53±0.10$^{c}$ | 34.61±0.1 $^{ef}$ | 66.40±0.02$^{cde}$ | 2.65±0.01$^{cde}$ | 21.58±0.02$^{cde}$ |
| | **60°C** | 4.92±0.53 $^{ab}$ | 0.39±0.01$^{ab}$ | 7.04$^{b}$ | 2.04±0.01$^{ab}$ | 36±0.01 $^{a}$ | 8.42±0.21$^{c}$ | 34.52±0.1$^{f}$ | 65.49±0.11 $^{efg}$ | 3.30±0.01$^{a}$ | 23.14±0.01$^{ab}$ |
| | **70° C** | 4.73±0.03 $^{ab}$ | 0.37±0.03$^{bc}$ | 7.04±0.03$^{b}$ | 2.12±0.03$^{ab}$ | 30.99±0.01 $^{d}$ | 4.21±0.11$^{d}$ | 35.74±0.44 $^{cdef}$ | 66.74±0.01$^{bcd}$ | 2.49±0.03$^{ef}$ | 21.65±0.03$^{cde}$ |
| **With ultrasound** | **1** | 3.71±0.06 $^{fg}$ | 0.36±0.01 $^{bc}$ | 6.90$^{d}$ | 1.83±0.01 $^{b}$ | 36.88±0.01 $^{a}$ | 26.02±0.01$^{a}$ | 38.22±0.4 $^{a}$ | 65.74±0.02 $^{ef}$ | 2.98±0.02$^{b}$ | 22.41±0.02$^{abcd}$ |
| | **2** | 4.68±0.06 $^{abc}$ | 0.35±0.01 $^{c}$ | 7.01±0.06 $^{bc}$ | 2.27±0.01 $^{a}$ | 35.13±0.01 $^{ab}$ | 17.34±0$^{b}$ | 36.48±0.28$^{abcd}$ | 67.38±0.03$^{cde}$ | 2.88±0.03$^{bc}$ | 22.16±0.03$^{bcde}$ |
| | **3** | 4.48±0.03 $^{bcde}$ | 0.34±0.01 $^{cd}$ | 7.15$^{a}$ | 2.03±0.01 $^{ab}$ | 34.42 ±0.01 $^{abc}$ | 17.34±0.01 $^{b}$ | 37.93±0.42 $^{ab}$ | 67.38±0.01 $^{bc}$ | 2.66±0.01$^{cde}$ | 21.06±0.02$^{def}$ |
| | **4** | 4.12±0.12 $^{cdef}$ | 0.30±0.02 $^{de}$ | 7.16$^{a}$ | 2.09±0.02 $^{ab}$ | 35.10±0.01 $^{ab}$ | 17.19±0.21$^{b}$ | 37.07±0.2 $^{abcd}$ | 68.61±0.4 $^{a}$ | 2.25±0.04 $^{f}$ | 20.71±0.03 $^{ef}$ |
| | **5** | 4.61±0.8 $^{abcd}$ | 0.34±0.01 $^{cd}$ | 6.92$^{d}$ | 2.08±0.01 $^{cd}$ | 31.74±0.01 $^{d}$ | 15.76±0.9 $^{b}$ | 36.78±0.06 $^{abcd}$ | 64.55±0.3 $^{gh}$ | 2.81±0.02$^{bcd}$ | 23.28±0.05 $^{ab}$ |
| | **6** | 3.67±0.17 $^{fg}$ | 0.33±0.01 $^{cd}$ | 6.94±0.01 $^{cd}$ | 2.09±0.01 $^{cd}$ | 32.49±0.01 $^{bcd}$ | 8.47±0.07$^{c}$ | 37.08±0.09 $^{abcd}$ | 66.10±0.11 $^{de}$ | 2.55±0.03$^{de}$ | 21.99±0.02$^{bcde}$ |
| | **7** | 3.39±0.01$^{g}$ | 0.26±0.01 $^{fg}$ | 6.95±0.04 $^{cd}$ | 2.09±0.01 $^{ab}$ | 30.74±0.01 $^{d}$ | 8.29±0.019$^{c}$ | 35.54±0.01 $^{def}$ | 64.11±0.01$^{h}$ | 2.82±0.04$^{bcd}$ | 22.94±0.02$^{abc}$ |
| | **8** | 3.25±0.08 $^{g}$ | 0.25±0.012 $^{g}$ | 7.05$^{b}$ | 2.22±0.012$^{ab}$ | 30.30±0.01 $^{d}$ | 8.34±0.25$^{c}$ | 36.35±0.12 $^{bcde}$ | 67.55±0.05 $^{b}$ | 2.24±0.01 $^{f}$ | 19.89±0.02 $^{f}$ |
| | **9** | 3.99±0.04 $^{ef}$ | 0.29±0.01$^{ef}$ | 6.91$^{d}$ | 2.22 ±0.01$^{a}$ | 32.83±0.01 $^{bcd}$ | 8.03±0.02$^{cd}$ | 37.45±0.32 $^{ABC}$ | 63.90±0.07 $^{h}$ | 3.5±0.02 $^{a}$ | 23.89±0.01 $^{a}$ |
| | **10** | 4.08±0.04$^{def}$ | 0.29±0.01 $^{ef}$ | 6.90±0.01 $^{d}$ | 2.23 ±0.01 $^{a}$ | 32.53±0.01 $^{bcd}$ | 8.42±0.21$^{c}$ | 37.26±0.04 $^{abcd}$ | 64.86±0.02$^{fgh}$ | 3.58±0.01$^{a}$ | 23.45±0.01$^{ab}$ |
| | **11** | 4.03±0.01$^{def}$ | 0.29±0.03 $^{ef}$ | 6.90±0.03 $^{d}$ | 2.23 ±0.03 $^{a}$ | 32.068±0.01 $^{cd}$ | 8.41±0.20$^{c}$ | 37.02±0.01 $^{abcd}$ | 64.89±0.15$^{fgh}$ | 3.43±0.02 $^{a}$ | 22.99±0.03 $^{a}$ |

Note: Means followed by the same lowercase letter in the columns do not differ statistically by Tukey's test at 5% probability. Results are expressed as mean ± standard deviation (n=3). TTA in % oleic acid. UT= ultrasound time; UA= ultrasound amplitude; DT= drying temperature. Control sample 50°C (No ultrasound; DT=50°C); Control sample 60°C (No ultrasound; DT=60°C); Control sample 70°C (No ultrasound; DT=70°C); E1 (UT=5min UA=30% DT=50°C); E1 (UT=15min UA=30% DT=50°C); E3 (UT=5min UA=70% DT=50°C); E4 (UT=15min UA=70% DT=50°C); E5 (UT=5min UA=30% DT=70°C); E6 (UT=15min UA=30% DT=70°C); E7 (UT=5min UA=70% DT=70°C); E8 (UT=15min UA=70% DT=70°C); E9, E10, E11 (Central points; UT=10 min UA=50% DT=60°C).

According to Malegori et al. (2022), water content is an important parameter that indicates the amount of water in the product and the water supply activity over the amount of water for massive, enzymatic, microbial and oxidative reactions.

The water content of dry seeds, whether or not subjected to the ultrasound process, is below the ideal, ranging from 3.25 to 5.09%. It is possible to observe that there was no difference between the control points, without the previous ultrasound process, with temperatures of 50°, 60°, and 70°C and treatments 2 and 5. The minimum amplitude used during the ultrasound could not cause significant differences between treatments with and without this process. Lower water content values were found in treatments 7 and 8, with greater amplitude and drying temperature, with variation in ultrasound times, shorter and longer, respectively. The pre-treatment ultrasound facilitates the removal of water during the drying of seeds, probably because the sponge effect leads to the formation of micropores in the cell walls of the material (Santos et al., 2020).

The National Health Surveillance Agency (ANVISA), through resolution n°263, defined flour as products obtained from the edible parts of one or more species of cereals, legumes, fruits, seeds, tubers, and rhizomes by milling and or other technological processes considered safe for food production and recommended that the maximum water content of flours should be 15% (BRASIL, 2015).

Water activity is a parameter importance for the shelf life of a product. However, the evaluation of food stability is not restricted to water activity, as food matrices are complex environments, and therefore, microbiological safety is a challenge for the food industry (Malegori et al., 2022). The $a_w$ values range from 0.25 to 0.41, indicating that the flours have low susceptibility to attack by microorganisms. The lowest value concerning this parameter was observed in treatment 8, whose conditions were pre-treatment of 15 min in ultrasound with amplitude of 70% and drying temperature of 70%. Brazilian legislation does not define a range of water activity (aw) considered ideal as flours. Nevertheless, Barros et al. (2019a) stated that reduced values for this parameter may represent more excellent stability of the product during its storage and that products with a water activity below 0.60 are less susceptible to deterioration caused by microorganisms and biochemical reactions. Thus, all experiments in the present study showed values that contribute to providing better stability to the product during storage.

Kowalski et al. (2016) presented results using airborne ultrasound to aid in drying raspberries. Baeghbali et al. (2020), when observing the effects of ultrasound and drying techniques on the physicochemical properties of okra, obtained water activity values from 0.14

to 0.22. However, although it is not necessary to reduce the occasional water activity of the low products, the authors claim that this increases the value of the autoxidation of lipids. In general, the values obtained for water content and aw of the produced flours were significantly reduced by up to 38.59% and 0.97, respectively, in relation to fresh seeds.

The pH is a determining factor for the development of microorganisms in food. pH values lower than 4.5 reduce the risk of microorganism growth, and food samples with a pH greater than 4.5 may indicate greater susceptibility to the development of microorganisms when associated with other factors, such as high water content (Ferreira, Vieira, and Nitschke, 2019). The pH of the treatments ranged from 6.90 to 7.16, values close to neutrality. A significant difference was observed between the samples. However, it was not possible to identify a correlation between the drying air temperature and the ultrasound variables (amplitude and time) in the pH of the samples. A higher pH value was observed in experiment 4 (7.16), which did not differ significantly from experiment 3 (7.15), experiments 3 and 4 showed higher pH when compared to control samples (7.04 to 7.07). Lower values regarding this parameter were observed in experiments 1, 9, and 11, which did not differ significantly from experiments 10, 6, and 7.

The samples showed low values of total titratable acidity (TTA), ranging from 1.83 to 2.27%, expressed in the percentage of oleic acid. Acidity is inversely proportional to pH values, thus being a significant factor related to food stability. In addition, this parameter plays an essential role in food taste, representing one of the basic tastes. Amadeu et al. (2021) observed pH and acidity results similar to those obtained in the present study when obtaining germinated pumpkin seed flour. Bouvie et al. (2016) obtained pH of 5.23 and 6.23 for Brazil nut (*Bertholletia excelsa*) mesocarp and exocarp, respectively. Barros et al. (2019b), when evaluating the influence of drying at different drying air temperatures of shelled sunflower seeds, obtained a pH ranging from 6.82 to 6.85 but did not find a statistically significant difference between the experiments. When analyzing the total titratable acidity, only seeds submitted to a temperature of 70°C were superior to the others, according to the Tukey test at a 5% probability comparison.

Control samples submitted only to the drying process at 50 and 60°C showed higher values of protein (35.71 and 36%) when compared to the other treatments, except for treatment 1 (36.88%), whose ultrasound conditions were milder (lower time and amplitude), with the possibility of reduced protein denaturation. According to Dotto and Chacha (2020), fresh pumpkin seeds have about 21.31% protein on a dry basis and 40% on a wet basis, which is

similar to the value observed in this study (37.84g/100g on a wet basis). The Brazilian Ministry of Health (Brasil, 2015) established that solid food with protein content higher than 5% (5 g/100g) could be considered food rich in protein. In this sense, it can be inferred that pumpkin seed flours are rich in protein and have a high potential to meet the demand for plant-based proteins.

Therefore, it was observed that the proteins are susceptible to denaturation by exposure to high temperatures. However, the concentration of some components occurs due to the reduction of the water content of the pumpkin seed. Consequently, the drying process does not significantly compromise the protein percentage in pumpkin seeds. This behavior can be explained by the fact that lower temperatures and exposure time reduce the probability of protein denaturation. Although the ultrasound process is considered a form of non-thermal energy, the mechanical friction and the agitation of the molecules that occur due to the propagation of ultrasonic waves cause the temperature to rise. According to Zhang and Abatzoglou (2020), although the heating effects caused by the ultrasound process are mild, they should not be ignored.

Fresh pumpkin seeds have a high lipid content (22.34 g/100g), a value similar to that observed by Amin et al. (2019) in seeds of two pumpkin varieties, which obtained lipid values corresponding to 23.5 and 17.6 g/100g. When comparing the fresh seeds with the dehydrated samples, an increase in the lipid values was noticed due to the drying process, which varied from 34.52 to 38.52. According to Amadeu et al. (2021), the drying process causes an increase in the other constituents due to the concentration process resulting from the elimination of part of the water from the product.

Albuquerque et al. (2021) did not observe changes in the number of macronutrients in ultrasound-treated cactus (*Opuntia ficus-indica*) fruit juice. The authors attributed this behavior to the characteristic tendency of ultrasonic waves to more strongly influence low molecular weight structures. The slight variations observed in the present study concerning the percentage of lipids can be attributed to the drying process, which performs the concentration of these components.

When observing the vitamin C values of the control experiments (without the ultrasound process), a reduction of it can be noted with the addition of the drying air temperature, which varied from 8.53 to 4.21 mg/100g; this decrease occurs due to the fact that vitamin C is a thermosensitive compound. Compared to other experiments, it was possible to observe that the control samples (50 and 60°C) did not differ significantly from experiments 6,

7, 8, 9, 10, and 11. On the other hand, the highest value of vitamin C was observed in experiment 1, which differed significantly from the other samples. This result may indicate that experiment 1 (which has mild ultrasound and drying conditions) allowed the retention of vitamin C in the product. For samples treated with maximum amplitude and maximum temperature during drying, a considerable reduction in vitamin C content was observed.

The loss of vitamin C in ultrasound processes occurs through oxidation processes that produce and utilize hydroxyl radicals under aerobic and anaerobic conditions. For Xu et al. (2021), the previous ultrasonic treatment did not promote significant differences or a positive effect on vitamin C content in vacuum-freeze-dried strawberry slices, with or without pre-treatment.

Cao et al. (2019), when performing the drying kinetics of lychee fruits by ultrasound-assisted hot air drying, found that there was a significant decrease in vitamin C content after drying and did not find significant differences between the vitamin C content of the samples in which drying was assisted by ultrasound. The author associated vitamin C losses with high temperatures, which are also present in ultrasound treatments. The microbubbles formed by the ultrasound cavitation process promote cell membrane destruction and oxidation during processing.

When observing the values obtained for the luminosity (L*) of the samples, it is verified that there was no statistically significant difference between the control experiments. Thus, it can be inferred that the drying temperature did not influence the luminosity of the control parameters (samples that were not subjected to pre-treatment with ultrasound). When comparing the control experiment to the experiments E1, E2, E3, and E4 (all submitted to a drying temperature of 50 °C), it was verified that experiment E4 presented a significant difference and a higher value of luminosity (68.61) when compared to the other experiments. This fact can be explained by the more intense occurrence of the cavitation phenomenon at the highest amplitude level (70%) and long process time (15 min).

Similar behavior regarding the luminosity parameter was observed when comparing the control experiment and experiments E9, E10, and E11 (all submitted to a drying temperature of 60° C). When comparing the control experiment to experiments E5, E6, E7, and E8 (all submitted to a drying temperature of 70° C), it was verified that the control experiment did not differ significantly from experiments E8 and E5 (submitted to the ultrasound process). The highest luminosity value was observed in experiment E8 (15 min of ultrasound and 70%

amplitude). When comparing all experiments, it was found that the highest luminosity was observed in experiment E4, and the lowest value was observed in the E10 sample.

According to Nowacka et al. (2017), the use of ultrasound as a drying pre-treatment enables color maintenance. However, longer treatment times can decrease the values of the L * parameter due to the phenomenon of cavitation, which occurs during ultrasound application. Medeiros et al. (2016) reported an increase in the luminosity (L* coordinate) of dried mangoes when compared to that of fresh fruit.

All samples showed positive values for the a* coordinate, indicating greater proximity to the red color. About the a* coordinate, positive values indicate greater proximity to the red color, and negative values indicate proximity to the green color. When comparing the control experiment to other experiments (E1, E2, E3, and E4), which were all submitted to a drying temperature of 50° C, it was found that the control experiment presented a value of 2.65 for the parameter a* and did not differ from samples E2 and E3. A higher value was observed in experiment E1, which was submitted to the lowest time and ultrasound amplitude when comparing the control experiment to the experiments (E9, E10, and E11), which were all submitted to a drying temperature of 60°C, it was verified that the control experiment presented a value of 3.30 and did not differ significantly from the other experiments.

The parameter a* in the control experiment (70°C) presented a value of 2.49 and did not differ statistically from experiments E6 and E8. On the other hand, it differed significantly from experiments E5 and E7, which presented higher values (2.81 and 2.82, respectively). It can be affirmed that the experiments that were submitted to milder conditions of ultrasound showed higher positive values of a*, indicating less darkening of the seeds.

No significant difference was observed between the control experiment and experiments E9, E10, and E11, which were all submitted to a drying temperature of 60°C. When comparing the control experiment to other experiments (E5, E6, E7, and E8), which were all submitted to a drying temperature of 70°C, it was verified that the control experiment did not differ significantly from the E8 and E6 experiments (submitted to the ultrasound process). The highest a* value was observed in experiment E10 (drying temperature of 60°C, 10 min of ultrasound, and 50% amplitude), and the lowest value was observed in experiment E8.

The parameter b* represents the yellow and blue coloration. All experiments showed positive values about the b* parameter, indicating that the product has a colorimetry closer to the yellow color. Negative values concerning this parameter are attributed to blue coloring, and positive values are associated with yellow coloring.

When observing the values obtained for the parameter b* of the control samples, it was verified that the dehydrated experiment using the drying temperature of 60 °C presented higher values. The control experiments at 50 and 70 °C showed no statistically significant difference. There was also no statistically significant difference between the control experiment (60°C) and experiments E9, E10, and E11. When comparing the control experiment (70° C) and the E5, E6, E7, and E8 experiments, it can be seen that a significant statistical difference was only identified in the E5 and E8 experiments. A lower value concerning this parameter was observed in experiment E8 (19.89), and a higher value was observed in experiment E9 (23.89).

In this sense, it can be observed that milder ultrasound conditions (shorter time and smaller amplitude) and drying temperature can favor the preservation of seed color when comparing the experiments submitted to more severe ultrasound conditions and the control experiments. In Figures 1a, 1b, and 1C, the Pareto graphs obtained for water content, water activity, and proteins are expressed.

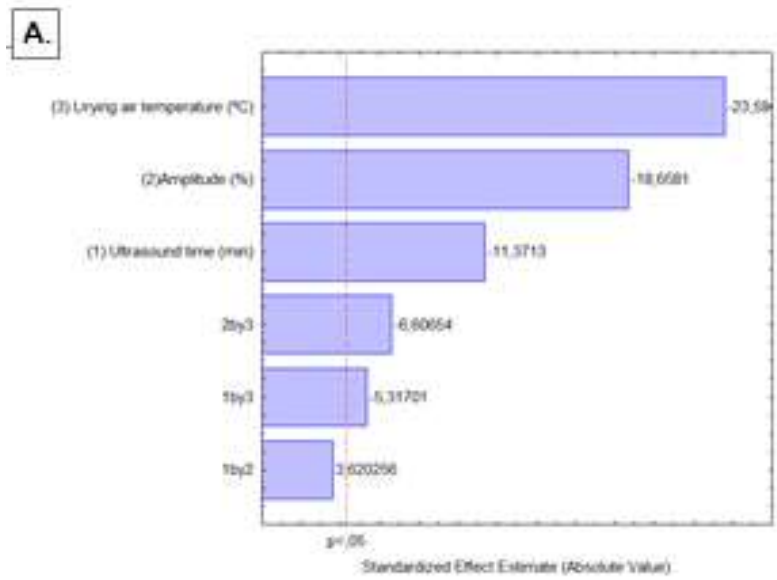


**Figure 1 A**- Pareto chart for the influence of drying temperature, amplitude, and ultrasound time in the following water content parameter.

Evaluating Figure 1a, it can be seen that the temperature of the drying air was the independent variable that had the most significant influence on the water content, followed by the amplitude used in ultrasound, ultrasound time, and the interaction between them. All independent variables showed decreasing effects (-) for water content.

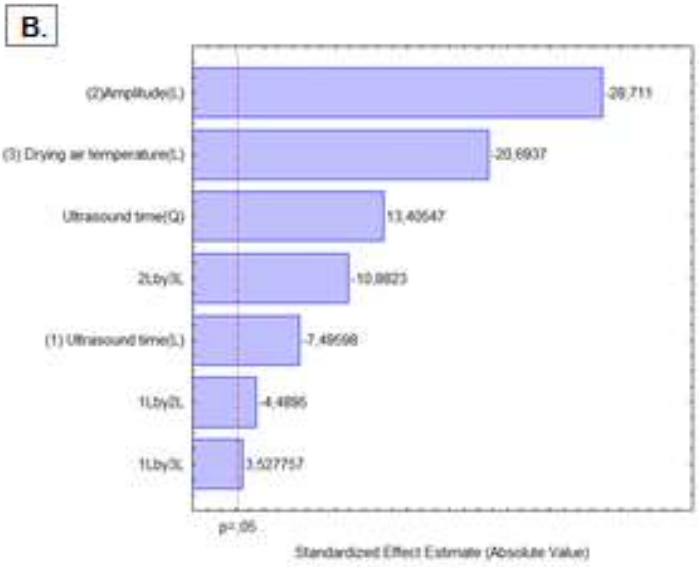


**Figure 1 B-** Pareto chart for the influence of drying temperature, amplitude, and ultrasound time in the following water activity parameter.

Regarding the Pareto graph obtained for the water activity (Figure 1b), it is observed that the variable that had the most significant influence on the response was the amplitude of the ultrasound, followed by the temperature of the drying air and the ultrasound time. The ultrasound amplitude and the drying air temperature showed decreasing effects (-) concerning water activity, while the ultrasound time showed an increasing effect (+).

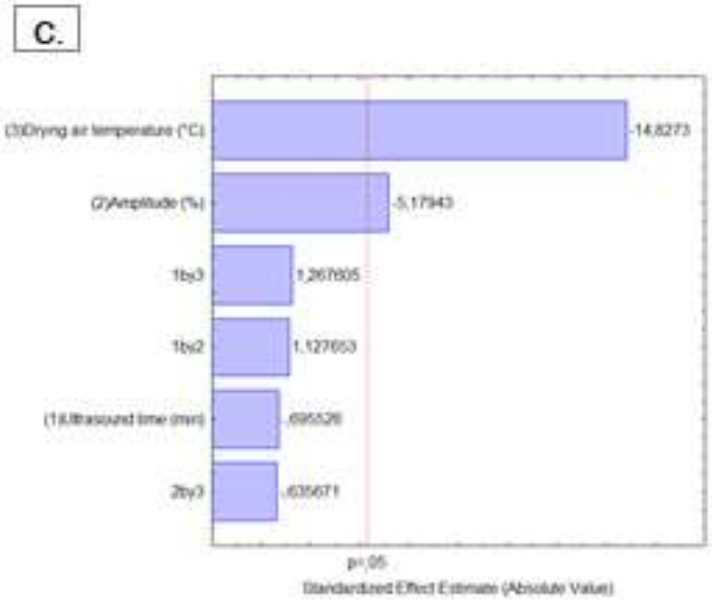

**Figure 1 C-** Pareto chart for the influence of drying temperature, amplitude, and ultrasound time in the following protein content parameter.

In Figure 1c, it can be seen that the significant parameters of these responses were the drying air temperature and the ultrasound amplitude, which showed decreasing effects on the protein content of the samples.

Table 3 expresses the analysis of variance (ANOVA) and the F test with 95% confidence only for the variables that were significant (water content, water activity, and proteins) in the ultrasound and drying process of pumpkin seeds; the other variables (pH, total titratable acidity, vitamin C, and lipids) were not significant for this confidence level, and therefore the response surfaces were not generated about these parameters.

**Table 3 -** ANOVA Table for the parameters of water content, water activity, and proteins.

| Factor | Source of variation | SQ | DF | QM | $F_{ocal}$ | $F_{ab}$ | $R^2$ (%) |
|---|---|---|---|---|---|---|---|
| Water content | Regression | 2.3523 | 3 | 0.7841 | 16.8091 | 6.59 | 98.76 |
| | Lack of fit | 0.1823 | 2 | 0.0911 | 13.3949 | 19.25 | |
| | Pure terror | 0.004 | 2 | 0.0021 | | | |
| | Total SS | 2.5389 | 7 | | | | |
| aw | Regression | 240567.7 | 5 | 48113.54 | 40941.42 | 5.05 | 99.16 |
| | Lack of fit | 4.557 | 3 | 1.52 | 2.30 | 9.01 | |
| | Pure terror | 1.31 | 2 | 0.66 | | | |
| | Total SS | 240573.6 | 10 | | | | |
| Proteins | Regression | 37.69 | 3 | 12.56 | 13.99 | 6.59 | 91.29 |
| | Lack of fit | 3.29 | 2 | 1.64 | 10.93 | 19.25 | |
| | Pure terror | 0.30 | 2 | 0.15 | | | |
| | Total SS | 41.29 | 7 | | | | |

Note: SQ= Sum of squares; DF = degree of freedom; QM= Medium square.

Through the ANOVA (Table 3), it was found that the calculated F of the regression is greater than the tabulated F concerning the parameters: water content (Equation 1), water

activity (Equation 2), and proteins (Equation 3), indicating that the first-order polynomial models presented for these parameters are statistically significant and adequately represent the influence of the independent variables (amplitude, ultrasound time and drying air temperature) on the independent variables.

According to Kamal et al. (2019), the aptitude of the models can be evaluated through the lack of fit test, and non-significant pure error values ($p<0.05$) indicate the acceptability of the models. The calculated F of the lack of fit obtained was lower than the tabulated F, indicating that the lack of fit models for these parameters was insignificant, so it can be inferred that the models fit well with the experimental data. The other parameters (color, pH, acidity, vitamin C and lipids) did not present statistically significant models for obtaining response surfaces.

$$WC = 5.17 + 0.006 \times DT + 0.002 \times y - 0.0005 \times DT \times y - 0.002 \times 10 \times DT + 0.006 \times 10 \times UA + 0.37 \quad (1)$$

$$WA = 0.38 - 0.00006 \times DT + 0.002 \times UA - 0.00006 \times DT \times UA + 0.0001 \times 10 \times DT - 0.00005 \times 10 \times UA - 0.04 \quad (2)$$

$$\mathrm{Pr} = 48.84 - 0.22 \times DT - 0.025 \times UA + 0.0035 \times 10 \times DT + 0.0015 \times 10 \times UA - 3.05 \quad (3)$$

Where: WC is Water content, WA is Water activity, Pr is proteins, DT is drying temperature, and UA is ultrasound amplitude.

From Figure 2, the response surface graphs for water content (a), water activity (b), and protein (c) are expressed.

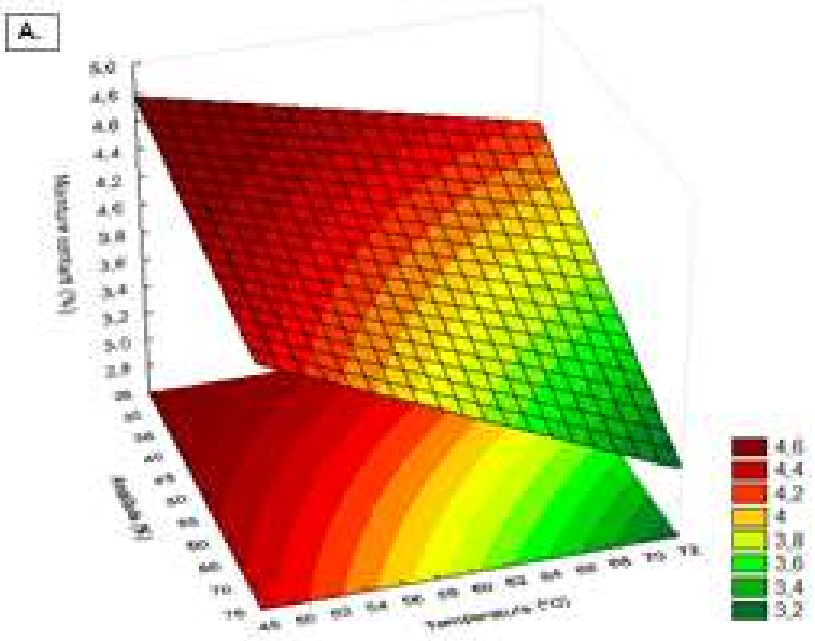


**Figure 2 A -** Response surface graph for the influence of drying temperature, amplitude and ultrasound time in the following water content parameter.

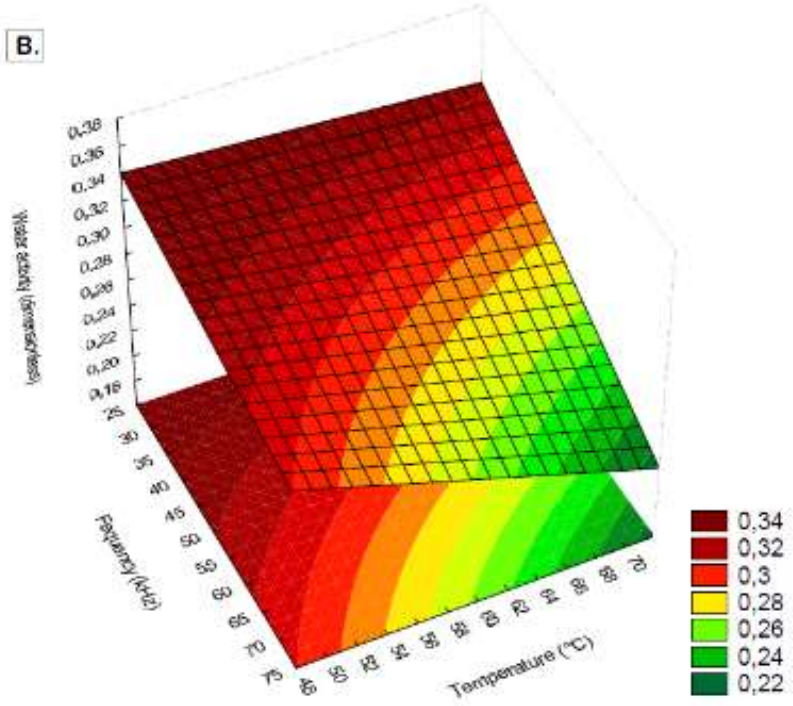


**Figure 2 B -** Response surface graph for the influence of drying temperature, amplitude and ultrasound time in the following water activity parameter.

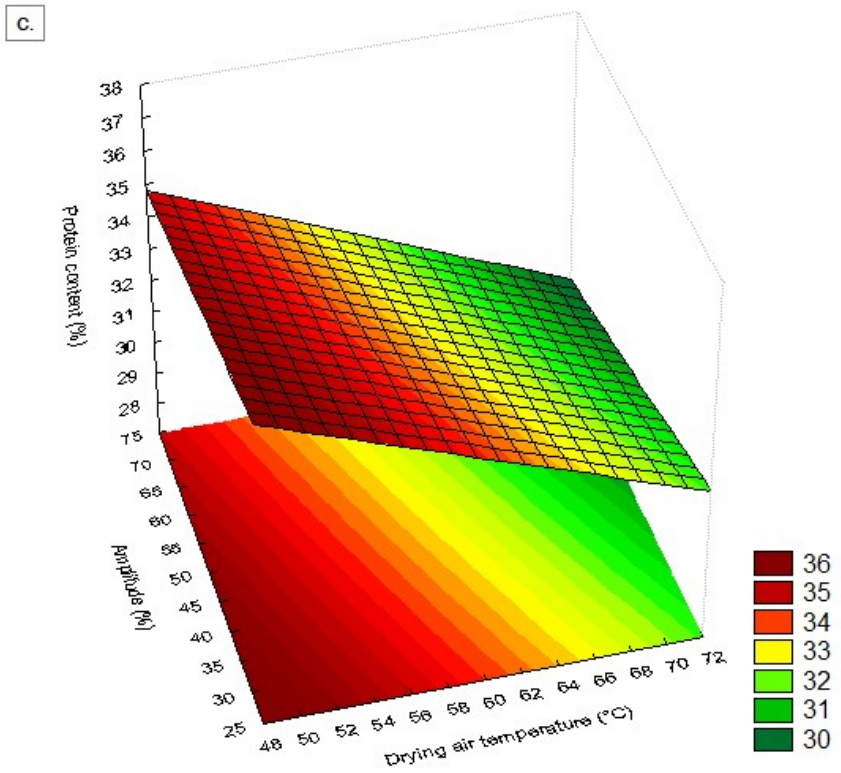


**Figure 2 C-** Response surface graph for the influence of drying temperature, amplitude and ultrasound time in the following protein content parameter.

From the response surface graph obtained for water content (Figure 2a) and water activity (Figure 2b), it can be inferred that lower values concerning these parameters were obtained when higher ultrasound frequencies and higher drying temperatures were used. Regarding the protein content (Figure 2c), it can be observed that the temperature of the drying air was the variable that had the most significant influence on this parameter and that higher values of proteins were obtained at the lowest levels of an air temperature of drying.

### 3.2 Antioxidant activity

The Control, E1, and E3 experiments (submitted to the same drying temperature) were selected to evaluate the effects of the ultrasound process on the antioxidant compounds. The results on the content of antioxidant compounds determined by the DPPH, $ABTS^{+}$, and FRAP methods are expressed in Figure 3.

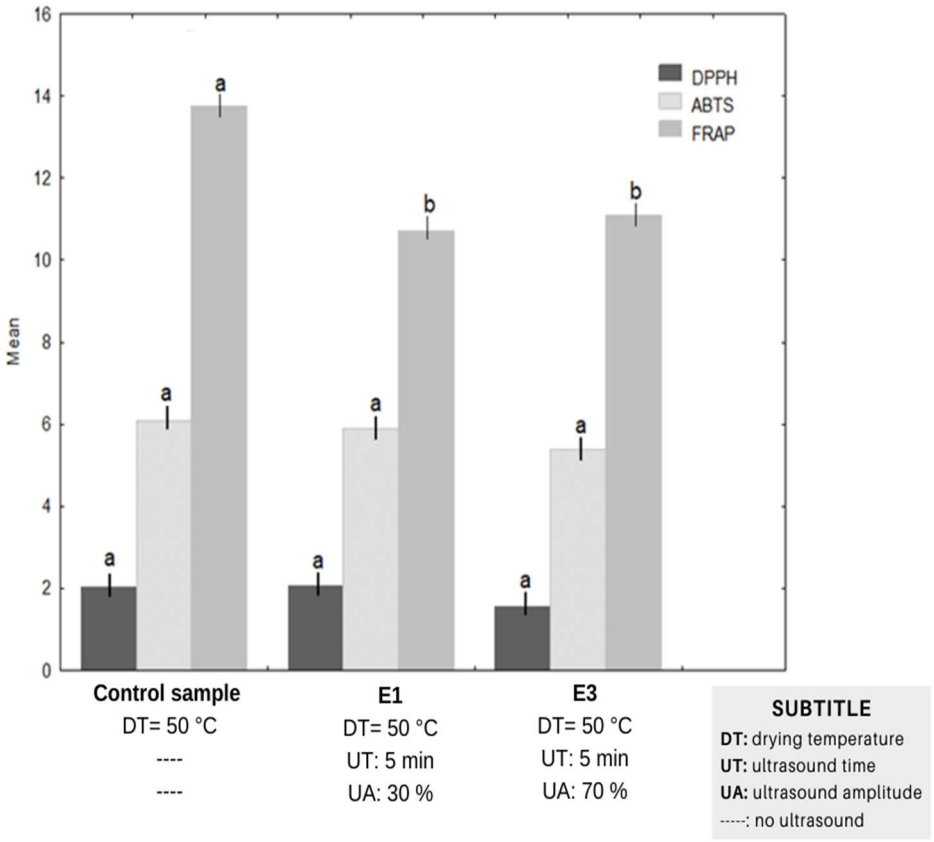


**Figure 3-** Bar graph of antioxidant activity by DPPH, ABTS$^{+}$, and FRAP methods of pumpkin seeds from Control, E1, and E3 experiments.

Note: Antioxidant activity measured with DPPH and ABTS+ expressed in mmol kg−1 equivalent to Trolox. The one by FRAP is expressed in mmol Fe2+ kg −1. Bars with the same letters do not differ from each other by Tukey's test at 5% error probability. E1 (drying temperature= 50°C; ultrasound time=5 min; ultrasound amplitude=30%); E3 (drying temperature= 50°C; ultrasound time=5 min; ultrasound amplitude=70%).

The antioxidant activity obtained from the DPPH method ranged from 1.57 to 2.07 Mmol TE $kg^{-1}$, but no significant difference was observed between the experiments. For the ABTS+ method, the experiments ranged from 5.40 to 6.09 Mmol TE $kg^{-1}$. A statistically significant difference was found only for the FRAP method, with the highest value for the control experiment (13.76 mmol $Fe^{2+}$ $kg^{-1}$). The low losses in the antioxidant compounds of the evaluated samples indicate that, although the ultrasound process does not effectively contribute to the retention of antioxidant compounds, it does not promote evident losses in the treatments applied.

According to Kahraman et al. (2021), the reduction of antioxidant activity in products that may have ultrasound-assisted waves is related to cellular damage caused by

mechanical stress from acoustic drying. However, some studies show that mild drying and ultrasound conditions can promote the retention of antioxidant compounds.

### 3.3 Individual Phenolic Compounds

The phenolic compound profiles of the control experiment (50°C), E1, and E3 pumpkin seed flours were quantified using High-Performance Liquid Chromatography (HPLC), and the results are presented in Table 4.

**Table 4** – Profile of phenolic compounds from the control experiments (50°C), E1 and E3.

| Components (mg $kg^{-1}$) | Pumpkin seed flours | | |
|---|---|---|---|
| | **Control (50°C)** | **E1** | **E3** |
| **FLAVANOLS** | | | |
| (+)-Catechin | 1.0831±0.02[a] | 0.283±0.01[c] | 1.044±0.09[b] |
| (-)-Epicatechin gallate | ND | 0.3271±0.01[a] | 0.2935±0.02[a] |
| (-)-Epigallocatechin gallate | 0.173±0.02[a] | ND | 0.0941±0.04[b] |
| Procyanidin A2 | ND | 0.4261±0.01[a] | 0.40±0.12[b] |
| Procyanidin B1 | 0.0951±0.02[a] | 0.0975±0.01[a] | 0.1009±0.03[a] |
| Procyanidin B2 | 8.3169±0.18[a] | 8.478±0.08[a] | 6.5856±0.03[b] |
| **FLAVONOLS** | | | |
| Myricetin | 0.4323±0.01[a] | 0.3739±0.02[b] | 0.2310±0.02[c] |
| Rutin | 0.0233±0.03[b] | 0.0263±0.02[a] | 0.038±0.01[c] |
| Quercetin 3-Glucoside | 0.1218±0.03[c] | 0.1307±0.01[a] | 0.1231±0.02[b] |
| Kaempferol 3-glucoside | 0.1601±0.02[a] | 0.1166±0.011[b] | 0.079±0.03[c] |
| **STILBENES** | | | |
| Cis-Resveratrol | 0.3813±0.05[a] | ND | ND |
| **FLAVANONES** | | | |
| Hesperidin | 1.1638±0.07[b] | 1.1905±0.12[a] | 0.6365±0.11[c] |
| **PHENOLIC ACIDS** | | | |
| Caftaric acid | 1.4097±0.11[a] | 0.7731±0.07[c] | 1.0509±0.08[b] |
| Caffeic acid | 0.1318±0.12[a] | ND | ND |

| | | | |
|---|---|---|---|
| Chlorogenic acid | 0.3231±0.01[a] | 0.1258±0.12[b] | 0.1235±0.04[b] |
| Gallic acid | 10.0356±0.09[b] | 12.22±0.08[a] | 8.6298±0.03[c] |
| Syringic acid | 0.1002±0.02[a] | 0.0689±0.03[b] | ND |

Note: Means followed by the same lowercase letter in the columns do not differ statistically by Tukey's test at 5% probability. Results are expressed as mean ± standard deviation (n=3). Control (No ultrasound; drying temperature= 50°C) E1 (drying temperature= 50°C; ultrasound time=5 min; ultrasound amplitude=30%); E3 (drying temperature= 50°C; ultrasound time=5 min; ultrasound amplitude=70%).

17 phenolics in which flavons are found were identified: 4 Flavon, flour, 1 Stilbene, and 5 phenolics acids. These results indicate that pumpkin seeds are a good source of phenolic compounds. Hussain et al. (2021), when comparing the different pumpkin fractions (pulp, peel, and seeds), verified that the highest amount of total phenolic contents was found in the pumpkin seed extract (224.61 mg GAE/100g powder) and that the lowest amount of phenolic content was found in pumpkin peel extract (93.40 mg GAE/100g powder). From them, the results are more significant than the pumpkin results, as they are a good source of phenolic compounds and have greater potential.

Flavonoids are divided into flavonols and flavanones. Regarding flavonols, it was possible to identify the presence of catechin, epicatechin, epigallocatechin gallate, procyanidin B1, procyanidin B2, and procyanidin A. Procyanidin A was the most abundant flavonol in the samples, ranging from 8.47 to 6.58 mg/kg. The highest value for this compound was observed in experiment 1 (submitted to the ultrasound process for 5 min and 30% amplitude), and the lowest value was observed in experiment 3 (submitted to the ultrasound process for 5 min and 70% amplitude). Procyanidins are commonly found in products of plant origin, they are known as condensed tannins with a high antibacterial and antioxidant capacity, in addition to having properties that favor the prevention of diseases such as cancer and cardiovascular diseases (LIU et al., 2018; CONG et al., 2020).

Non-flavonoids are divided into phenolic acids and stilbenes. Among the phenolic acids present (Caftaric, caffeic, chlorogenic, gallic, and syringic) in the evaluated samples, gallic acid was predominant compared to the other phenolic acids identified, presenting values that ranged from 8.62 to 12.22 mg/kg. A higher value for this acid was obtained in experiment 1, which used the lowest ultrasound frequency.

Regarding stilbenes, only the presence of cis-resveratrol was observed. However, this compound was detected only in the control sample, with a magnitude of 0.3813 mg/kg. Therefore, it can be inferred that the experiments submitted to the ultrasound process suffered

damages concerning this component. According to Santos et al. (2021), cis and trans-resveratrol stilbenes have been widely studied for their benefits to consumer health, as they are bioactive compounds of high nutritional interest.

The nutraceutical role of phenolic compounds is related to the ability of these substances to neutralize radicals and reduce oxidants in the body, reducing the possibility of developing degenerative diseases in humans (FERREIRA et al., 2021). By analyzing all the phenolic compounds detected in the samples, it can be seen that the relationship behaves differently with an exposure to the ultrasound process, in some, it occurs greater with the application of ultrasound pre-treatment. In contrast, all compounds are degraded with exposure to the process. Similarly, the same occurs concerning the amplitude applied in the process.

The different responses of phenolic compounds and flavonoids to treatments may be due to differences in the reactivity of phenolic compounds. Furthermore, differences in the chemical structures of phenolic compounds likely cause variations in their binding state. Therefore, the methods applied to promote the extraction of phenolic compounds in the same food matrix may not be the same.

The increase in the number of phenolic compounds during some processes occurs due to changes in the structures of the molecules associated with the phenolic compounds. On the other hand, the reductions in the levels of some phenolic compounds may have been caused by the heat-induced polymerization of phenolic compounds, increasing their molecular weight and making them insoluble (Ahmed et al., 2020).

Despite the experiments being submitted to the same drying air temperature, they were also submitted to different ultrasound conditions: without ultrasound (control), experiment 1 (30% amplitude), and experiment 3 (50% amplitude). The effect of the propagation of ultrasonic waves with high amplitude causes more significant agitation of the medium and, consequently, an increase in the temperature of the medium through which the propagation occurs, changing the profile of phenolic compounds.

### 3.4 Fatty acids composition

The fatty acids identified in the pumpkin seed flours (control experiment (50°C), E1, and E3) are shown in Table 5.

**Table 5** – Fatty Acids (%) composition from the control experiments (50°C), E1 and E3.

| Compounds | Pumpkin seed flours | | |
|---|---|---|---|
| | **Control (50°C)** | **E1** | **E3** |
| Phthalic acid | 0.42 | 0.67 | 0.52 |
| Palmitic acid | 14.54 | 15.87 | 16.20 |
| Linoleic acid | 28.70 | 26.69 | 26.00 |
| Oleic acid | 40.36 | 37.24 | 37.50 |
| Stearic acid | - | 16.71 | 16.61 |
| Nonadecanoic acid | 14.61 | 1.48 | - |
| Tributyl acetyl citrate | - | 0.17 | - |
| Methyl ricinoleate | - | 0.27 | 0.21 |
| cis-13-Eicosenoic acid, methyl ester | 0.27 | 0.29 | 0.29 |
| cis-11-Eicosenoic acid, methyl ester | 0.18 | - | |
| Methyl arachidonate | - | - | 1.28 |
| Dodecane, 1,12-di(2-nitro-3-ethoxyphenoxy)- | - | - | 0.15 |
| 2-Ethylbutyric acid | - | 0.15 | - |
| Hexadecanoic acid, 2-hydroxy-1-(hydroxymethyl) | - | 0.15 | - |
| Hexanedioic acid, bis(2-Ethylhexyl) ester | - | - | 0.42 |
| Nonadecanoic acid | 1.04 | - | - |
| Oxalic acid | - | - | 0.56 |
| Behenic acid | 0.18 | 0.31 | 0.25 |
| % Total identified | 100.00 | 100.00 | 100.00 |

Note: Control (No ultrasound; drying temperature= 50°C) E1 (drying temperature= 50°C; ultrasound time=5 min; ultrasound amplitude=30%); E3 (drying temperature= 50°C; ultrasound time=5 min; ultrasound amplitude=70%).

From Table 5, it was possible to analyze the fatty acid composition of pumpkin seed flours. The main fatty acids identified were linoleic, oleic, stearic, palmitic, and nonadecanoic, representing 98.21% of the total identified fatty acids in the control experiment, 97.99% in E1, and 96.31% in E3.

The total fatty acids identified in pumpkin seed flours show that unsaturated fatty acids are the ones that make up the vast majority of these samples. Oleic acid was the main fatty acid found in all flour samples, followed by linoleic acid. Oleic acid (omega 9) showed higher values in the control experiment (40.36%) and lower values in the E1 experiment (37.24%), indicating that the ultrasound process may slightly interfere with the oleic acid content of the samples and the total fatty acid composition. Linoleic acid (omega 6) showed higher values in the control experiment (28.70%) and lower values in the E3 experiment (26%).

Unsaturated fatty acids have been extensively studied because they protect against cardiovascular disease. They are essential for the healthy growth and development of the brain and nervous system; they are also reported to have health benefits in improving coronary heart disease, hypertension, and arthritis, and fighting inflammation, autoimmunity-related disorders, and cancer. Furthermore, only two fatty acids are essential for humans, linoleic and linolenic, these acids cannot be synthesized in the human body and must therefore be supplied through the diet (Dotto et al., 2020).

According to Kaseke et al. (2020), processes involved in seed oil production, including seed drying processes, seed pre-treatment, and oil products, have originated concerning a change in the composition and functioning properties of the oils. The ultrasound process as a pre-treatment of the drying process did not promote many changes in the composition of unsaturated fatty acids in the evaluated samples. However, Sanwal et al. (2022) suggest that ultrasound-assisted oil extraction improves the extraction of all fatty acids in linseed oil, chia seeds, and papaya seeds. Rezig et al. (2019) studied pumpkin (*Cucurbita pepo*), watermelon (*Citrullus lanatus*), and melon (*Cucumis melo*) seeds as new alternatives for oil production and found that these raw materials have high values of unsaturated fatty acids, mainly with oleic and linoleic acid.

Figure 4, is expressed as scores plotted in PC1 and PC2. PCA was applied to the resulting matrix to verify the influence of ultrasound amplitude on the fatty acid (FA) and phenolic (P) profiles.

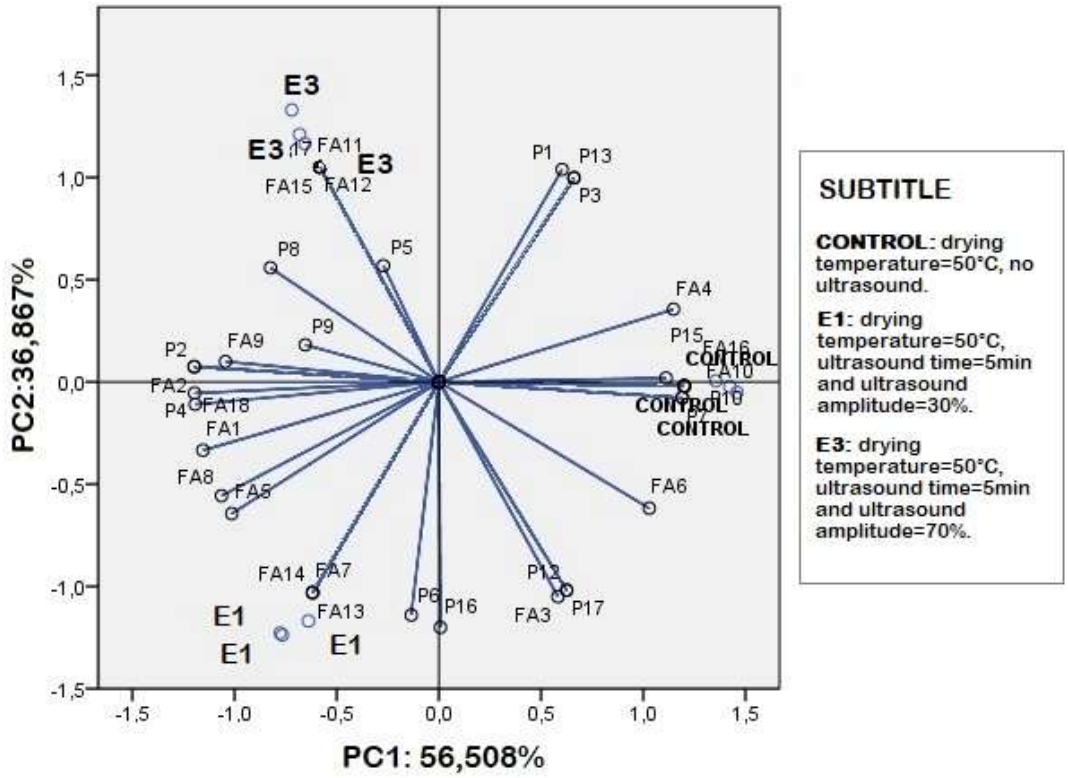


**Figure 4-** Analysis of the main biplot components of Fatty Acids and Phenolics phenolic compounds profile of pumpkin seeds from Control, E1, and E3 experiments.

Note: Control (No ultrasound; drying temperature= 50°C) E1 (drying temperature= 50°C; ultrasound time=5 min; ultrasound amplitude=30%); E3 (drying temperature= 50°C; ultrasound time=5 min; ultrasound amplitude=70%); FA1= Phthalic acid; FA2= Palmitic acid; FA3= Linoleic acid; FA4= Oleic acid; FA5= Stearic acid; FA6= Nonadecanoic acid; FA7= Tributyl acetyl citrate; FA8= Methyl ricinoleate; FA9= cis-13-Eicosenoic acid, methyl ester; FA10= cis-11-Eicosenoic acid, methyl ester; FA11= Methyl arachidonate; FA12= Dodecane, 1,12-di(2-nitro-3-ethoxyphenoxy)-; FA13=2-Ethylbutyric acid; FA14= Hexadecanoic acid, 2-hydroxy-1-(hydroxymethyl); FA15= Hexanedioic acid, bis(2-Ethylhexyl) ester; FA16= Nonadecanoic acid; FA17= Oxalic acid; FA18= Behenic acid; P1= Catechin; P2= Epicatechin gallate; P3= Epigallocatechin gallate; P4=Procyanidin A2; P5= Procyanidin B1; P6= Procyanidin B2; P7= Myricetin; P8= Rutin; P9= Quercetin 3-Glucoside; P10= Kaempferol 3-glucoside; P11= Cis-Resveratrol; P12= Hesperidin; P13= Caftaric acid; P14= Caffeic acid; P15= Chlorogenic acid; P16= Gallic acid; P17= Syringic acid.

Which represents 93.375% of the total variance, indicating a good representation of the behavior of the samples. The PC1 axis represented 56.508% of the total variance and was responsible for separating the control samples from the E1 and E3 samples since the samples

that were submitted to ultrasound pre-treatment (E1 and E3) were grouped on the left side of the axis and the sample control clustered on the right side of the axis. Through this observation, it is possible to verify the influence of the pre-treatment with ultrasound on the profile of fatty acids and phenolic compounds of the samples.

The ultrasound process influenced the increase in the concentration of some phenolic compounds (Procyanidin B1, Rutin, Quercetin, Epicatechin gallate, Procyanidin A2 and Procyanidin) and of some fatty acids (Methyl arachidonate, Dodecane, Hexanedioic acid, bis(2-Ethylhexyl) ester, Oxalic acid, Behenic acid, cis-13-Eicosenoic acid, methyl ester, Palmitic acid, Linoleic acid, Oleic acid, Stearic acid, Nonadecanoic acid, Tributyl, Palmitic acid, Tributyl acetyl citrate, Methyl ricinoleate, 2-Ethylbutyric acid, Hexadecanoic acid, 2-hydroxy-1-(hydroxymethyl))

Although, the ultrasound process caused the reduction of the following phenolic compounds: Catechin, Epigallocatechin gallate, Caftaric acid, Chlorogenic acid, Syringic acid, Hesperidin, Kaempferol 3-glucoside, Myricetin and Nonadecanoic acid, and the following fatty acids: Nonadecanoic acid, cis -11-Eicosenoic acid, methyl ester; Oleic acid, Nonadecanoic acid, and Linoleic acid.

The PC2 axis represented 36.867% of the total variance of the samples and was responsible for separating the experiments pre-treated with ultrasound. Higher values of PC2 were observed in experiment E3, the control sample assumed an intermediate value of PC1, and lower values were observed in experiment E1. It was also possible to verify that the ultrasound amplitude directly influenced the profile of phenolic compounds and fatty acids in the samples. A tendency was observed to increase the concentration of Phthalic acid, Methyl ricinoleate, Stearic acid, Hexadecanoic acid, 2-hydroxy-1-(hydroxymethyl), Tributyl acetyl citrate, 2-Ethylbutyric acid, Linoleic acid, Nonadecanoic acid, Procyanidin B2, Gallic acid, Hesperidin, Syringic acid in samples subjected to low amplitude ultrasound (30%).

According to Li et al. (2019), hesperidin has a molecular structure of hesperetin glycosides, and the thermal degradation process starts from the deglycosylation process into flavonoids. Additionally, the authors verified through multivariate analysis that chlorogenic acid, the main polyphenol in loquat, degraded into caffeic acid and protocatechuic acid and isomerized into cryptochlorogenic acid during vacuum drying. This fact can be explained, since higher ultrasound amplitudes release more heat in the samples, favoring the thermal degradation of more sensitive compounds.

Meanwhile, it was observed that samples submitted to high amplitude ultrasound (70%) promoted higher concentrations of Catechin, Epigallocatechin gallate, Caftaric acid,

Chlorogenic acid, Procyanidin B1, Rutin, Methyl arachidonate, Dodecane, 1,12-di( 2-nitro-3-ethoxyphenoxy), Oxalic acid, Hexanedioic acid, bis(2-Ethylhexyl) ester. The control sample favored higher concentrations of cis-13-Eicosenoic acid, methyl ester, Palmitic acid, Procyanidin A2, Nonadecanoic acid, cis-11-Eicosenoic acid, methyl ester, Myricetin, Kaempferol 3-glucoside, Epicatechin gallate.

According to Li et al. (2020), the ultrasound fields cause shear stress in the molecules, generating a thermal effect and the formation of free radicals. Furthermore, the authors observed that these effects are amplified when there is an increase in the ultrasound amplitude. In general, total phenolic contribute to the antioxidant activity that acts in the breaking of chains, free radical scavengers, and electron donors (Kainama et al., 2020). Given this, we can infer that the changes observed in the profile of phenolic compounds were caused by the ultrasonic waves that allowed the formation of free radicals. Despite this, as seen before, there were no changes concerning the antioxidant capacity of the samples, evidencing the presence of other antioxidant compounds in addition to the phenolic compounds present. It was also verified that the phenolic compounds behaved in different ways in face of the variables applied in the processes.

More information is needed to interpret changes in the fatty acid composition and phenolic profile of pumpkin seeds. However, these findings suggest that convective drying is a promising drying technology to preserve and that the ultrasound process can promote the reduction of drying time and maintenance of phenolic compounds and fatty acids. However, the ultrasound amplitude is thoroughly chosen aiming at the main compounds of interest in a product.

## CONCLUSIONS

The results of this study explained that the convective drying of pumpkin seeds using ultrasound as a pre-treatment is a good alternative to produce flour and promote the use of this industrial by-product. The optimized condition was established when the seeds were kept at 15 min of ultrasound at an amplitude of 70% and consequently dried at 70 °C (Experiment E8), which showed the lowest water. E1 had the highest levels of vitamin C and lipids, however, higher acidity and lower pH values were obtained in the control sample, dried at 70 °C. Regarding the change in seed color after drying, the ultrasound process failed to generate high results in all treatments.

The antioxidant potential was not affected by the application of the ultrasound process ($p < 0.05$). However, alterations in the phenolic profile were observed, due to the

formation of free radicals by ultrasound waves. It was also found that most of the fatty foods found in the samples are unsaturated and that oleic and linoleic foods are predominant in the samples. When comparing the sample pre-treated with ultrasound and the control sample, it was observed that the control sample had higher values of fat recipes, indicating that the ultrasound process promoted the ingestion of some preparations.

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## *APPENDIX D - PAPER 4- (SUBMITTED TO INTERNATIONAL JOURNAL OF BIOLOGICAL MACROMOLECULES )*

## *SPRAY-DRY PUMPKIN SEED OIL WITH DIFFERENT ENCAPSULATING AGENTS TO OBTAIN ANTIOXIDANT-RICH DRY PARTICLES*

**Abstract:**

This study examines the efficacy of different encapsulating agents in the protection and release of pumpkin seed oil, focusing on antioxidant capacity, total phenolics, and instrumental color properties of the microcapsules. Three encapsulating agents were tested: modified maltodextrin (Capsul - C), conventional maltodextrin (M), and a combination of both (CM). The objective was to evaluate the performance of these agents in the encapsulation and release of the oil under simulated gastrointestinal tract conditions. Principal Component Analysis (PCA) was employed to normalize and scale the data, facilitating the understanding of correlations among the studied parameters. An in vitro gastrointestinal tract model was developed to simulate the oral, gastric, and intestinal phases, allowing for a detailed assessment of the release behavior of the microcapsules. The results showed significant differences in antioxidant capacity and oil release among the samples encapsulated with different agents, highlighting the influence of the type of encapsulant on the protection and release of the oil. Statistical analysis indicated that the lowest release rates occurred in the salivary fluid phase, suggesting that the composition of the microcapsule wall offers effective protection against early degradation. This study contributes to food engineering and encapsulation technology, providing insights into the performance of encapsulating agents in protecting bioactive compounds. The findings have practical implications for the development of functional food products, where controlled release of active ingredients is desirable, and pave the way for future investigations into the optimization of encapsulation techniques for the effective delivery of nutrients and bioactive compounds in the gastrointestinal tract.

**Keywords:** Bioactive Compounds, Controlled Release, Microencapsulation, In Vitro Simulation, Food Engineering.

## 1. Introduction

Microencapsulation is a versatile and promising technology widely applied in various sectors, such as biomedical engineering, agriculture, smart coatings, the textile industry, and cosmetics (Lu et al., 2021). In the food industry, this technique plays a crucial role in the development of functional products, fat reduction, sensory quality improvement, food preservation, and inhibition of microbial growth (Calderón-Oliver & Ponce-Alquicira, 2022; Choudhury et al., 2021).

One of the most widely used microencapsulation technologies in the food industry is spray drying. It is an efficient thermal process that converts liquid foods into powder (Dantas et al., 2024; Yan et al., 2022). This versatile technique is commonly employed to produce powdered milk, instant coffee, powdered juices, and flavor enhancers, among other products. The transformation of these liquid foods into shelf-stable powders offers practical benefits like easier storage, transportation, and extended shelf life. In this method, the liquid is atomized into small droplets that come into contact with a preheated drying gas, resulting in the rapid evaporation of the present water and the formation of high-quality dry solid particles (Dantas et al., 2024; Homayoonfal et al., 2022).

The primary aim of spray drying encapsulation is to maximize the nutritional, sensory, and economic quality of food products (Homayoonfal et al., 2022) and protect active ingredients such as flavors, nutrients, and colorants (Yan et al., 2022). This technique can enhance the morphology and texture of dry food, preserve natural components for extended periods, and increase storage times in the food industries (Fathi et al., 2022).

Besides being convenient and energy-efficient, spray drying allows the production of dry powders from aqueous solid lipid microparticles (Wolska et al., 2021) and the transformation of bioactive compounds from food waste and by-products into stable powders, which are easily storable and incorporable into other products (Banožić et al., 2021). This not only enhances the value of food waste but also aligns with the growing trend towards sustainability in the food industry. By utilizing food waste and by-products, spray drying contributes to waste reduction and promotes the efficient use of resources, thereby supporting environmental conservation and economic efficiency. This approach not only minimizes the environmental impact but also creates new opportunities for developing innovative and sustainable food products

Pumpkin seed oil has garnered interest due to its health benefits, such as antioxidant

and antimicrobial activities, and its richness in bioactive compounds (Šamec et al., 2022; Hagos et al., 2023; Kang et al., 2021; Sumara et al., 2022). However, its application in the food industry is still limited, possibly due to the need for more studies on its chemical composition (Kalyna et al., 2022) and techniques that promote the stability of its components during processing. Previous studies have shown the development of an approach that combines drying and ultrasound pretreatment to extract pumpkin seed oil (Barros et al., 2023). Initially, we identified optimal conditions to maximize extraction yield and its antioxidant potential. However, like other vegetable oils, pumpkin seed oil presents stability limitations, hindering its application and storage.

Rich in unsaturated fatty acids, pumpkin seed oil is susceptible to lipid oxidation, which can lead to the deterioration of its nutritional and sensory properties. Additionally, the presence of bioactive compounds, such as natural antioxidants, can be compromised during processing and storage, reducing the health benefits associated with this oil. These limitations pose a challenge for the food industry, restricting the application of pumpkin seed oil in food products and complicating its conservation during storage.

To overcome these limiting factors, this study proposes spray drying as a promising strategy for microencapsulating pumpkin seed oil. Spray-drying microencapsulation has proven effective in protecting vegetable oils against oxidation, improving their stability, and prolonging their shelf life (Pattnaik et al., 2021; Yakdhane et al., 2021). Encapsulating pumpkin seed oil in a protective matrix is expected to minimize the degradation of its bioactive components and unsaturated fatty acids, preserving its nutritional and sensory properties. Thus, spray drying microencapsulation can enable the application of pumpkin seed oil in a wide range of food products and facilitate its storage and distribution.

Maltodextrin is one of the most used wall materials in microencapsulation, acting as a protective barrier for the active principle, controlling its release, and retaining the properties of the encapsulated oils for a long time (Freitas et al., 2021; Yeddes et al., 2022). Additionally, maltodextrin improves encapsulated essential oils' thermal and oxidative stability, protecting volatilization and environmental conditions (Martins et al., 2021). However, the porosity of maltodextrin and the hollow structure of the particles that contribute to oxygen contact can lead to the oxidative degradation of fatty acids and antioxidants in oil-in-water emulsions (Sánchez et al., 2016).

The addition of Octenyl Succinic Anhydride modified (OSA) maltodextrin

significantly maximizes the stability of microcapsules, reducing the oxidation of essential oils by approximately 80% compared to the use of conventional maltodextrin, as demonstrated by Sotelo-Bautista et al. (2020). Moreover, the study by Xiao et al. (2019) reveals that OSA-modified starch and maltodextrins positively influence the release dynamics of essential oils. Components such as esters, alcohols, and phenols are released more effectively, with this process being directly influenced by factors such as temperature and relative humidity.

Despite advances in microencapsulation technology, challenges remain to be overcome, such as increasing the entrapment efficiency of hydrophilic and semi-polar active compounds and selecting appropriate manufacturing methods (Subroto et al., 2023). However, microencapsulation remains an effective method for developing functional foods, biopharmaceuticals, and sustainable products, contributing to waste reduction and the utilization of by-products (Hussain, 2023; Rana et al., 2021).

Given the above, it becomes evident that further studies are needed on the microencapsulation of pumpkin seed oil, exploring its potentialities and challenges to promote an alternative use of this raw material. This technology can contribute to the development of innovative functional foods with beneficial health properties and greater stability, meeting consumer demands for quality products.

## 2. Materials and Methods

### 2.1 Materials

Pumpkin seeds (*Cucurbita maxima*) were provided by ISM Alimentos, located in Fortaleza-CE, Brazil (in the year 2023). For the encapsulation process, the following materials were used: Capsul® (CA), a modified maltodextrin supplied by GastronomyLab®, Distrito Federal, Brazil, and Amisil® (AM), an unmodified maltodextrin provided by Ingredion, São Paulo, Brazil. A mixture of CA and AM was prepared in a 50:50 (w/w) ratio, referred to as CAM. All chemicals and reagents used in the study were of analytical grade with a purity of >99%.

### 2.2 Extraction of Pumpkin Seed Oil

The extraction process of pumpkin seed oil followed the experimental protocol proposed by Barros et al. (2023). For this, the pumpkin seeds were dried at 50 °C in an oven with air circulation at a speed of 580 °C for 480 minutes. After drying, the seeds were cold-

pressed using a mechanical pressing method (model, brand, state, and country) at room temperature (25±2 °C). The extracted oil was centrifuged (model, brand, state, and country) at 10.000 rpm for 30 minutes and stored in light-protected glass containers at -20 °C until further steps were carried out.

### 2.3 Preparation of Extracts and Emulsions

Three emulsion formulations were prepared using pumpkin seed oil and different encapsulating agents either in isolation (C, M) or combined (CM) at a concentration of 15% (concentrations defined based on preliminary tests, data not shown). The pumpkin seed oil was slowly added and diluted at a ratio of 1:10 in 50% ethanol, along with 15% of the encapsulating agent (C, M, or CM), forming three extracts. To obtain the emulsion, a high-speed homogenizer (Ultra-Turrax IKA T18, USA) was used at 22,000 rpm for 4 minutes, followed by 25.000 rpm for 2 minutes.

### 2.4 Spray Drying Process

The spray drying process of the prepared emulsions was carried out using a Spray Dryertype dryer, model MSDi 1.0, manufactured by Labmaq (Brazil). The drying conditions were maintained based on previous studies conducted by Nogueira et al., 2024, which were as follows: drying air temperature of 140 °C, air flow rate of 25 m/s, average volumetric flow rate of 200 mL/h (corresponding to 20% of the equipment's capacity), 1.0 mm spray nozzle, and drying air flow rate of 35 L/min. After the drying process, three experimental groups were obtained: C (Pumpkin seed oil particles with the addition of Capsul®), M (Pumpkin seed oil particles with the addition of Amisil®), and CM (Pumpkin seed oil particles with the addition of Capsul® and Amisil®). The obtained dry particles were collected, weighed, and then sealed in dark plastic bags and stored (for no more than 1 month) at -20 °C until further use.

### 2.5 Characterization of Extracts and Pumpkin Seed Oil Powder

#### 2.5.1 Antioxidant Capacity and Total Phenolics

The antioxidant capacity of the extracts and pumpkin seed oil powder was determined using free radical scavenging methods, employing DPPH (Kim et al., 2002) and iron chelation power (FRAP) (Rufino et al., 2006). For the construction of the analytical curve,

the Trolox analytical standard was used, with the results expressed in Trolox equivalents per kg of pumpkin seed oil powder and extract (mmol TE kg−1) for the DPPH method, and in mmol of Fe2+ per kg of pumpkin seed oil powder for the FRAP method. The Folin- Ciocalteu reducing capacity was determined according to the methodology proposed by Singleton and Rossi (1965). Absorbance readings were performed on a UV-visible spectrophotometer, model UV 2000A (Instrutherm, Brazil). The activity of the DPPH radical (1,1-diphenyl-2-picrylhydrazyl) was measured by suppressing the maximum absorption at 517 nm. The method consisted of mixing 100 µL of the sample with 2.90 mL of 100 mmol ethanolic solution of the DPPH radical, followed by incubation in the dark for 30 minutes. The FRAP reagent was prepared by mixing 25 mL of acetate buffer solution (300 mmol; pH 3.6), 2.5 mL of TPTZ solution (10 mmol TPTZ in 40 mmol HCl), and 2.5 mL of FeCl3 (20 mmol) in aqueous solution. Subsequently, 90 µL of the previously diluted powders/extracts and 270 µL of water were added to 2.7 mL of the FRAP reagent (maintained at 37 °C for 30 minutes in a thermoreactor model IT2002 AAKER, Brazil). Absorbance was measured at 595 nm in the spectrophotometer. The Folin-Ciocalteu reducing capacity was determined using 0.10 mL of the sample, 7.90 mL of water, and 0.50 mL of the Folin-Ciocalteu reagent. Absorbance at 765 nm was read, and the results were expressed in mg kg−1 of pumpkin seed oil powder or mg/L of extract, compared to a gallic acid calibration curve of 25-500 mg/L (GAE).

### 2.5.2 Color

Instrumental color was determined using the ColorQuest-XE colorimeter (HunterLab, Virginia, USA) according to the average of the readings of the CIE lab coordinates, being L*, a*, b*, Chroma (c*) (Equation 1), Browning Index (BI) (Equation 2), and Hue Angle (h*).

$$\mathbf{C^*} = \sqrt{\boldsymbol{a^2 + b^2}} \quad \textbf{[1]}$$

$$BI = [100(x - 0{,}31)]/0{,}17 \quad [2]$$

$$\text{onde: } x = (a + 1{,}75L)/(5{,}645L + a - 3{,}012b) \quad [3]$$

### 2.6 Microencapsulation Efficiency and Yield

To determine the microencapsulation efficiency (ME), 1.5 g of the powder was added to 15 mL of hexane and adequately mixed to extract free oil at 25 °C for 2 minutes. After filtration, the powder retained on the filter was collected and washed with 20 mL of hexane. This process was repeated three times. The extracted oil samples were combined and subjected to solvent evaporation at 60 °C. The resulting oil was weighed, and the surface oil (WSO) was determined by weight difference, as previously described (Premi and Sharma, 2017; Frascareli et al., 2012). The total oil (WTO) was extracted using the Soxhlet method (AOCS, 1989), and its weight was measured by differences. The microencapsulation efficiency was calculated using the following equation:

$$\mathbf{ME\ (\%) = [(WTO - WSO) / WTO] \times 100} \quad \mathbf{[4]}$$

Where: WTO is the weight of the total oil (g), and WSO is the weight of the surface oil (g).

The process yield was calculated by the collected powder's mass ratio to the input feed's mass (Oliveira et al., 2021).

### 2.7 Characterization of the Obtained Particles

#### 2.7.1 Zeta Potential and Particle Size

The zeta potential of the powder particles was determined using the Zeta Sizer Nano ZS90 (Malvern Instruments, UK). In this case, the systems were diluted 1/500 using ultrapure water filtered through 0.45 and 0.22 μm nitrocellulose filters at different pH levels (the same as the emulsion itself) and then placed in disposable polycarbonate cuvettes with gold-plated copper electrodes and a curved capillary tube. Measurements were reported as the mean and standard deviation of five determinations per sample.

Particle size was measured by dynamic light scattering using a Zeta Sizer Nano ZS90 (Malvern Instruments, UK) equipped with a He-Ne laser (633 nm). According to the equipment specifications, measurements were carried out at 25 °C and a fixed scattering angle of 90°, within the range of 0.6 nm to 6 μm. Samples were diluted 1/70 using ultrapure water filtered through 0.45 and 0.22 μm nitrocellulose filters (Merck Millipore, Ireland) and then placed in disposable polystyrene cuvettes with a 1 cm optical path. The refractive

index of both the dispersed phase (1.48) and the continuous phase (1.33) were used. Droplet size distributions were obtained as graphs of the relative intensity of light scattered by particles of different sizes and are reported as the mean of ten readings.

### 2.7.2 Water Activity and Moisture Content

The powder particles' water activity was measured using a water activity meter (Labmaster-aw, Novasina AG, Neuheimstrasse, Switzerland) at 25 °C. The moisture content was determined according to standard AOAC methods (925.09) (AOAC, 2005).

### 2.7.3 Bulk, Tapped, and True Density

The determination of bulk and tapped density followed the procedure proposed by Tonon (2009), consisting of weighing the sample inside a graduated cylinder to subsequently verify the volume it occupies. For obtaining the tapped density, the graduated cylinder was subjected to vibrations (50 times) against a rigid surface at a height of 5 cm, and the final volume occupied by the sample in the graduated cylinder was recorded, allowing the calculation of the tapped density. True density was determined using the pycnometric method, employing hexane at 25 °C. This process involves measuring the mass of the sample relative to its volume using a pycnometer calibrated with water at the specified temperature for the measurements (Buczek & Geldart, 1986).

### 2.7.4 Solubility, Hygroscopicity, and Wettability

Solubility was determined following the modified method of Eastman and Moore (1984) by Cano-Chauca et al. (2005). One gram (1g) of the sample was mixed with 100 mL of distilled water under maximum agitation in a magnetic stirrer for 5 minutes. The mixture was then centrifuged (Rotina 380R, Hettich, Oslo, Norway) at 2,600 rpm for 5 minutes, and a 25 mL aliquot of the supernatant was dried in an oven at 105 °C for 24 hours. Hygroscopicity was evaluated according to the adapted method of Cai and Corke (2000), where powder samples, weighing approximately 1 g each, were placed in a hermetic container containing a saturated sodium chloride (NaCl) solution, which provides a relative humidity of 75.29% at a temperature of 25 °C. The samples remained under these conditions for a period of seven days. After this interval, the powders were weighed and dried in an oven at 105 °C for 24 hours. At the end of the drying

process, the samples were weighed again. The wettability of all samples was evaluated based on the estimation of the time (min) required to immerse 1 g of powder particles on the surface of 400 mL of distilled water at 25 °C (Saifullah et al., 2016; Mahdi et al., 2020).

### 2.7.5 Porosity, Carr Index, Hausner Ratio, and Angle of Repose

The intergranular porosity of the samples was estimated using Equation 5, based on the difference between bulk density and true density. The Hausner Ratio (HR) (Hausner, 1967) and the Carr Index (CI) were calculated by the ratio between tapped density and bulk density according to Equations 6 and 7, respectively, following the methodology of Bhusari et al. (2014).

$$\epsilon = 1 - (\frac{\rho_{ap}}{\rho_{abs}}) \times 100 \quad [5]$$

$$HF = \left(\frac{\rho_C}{\rho_a}\right) \quad [6]$$

Where: HF represents the Hausner ratio (dimensionless); ρc denotes the tapped density (g/cm³); ρa signifies the bulk density (g/cm³).

$$CI = \left(\frac{\rho_{Cp} - \rho_{ap}}{\rho_{Cp}}\right) \times 100 \quad [7]$$

Where: ε represents the interparticle porosity (dimensionless), ρap denotes the bulk density (g/cm³), ρabs signifies the absolute density (g/cm³), ρcp indicates the tapped density (g/cm³), FH corresponds to the Hausner ratio, and IC represents the Carr index.

The angle of repose was determined from the geometry of a cone formed by a 30 g sample mass. After forming the pile on a flat surface, the diameter and height of the pile were measured, and the angle of repose was calculated according to Equation 8 (Mohsenin, 1986).

$$\theta = Arctang\left(\frac{2h}{D}\right) \quad [8]$$

Where: θ is the angle of repose, h is the height of the pile, and D is the diameter of the pile.

### 2.8 Statistical Analysis

All tests were performed in triplicate, and the results were evaluated by analysis of variance (ANOVA) and mean comparison by Tukey's test at a 5% probability level, using the software Past (Paleontological Statistics Software Package for Education and Data Analysis). Multivariate statistics were applied using principal component analysis (PCA) with data pre-treatment for normalization and scaling to verify the correlation between solubility parameters, zeta potential, release rate, true density, and particle size for the encapsulated oil samples using the PAST program (Hammer et al., 2021).

## 3. Results and Discussion

### 3.1 Antioxidant Capacity, Total Phenolics, and Color of the Prepared Extracts and Pumpkin Seed Oil Powder

The antioxidant capacity, total phenolics, and color parameters of the extracts prepared with the different encapsulating agents in isolation (C, M) and/or combined (CM) are presented in Figure 1. According to Nogueira et al. (2024), evaluating the extracts before drying is essential to ensure the quality and stability of bioactive compounds such as phenolics and antioxidants. This initial evaluation allows for determining the concentration of these compounds and understanding their behavior during drying, which can cause degradation due to heat and oxidation.

Knowing the composition of the extract before drying enables the optimization of process parameters, ensuring the preservation of functional properties. Comparing the results before and after drying validates the method's effectiveness and ensures the quality of the final product, which is crucial for industrial applications such as the encapsulation of extracts for food or supplements. Therefore, this step is fundamental to maintaining the integrity and

functionality of bioactive compounds during processing and storage.

Figure 1 shows the results obtained for the antioxidant activity of the extracts before drying and the microencapsulated powders at the end of the drying process.

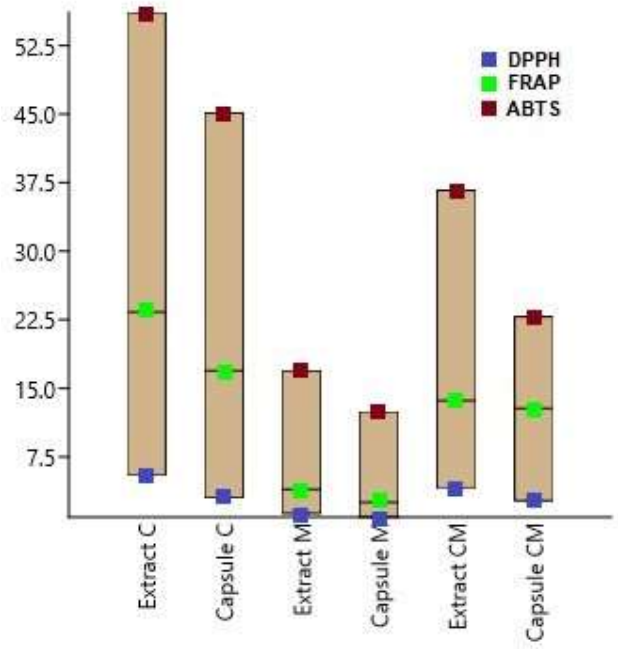



**Figure 1 -** Antioxidant Capacity of Extracts and Microcapsules by DPPH, FRAP, and ABTS Methods

Note: C = Capsul 15%; CM = Capsul 7.5% + Maltodextrin 7.5%; M = Maltodextrin 15%. AOX ABTS/DPPH − Antioxidant activity expressed as Trolox equivalent in mM kg−1 (mM TEAC kg−1). FRAP − Ferric reducing antioxidant power expressed in millimoles of Fe2+ (mM Fe2+ kg−1). FCRC − Folin–Ciocalteu reducing capacity expressed as mg kg−1 gallic acid equivalent.

Source: Author (2024).

According to Valková et al. (2022), the spray drying process reduces the antioxidant activity in β-glucan powder due to significant structural differences between the microencapsulated powders. Sun et al. (2020) associated these losses with the temperature used during the spray drying process, which causes a reduction in the retention efficiency of these compounds. Although a reduction in antioxidant activity is expected due to the drying process, Figure 1 shows that the encapsulating agents directly influence this parameter.

Among the experiments presented, the experiment containing Capsul showed superior antioxidant capacity values. This behavior can be explained by the fact that Capsul

(maltodextrin modified by OSA) promotes greater emulsion stability due to the formation of more soluble complexes, which induces a higher quantification of the antioxidant compounds available in the oil. A similar behavior was observed by Wu et al. (2020), who reported that modified maltodextrin (OSA) can form soluble complexes with whey protein isolate, potentially improving its antioxidant capacity.

This justification is based on the research conducted by Xu et al. (2020), who found that cinnamon essential oil emulsions stabilized with OSA-GA significantly increased chitosan-based polyelectrolyte films' antioxidant and antimicrobial activities. Additionally, studies developed by Zhang et al. (2021) observed that modifying starch with OSA can increase swelling power and water solubility, potentially improving emulsion stability.

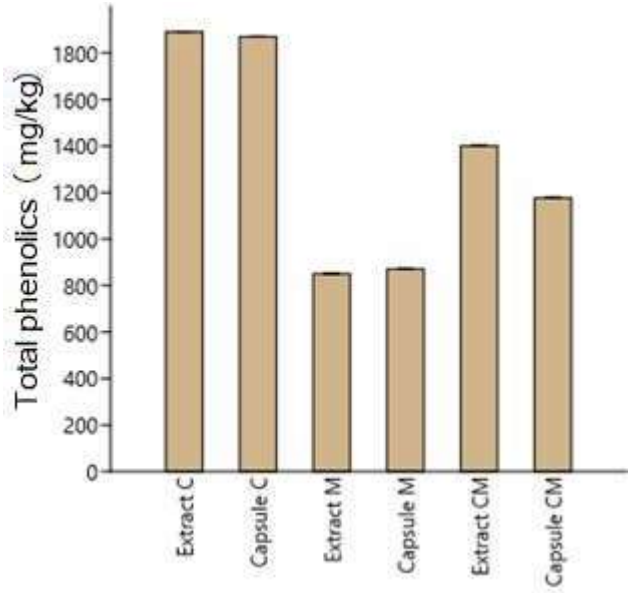



**Figure 2 -** Total phenolic content in extracts (C, M, and CM) and microcapsules (C, M, and CM).

The experiment utilizing only Capsul as the encapsulating agent exhibited superior values regarding total phenolic content (TPC), followed by the experiment employing a mixed encapsulating agent (CM). This finding indicates that the wall material significantly influences the preservation of TPC in the samples. Therefore, it can be concluded that Capsul effectively protects the phenolic compounds within the matrix during thermal processing, which inherently induces the degradation of these compounds due to exposure to elevated temperatures.

This observation aligns with previous research. Özbek and Ergönül (2020) demonstrated that optimizing the wall material composition with whey protein concentrate, maltodextrin, and gum arabic can effectively preserve the TPC in freeze-dried pumpkin seed oil. Similarly, Ferro et al. (2020) suggested that maltodextrin can be effectively employed to

preserve TPC in encapsulated pumpkin powder, achieving encapsulation efficiencies of 89% and 94%.

**Table 1 -** Drying Yield and Encapsulation Efficiency

| Parameter | Sample | | |
|---|---|---|---|
| | **C** | **M** | **CM** |
| Drying Yield (%) | 92.11 [a]±0.01 | 79.12 [b]±0.31 | 90.23 [a]±0.02 |
| Encapsulation Efficiency (%) | 90.64 [a]±0.01 | 78.71 [b]±0.31 | 89.81 [a]±0.02 |

Note: C =Capsul 15%; CM= Capsul 7,5% + Maltodextrina 7,5% e M= Maltodextrina 15%.

According to Table 1, it was found that the experiments containing Capsul (C and CM) showed higher yield and efficiency compared to the experiment containing only conventional maltodextrin, indicating that the modification of the encapsulating agent can promote higher yield and efficiency in the pumpkin seed oil encapsulation process. However, it is important to highlight that the experiment containing 50% modified maltodextrin did not show a statistically significant variation compared to the experiment containing only modified maltodextrin regarding these parameters. This indicates that using 50% modified maltodextrin is sufficient to increase the yield and efficiency of the spray drying process.

The encapsulation efficiency of the samples ranged from 78.71% to 90.64%, indicating good process efficiency as it approaches the maximum efficiency value of 100%. Cai et al. (2023) observed similar values for this parameter in the encapsulation of soybean oil using maltodextrin as the encapsulating agent (74.7% and 91.2%). Araújo et al. (2020) conducted a study on oil encapsulation using maltodextrin as the encapsulating agent and obtained remarkable results. The researchers demonstrated that the microencapsulation yield ranged from 75.75% to 90.19%, indicating a high capacity of maltodextrin to trap and protect the oil within the microcapsules.

The food industry produces large quantities of powders and involves unit operations such as storage, transportation, dosing, and mixing (Lanzerstorfer, 2020). Density values are important for the fundamental understanding of the matter and processes, the composition of these mixtures, and the associated unit operations, especially concerning packaging and storage (Doan-Nguyen and Crespy, 2022).

### 3.2 Zeta Potential and Particle Size

Table 2 expresses the magnitude of the values obtained for the parameters zeta potential and particle size of the pumpkin seed oil samples encapsulated with different formulations.

**Table 2** - Zeta Potential and Particle Size

| Sample | Zeta Potencial (mV) | Particule Size (µm) |
|---|---|---|
| C | $-39.10^{A} \pm 0.63$ | $2.66^{A} \pm 0.27$ |
| M | $-40.30^{A} \pm 0.71$ | $1.90^{B} \pm 0.06$ |
| CM | $-44.033^{B} \pm 0.51$ | $2.59^{A} \pm 0.09$ |

Note: C =Capsul 15%; CM= Capsul 7,5% + Maltodextrina 7,5% e M= Maltodextrina 15%.

The samples' zeta potentials varied from -44.033 to -39.10 mV, with the highest values recorded in the experiments designated as C and M. These, in turn, did not show statistically significant differences between them. Negative zeta potential values are usually obtained due to residual silanols and negatively charged phosphate groups (Dembek et al., 2022).

The zeta potential, a surface characteristic formed at the solid-liquid interface, is used to estimate nanomaterials' surface charge and stability (Dembek et al., 2022). Therefore, higher zeta potential values indicate greater stability of the samples. This property directly influences nanomaterials' tendency to form aggregates and interact with cell membranes (Varsou et al., 2020), affecting their biological activity (Sizochenko et al., 2021).

However, the sample designated as CM, encapsulated using a combination of two distinct types of maltodextrin, exhibited higher zeta potential values than the other samples. This phenomenon suggests that the interaction between the two materials significantly influences the zeta potential, as shown in Figure 4. This study's findings align with the results presented by Chang et al. (2020), who investigated the zeta potential of encapsulated fish oil and observed a variation in the magnitude of this parameter between -41.3 and -41.6 mV.

The behavior observed in the zeta potential of the samples can be justified by several factors. The encapsulation efficiency, oil loading rate, and the liposome's average

particle size directly influence the encapsulated oil's zeta potential (Shi et al., 2021). Additionally, the total solids (TS) and oil load (OL) in the emulsion also affect this property (Tambade et al., 2020). According to Aisyah et al. (2022), the type of oil and the combination of encapsulation materials, such as gelatin, gum arabic, whey protein isolate, and maltodextrin, play a significant role in the zeta potential of patchouli oil capsules.

The particle size ranged from 1.90 to 2.66 μm, with the lowest value presented in the sample containing only conventional maltodextrin. The samples containing modified maltodextrin (CM and C) did not show a statistically significant difference regarding this parameter. According to Zhao et al. (2019), capsules smaller than 30 μm are classified as microcapsules, exhibiting better tightness and faster-sustained release rates than larger capsules. The particle size in food powders is crucial to achieving low packing density, as it influences the interaction between shape, particle size, interparticle cohesion, and friction (Elmsahli and Sinka, 2020).

### 3.3 Physical Properties of Powder Particles

Table 3 contains the physical properties of pumpkin seed oil powder particles obtained through spray drying with different encapsulating agents. According to Rivero et al. (2020), evaluating physical parameters in food powders, such as density, hygroscopicity, and solubility, is essential to understanding how these variables influence product quality and meet specific production needs.

Additionally, analyzing the density of food powders allows for a better understanding of how these variable parameters influence the final product quality, providing more suitable options for different production demands (Pui and Lejaniya, 2021). Thus, evaluating these physical parameters is fundamental to ensuring the efficiency and quality of food powders and optimizing the involved industrial processes.

**Table 3 -** Physical properties of microencapsulated powders.

| Parameter | Sample | | |
|---|---|---|---|
| | **C** | **M** | **CM** |
| Water activity, $a_w$ | 0.34 $^{a}$ ±0.01 | 0.36 $^{a}$ ±0.01 | 0.28 $^{b}$ ±0.22 |
| Moisture content (% b.u.) | 5.60 $^{a}$ ±0.12 | 5.03 $^{ab}$ ±0.26 | 4.16 $^{b}$ ±0.02 |

| | | | |
|---|---|---|---|
| Bulk density (g/cm$^{-3}$) | 0.29$^{b}$±0.001 | 0.39$^{a}$±0.001 | 0.38 $^{a}$±0.001 |
| Tapped density (g/cm$^{-3}$) | 0.35 $^{b}$±0.001 | 0.48 $^{a}$±0.001 | 0.48 $^{a}$±0.002 |
| True density (g/cm$^{-3}$) | 0.26 $^{a}$±0.001 | 0.20 $^{b}$±0.001 | 0.18 $^{b}$±0.001 |
| Solubility (%) | 78.74$^{b}$±0.36 | 79.04 $^{b}$ ±0.09 | 80.41$^{a}$±0.1 |
| Hygroscopicity (g/100g) | 1.24$^{b}$±0.03 | 1.741$^{a}$±0.1 | 1.740$^{a}$±0.02 |
| Wettability (g/s) | 0.64 $^{b}$ ±0.09 | 0.68 $^{b}$ ±0.09 | 1.12 $^{a}$±0.1 |
| Porosity (%) | 1.11 $^{b}$±0.04 | 1.96 $^{a}$±0.17 | 2.07 $^{a}$±0.11 |
| Carr Index- IC (%) | 18.18$^{a}$±1.09 | 20.83$^{a}$±0.91 | 18.51$^{a}$±0.09 |
| Hausner Factor- HF | 1.22 $^{a}$±0.09 | 1.26 $^{a}$±0.06 | 1.22 $^{a}$±0.01 |
| Angle of repose (º) | 49.51$^{a}$±0.01 | 49.55 $^{a}$±0.001 | 48.94 $^{a}$±0.002 |
| L* | 86.69 $^{a}$ ±0.12 | 83.95 $^{b}$ ±0.02 | 86.8 $^{a}$ ±0.06 |
| a* | 0.26 $^{a}$ ±0.016 | -0.685 $^{b}$ ±0.004 | 0.215 $^{a}$ ±0.004 |
| b* | 12.19 $^{ab}$ ±0.01 | 11.05 $^{b}$ ±0.02 | 13.52 $^{a}$ ±0.004 |
| c* | 12.19$^{b}$±0.02 | 11.08$^{c}$±0.02 | 13.52$^{a}$±0.01 |
| Hue | 1.55 $^{a}$±0.01 | -1.51 $^{b}$±0.01 | 1.55 $^{a}$±0.01 |

Nota: C =Capsul 15%; CM= Capsul 7,5% + Maltodextrina 7,5% e M= Maltodextrina 15

As observed in Table 3, the samples showed water activity ranging from 0.28 to 0.36, with the lowest value observed in the CM sample. The moisture content of the samples varied from 4.16 to 5.60, with the highest value in the C sample. The C and M samples did not show a statistical difference between them. These results indicate that the samples have little water available for the development of microorganisms, thus resulting in greater microbiological stability. As mentioned by Alp and Bulantekin (2021), foods with reduced water activity and moisture content are less susceptible to microbial growth.

Bulk density is a simple yet important property for the evaluation of powders. However, it should not be assessed individually as its magnitude increases with the stress added by the consolidation of the samples (Lanzerstorfer, 2020). The true density of powders influences various flow metrics, such as aspect ratio and average particle size (Kiani et al., 2020).

The samples containing traditional maltodextrin (CM and M) did not show a statistically significant difference regarding the bulk, tapped, and true density parameters. It is also possible to observe that these samples presented higher bulk and tapped densities than those containing only capsul (modified maltodextrin). However, they showed lower

values concerning true density.

The bulk density of the powders ranged from 0.29 to 0.39 g/cm³, indicating that the powders are classified as medium density. According to Wessely et al. (2022), powders with bulk densities higher than 0.1 g/cm³ and lower than 0.3 g/cm³ are considered medium density. The powder's composition is one factor affecting the density and other relevant properties for the processing of binary mixtures, including powder flowability (Fathollahi et al., 2020). The findings obtained by Fournaise et al. (2020) indicated that the lipid content and pre-treatment of dairy concentrates affected the flowability and bulk density of the powder, respectively.

The water solubility of powdered materials determines their ability to dissolve and disperse in aqueous liquids, directly affecting their applicability in various products (Santos et al., 2024). The CM sample showed higher solubility values (80.41%) and wettability (1.12 g/s) than the C and M samples, indicating that the modified and traditional maltodextrin mixture favors solubility and wettability.

A correlation between the density of the samples and porosity was observed, as denser particles showed higher porosity (2.07%). This finding corroborates the studies conducted by Haferkamp et al. (2021), who found that porosity increases with an increased volumetric fraction of denser milk powder. It can also be inferred that the particles' shape, size, interparticle cohesion, and friction affect the compaction density in fine and cohesive powders (Elmsahli and Sinka, 2020).

The M and CM samples showed higher hygroscopicity values (1.96 and 2.07 g/100g) than the C sample, which showed hygroscopicity equivalent to 1.24 g/100g. Culina et al. (2023) found similar hygroscopicity values in encapsulated sea buckthorn berry oil, which ranged from 1.5 to 7.06 g/100 g.

According to Bhusari et al. (2014), the Hausner Factor (HF) and Carr Index (CI) are used to evaluate the cohesion and flow properties of powders. While HF is associated with friction between particles, CI indicates their aggregation capacity. HF ranged from

1.22 to 1.26, and CI ranged from 18.18 to 20.83, but no statistical difference was observed between the samples regarding these parameters, indicating that the samples have similar behavior concerning particle friction and aggregation.

The angle of repose, or angle β r, is used to characterize the flowability of granular materials and is important for handling materials and designing containers and

processing equipment (Elekes and Parteli, 2021; Madrid et al., 2022). According to Pekel et al. (2022), the angle of repose is positively correlated with compressibility and the Hausner factor.

No statistically significant difference was observed regarding the angle of repose, which ranged from 48.94 to 49.55º. This behavior was expected, as there was no statistically significant difference in the Hausner Factor, a parameter intrinsically correlated with the angle of repose. According to Macho et al. (2020), more cohesive materials have angles of repose greater than 40º, so the samples are classified as cohesive.

The luminosity (L*) color parameter showed significant variations among the analyzed samples. The CM sample exhibited higher luminosity values (86.8), which was not significantly different from the C sample (86.69) but differed from the M sample. Comparing the extracts with the microencapsulated powders, an increase in luminosity was observed after the encapsulation drying process, indicating that this process directly influences this color parameter.

The a* parameter represents red, and the b* represents yellow and blue coloration (Barros et al., 2024; Simão et al., 2022). The a* parameter ranged from -0.685 to 0.26 for the microcapsules, but only the M experiment differed significantly from the others. Comparing the extracts with the microcapsules, the spray drying process observed an increase in a* values. There was a reduction in the b* parameter due to the drying process, meaning the samples became more yellowish. Additionally, the M sample showed lower values (11.05) concerning this parameter and differed significantly from the CM sample, which showed a higher value (13.52).

One of the factors that can be associated with the change and difference in color, mainly related to obtaining more yellow tones, is the Maillard reaction, a chemical process that occurs during food processing and storage, significantly affecting their sensory and nutritional characteristics. During heating, the Maillard reaction considerably impacts the flavor and color of foods (Liu et al., 2022). This reaction can also occur during storage, along with lipid oxidation (Li et al., 2020).

The effects of the Maillard reaction on the structure and flavor of foods can be positive or negative, potentially resulting in the loss of essential amino acids (Poojary and Lund, 2021). Various factors influence the formation of Maillard reaction products in foods, including pH changes, high temperature, ionic strength, and the presence of digestive enzymes (Nooshkam et al., 2020). Therefore, it is essential to understand the mechanisms

and factors that affect the Maillard reaction to control its effects on foods, aiming to preserve the sensory and nutritional quality of products during processing and storage.

Experimental findings suggest a correlation between the use of modified maltodextrin and the preservation of bioactive compounds, simultaneously associated with a more yellowish hue of the samples. This observation indicates that the chromatic variation is not attributable to lipid oxidation but rather to the inherent color differences of the encapsulating agents used at the beginning of the encapsulation process. This hypothesis is visually corroborated by the comparative analysis presented in Figure 3, where it is observed that modified maltodextrin (Figure 3c) exhibits a significantly darker coloration compared to conventional maltodextrin (Figure 3b).

This phenomenon suggests that the optical properties of the encapsulating materials, specifically modified maltodextrin, directly influence the visual perception of the samples without compromising the integrity of the bioactive compounds. Therefore, the distinction in coloration between the encapsulating agents emerges as a determining factor in the final appearance of the samples, reinforcing the importance of considering the physicochemical characteristics of the materials used in the encapsulation process for preserving and presenting bioactive compounds.

The chroma color parameter, which ranged from 11.08 to 13.52, indicates the ability of dichlorides to recognize color differences, expanding the difference between neighboring pixels in the image space using a Poisson equation (Miyazaki et al., 2021). Although higher values were observed in the CM experiment compared to the M experiment, there was no statistically significant difference concerning the C experiment. These results suggest that the experiments with capsules showed more vibrant colors when compared to the experiment containing only traditional maltodextrin, corroborating the findings of Paiva et al. (2023), who also observed a reduction in chroma (C*) values with the increase in the proportion of maltodextrin.

Chroma acts as a quantitative descriptor of color, facilitating the evaluation of hue variation compared to a gray color of equivalent luminosity. Chroma values close to zero indicate neutral colors (shades of gray), while values close to 60 suggest vibrant colors. Higher chroma values correlate with a more pronounced color intensity, as the human eye perceives (Santos et al., 2023). Therefore, the results obtained in the experiments with capsules, which showed higher chroma values, indicate greater vibrancy and color intensity than the experiment containing only traditional maltodextrin.

The hue angle (h) color parameter represents the relationship between the

intensity of a color and its distance from the luminance of the colored stimulus (Wang et al., 2022). Only the M microcapsule showed a negative value concerning the parameter and differed significantly from the others. The hue and chroma of color can affect the perception of flavors, as found by Wang and Li (2022) in beers (Wang and Li, 2022). On the other hand, the hue angle (h) can be strongly related to some phenolic compounds (Muzolf-Panek and Waśkiewicz, 2022).

### 3.4 Antioxidant Capacity and Total Phenolics of Powder Particles

Table 4 presents the results of the evaluation of antioxidant capacity and total phenolics of the pumpkin seed oil microcapsule formulations.

**Table 4 -** Antioxidant Capacity, Total Phenolics, and In Vitro Digestibility of Pumpkin Seed Oil Microcapsules

| Parameter | Sample | | |
|---|---|---|---|
| | **C** | **M** | **CM** |
| DPPH (mMTrolx/Kg) | $3.02^{a} \pm 0.05$ | $0.90^{c} \pm 0.03$ | $2.71^{b} \pm 0.22$ |
| FRAP (AOX mmol/Kg) | $16.91^{a} \pm 0.07$ | $2.53^{c} \pm 0.08$ | $12.81^{b} \pm 0.03$ |
| $ABTS^{+}$(mMTrolx/Kg) | $45.12^{a} \pm 0.01$ | $12.45^{c} \pm 0.02$ | $22.81^{b} \pm 0.04$ |
| Fenólicos totais (mg/Kg) | $1870^{a} \pm 45.12$ | $871.29^{b} \pm 49.94$ | $1177^{b} \pm 49.94$ |
| **In vitro digestibility** | | | |
| Fase 1 | $1.83^{A} \pm 0.01$ | $0.73^{B} \pm 0.01$ | $1.96^{A} \pm 0.02$ |
| Fase 2 | $10.54^{B} \pm 0.02$ | $9.14^{C} \pm 0.012$ | $11.12^{A} \pm 002$ |
| Fase 3 | $27.35^{A} \pm 0.04$ | $21.13^{B} \pm 0.01$ | $27.88^{A} \pm 0.01$ |

Note: C =Capsul 15%; CM= Capsul 7,5% + Maltodextrina 7,5% e M= Maltodextrina 15%.

Table 4 shows that the microcapsules produced using Capsul as the encapsulating agent exhibited higher values compared to the samples that used only conventional maltodextrin (M) and the combination of maltodextrin with Capsul (CM), both in terms of antioxidant capacity, regardless of the method used, and related to the total phenolic content.

The ABTS method resulted in higher antioxidant activity values for all experiments, ranging from 12.45 to 56.13 mMTrolx/Kg, compared to the values obtained by the FRAP and DPPH methods. According to Amorim et al. (2023), this difference can be attributed to each method's distinct mechanisms of action. Therefore, using a wide range of assays to determine antioxidant capacity is recommended to obtain a more comprehensive and accurate evaluation.

Comparing the values obtained regarding the antioxidant activity of the samples and the phenolic compound content, it was found that there is a degradation of these thermosensitive compounds due to the high-temperature spray drying process. Sun et al. (2020) identified this behavior, finding that increasing the inlet temperature in spray drying decreases antioxidant activity (Sun et al., 2020). However, studies have shown that this effect can be reduced with the use of drying adjuvants (Nguyen et al., 2022).

From the data observed in Table 1 and Figure 1, it was found that the experiment containing only Capsul (modified maltodextrin) as the encapsulating agent showed higher antioxidant capacity values compared to the other samples, indicating that modified maltodextrin can better preserve antioxidant activity compared to traditional maltodextrin. Additionally, it can be seen that there is a reduction in antioxidant capacity during the spray drying process, as, despite the concentration of compounds during drying, the microcapsules show lower antioxidant capacity compared to the extracts.

The experiment containing only Capsul as the encapsulating agent showed higher values regarding total phenolic content, followed by the experiment with mixed encapsulating agent (CM). This indicates that the wall material used strongly influences the maintenance of the total phenolic content of the samples. We can conclude that Capsul protects the phenolic compounds in the matrix from thermal processing, which naturally induces the loss of these compounds due to exposure to high temperatures.

Studies developed by Özbek and Ergönül (2020) indicated that optimizing the composition of the wall material with whey protein concentrate, maltodextrin, and gum arabic can effectively preserve the total phenolic content in freeze-dried pumpkin seed oil. Studies

developed by Ferro et al. (2020) suggested that maltodextrin can preserve the total phenolic content in encapsulated pumpkin powder with an encapsulation efficiency of 89 and 94%, respectively.

Additionally, some studies, such as the one developed by Hong-Bing et al. (2010), show that modified maltodextrin is more suitable for applications in the food industry due to its resistance to retrogradation, Maillard reaction capability, better clarity, and rapid dissolution properties.


**Figure 3 -** Images of Encapsulated Oils; A) Capsul 15%; B) Maltodextrin 15%; C) Capsul 7.5% + Maltodextrin 7.5%
Source: Author (2024)

### 3.5 In Vitro Gastrointestinal Tract Model System

The release of microcapsules containing pumpkin seed oil (PSO) was investigated using different encapsulating agents—maltodextrin (M), modified maltodextrin (C), and a combination of both maltodextrins (CM)—through an in vitro simulation of the gastrointestinal digestive process, with the results presented in Table 3. This study created simulated phases miming salivary, gastric, and intestinal fluids.

A significantly distinct release behavior was observed among the samples throughout the simulated phases ($p < 0.05$), with the lowest release rates being recorded in the salivary fluid phase, ranging from 0.73 to 1.96%. In this phase, there was no significant difference between the C and CM samples, which showed higher values concerning this parameter. The non-protein nature of the wall material of the microcapsules may have been a contributing factor to the reduced release observed, regardless of the concentration used, protecting against the degradation of the microcapsules before the gastric and intestinal phases, as observed by Bastos et al. (2020). This release pattern corroborates the findings of Santos et al. (2024), who reported similar observations during the encapsulation of avocado oil.

In the gastric fluid phase, all tested conditions showed release values close to 10%

($p < 0.05$). This result suggests a gradual release of the encapsulated material in the strongly acidic environment characteristic of the in vitro digestion system. This behavior is typical in controlled release systems, where the encapsulation matrix ensures the protection of the content until it reaches more alkaline pH environments. Previous studies support these findings, such as Pham et al. (2020) in their studies with encapsulated flaxseed oil, which indicated that most encapsulated oil (66-80%) is released in the intestinal phase, and 5-17% is released in the gastric phase. Li et al. (2022) reported that encapsulated goose liver oil shows higher thermal and oxidative stability and 75.2% of the microcapsules can be released in the intestinal region during digestion.

In the intestinal phase, the final stage of the in vitro digestion system, the highest release was detected in samples prepared with CM, a mix of maltodextrin and modified maltodextrin, which showed a magnitude of 27.88%, while the sample containing only traditional maltodextrin (M) released only 21.13% ($p < 0.05$). Therefore, we can infer that the encapsulating materials affect the oil release in the gastrointestinal tract, affecting the water retention capacity and interaction with the bioactive material, as observed by Abraham et al. (2020).

According to Jia et al. (2021), the higher release rate of encapsulated oil in the simulated gastric fluid indicates that the complex of wall materials leads to the bioavailability of the core material under intestinal conditions. Therefore, it can be inferred that the CM sample presents a higher availability of the oil to be absorbed in the intestine.

### 3.6 Structural (FT-IR) and Morphological (SEM) Properties

The FTIR spectra of pumpkin seed oil encapsulated with conventional and modified maltodextrin are shown in Fig. 4. According to Frota et al. (2024), the addition of components to the matrix can favor changes or the appearance of new bands in the FTIR spectra of the samples, indicating possible interactions between the wall materials and the core material. According to Vongsvivut et al. (2012), using ATR-FTIR spectroscopy in microencapsulated oils can determine the compositions of fatty acids with high precision (Vongsvivut et al., 2012).

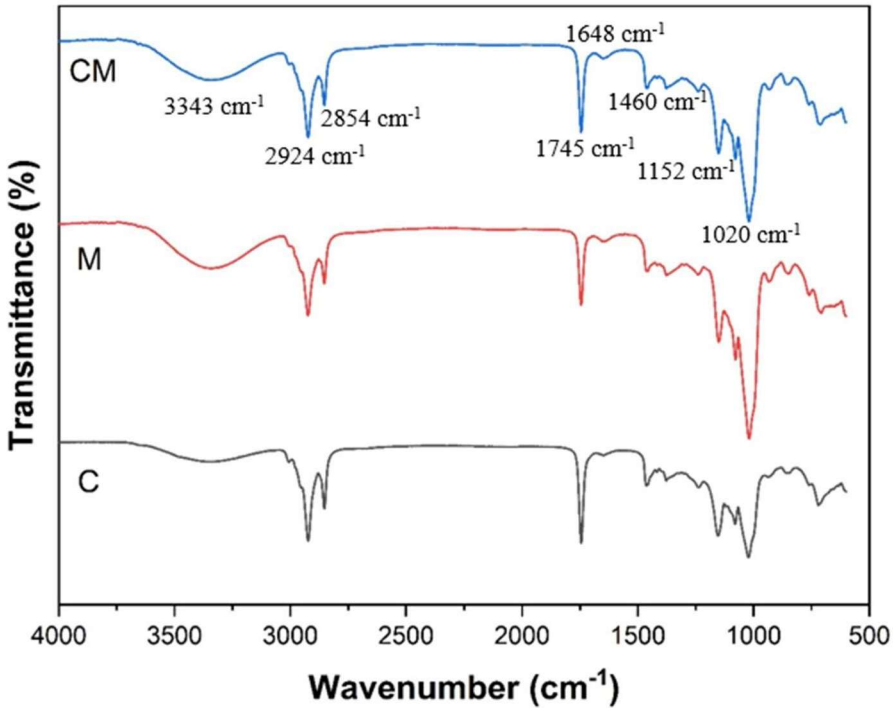



**Fig. 4-** FTIR spectra of encapsulated pumpkin seed oil.

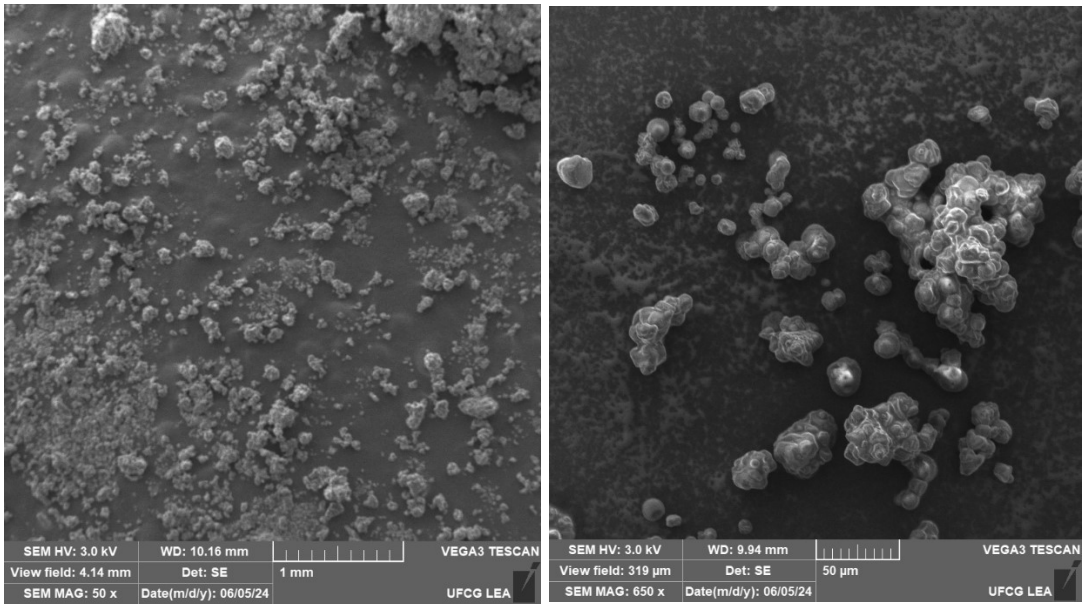
The main fatty acids identified by Barros et al. (2024) in pumpkin seed oil were linoleic, oleic, stearic, palmitic, and nonadecanoic acids, representing 98.21% of the total fatty acids identified through gas chromatography. In all analyzed spectra, characteristic bands of the fatty acids composing the oil inside the microcapsule are indicated. At 2924 and 2854 $cm^{-1}$, the C-H stretching of methyl ($-CH_3$) and methylene ($-CH_2$) structures in the oils is represented.

Additionally, bands in this region indicate cis-alkene (- HC=CH-) in unsaturated fatty acid molecules (da Silva et al., 2022). The band at 1745 $cm^{-1}$ is associated with fatty acid esters (C=O) (Mohammadi et al., 2023), while the bands at 1460 and 1152 $cm^{-1}$ represent deformations of the $–CH_3$ and $–CH_2$ groups (da Silva et al., 2022), and the band at 1020 $cm^{-1}$ corresponds to stretching vibrations (C-OH) (Baharom et al., 2020). The band at 3343 $cm^{-1}$ is linked to the presence of free hydroxyls (OH) (Wang et al., 2023). Furthermore, the bands at 1020 $cm^{-1}$ and 1152 $cm^{-1}$ are also associated with the stretching vibration of the glycosidic bond (C-O-C) belonging to maltodextrin (Wang et al., 2021).

Similar results were observed by Baharom et al. (2020) in sunflower oil as the core content and UF as the microcapsule shell, demonstrating chemical properties similar to

those observed in the present study, characterized by FTIR with the stretching peak at 1537.99 - 1538.90 $cm^{-1}$ (-H in $-CH_2$), 1235.49 - 1238.77 $cm^{-1}$ (C-O-C vibrations in ester), and 1017.65 - 1034.11 $cm^{-1}$ (C-OH stretching vibrations).

Figure 5 illustrates the morphology of the pumpkin seed oil particles. According to Falsafi et al. (2020), the analysis of particle morphology using scanning electron microscopy (SEM) is crucial for the successful development of products and understanding how formulation ingredients affect the structural and (bio)functional properties of bioactive-loaded carriers. Additionally, SEM analysis helps determine the potential for agglomeration, smooth surface, and particle size in powders, aiding in determining product quality (Jain, 2020).



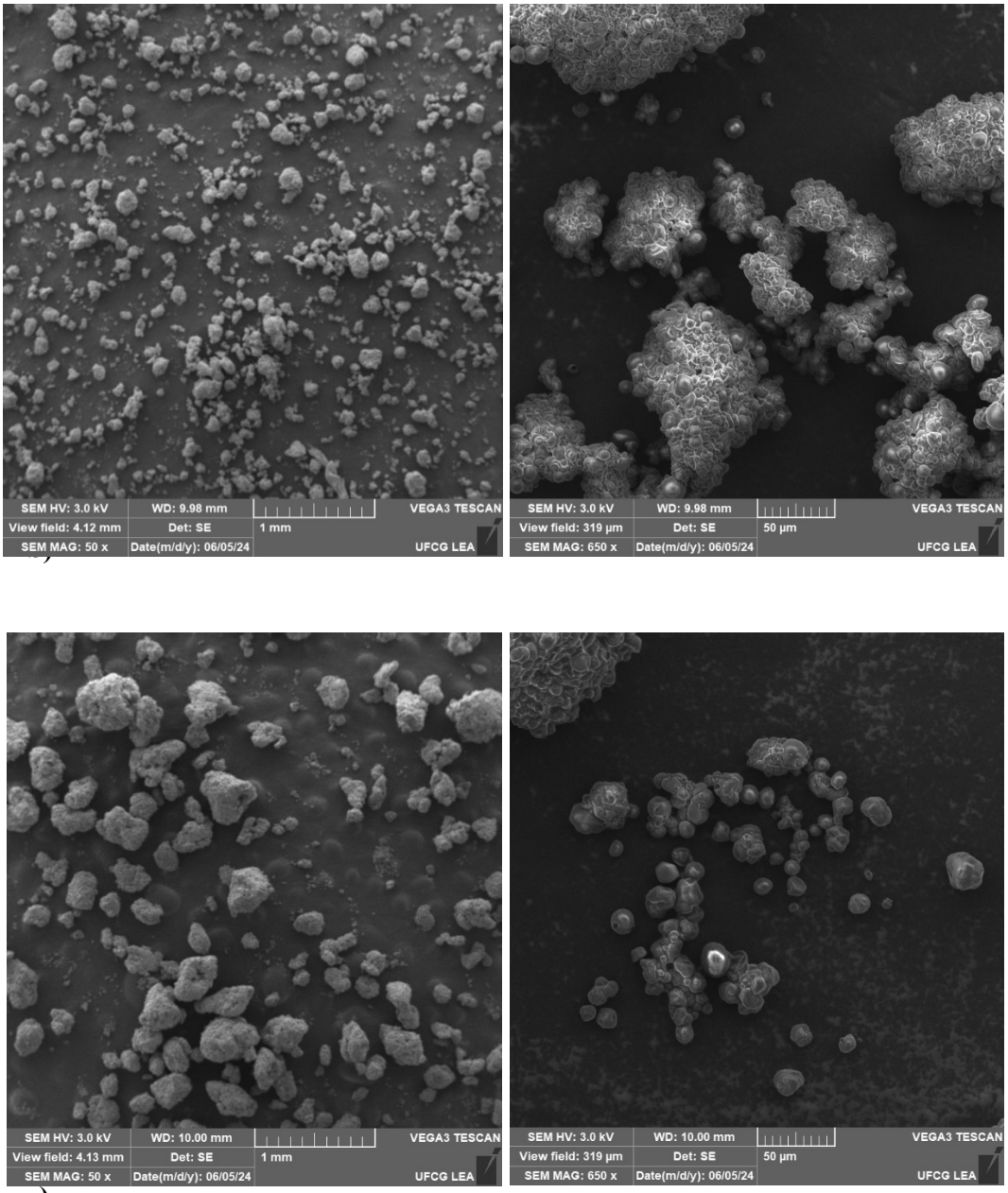



**Figure 5 -** Scanning Electron Microscopy of (A) sample C, (B) sample M, and (C) sample CM at 50x and 650x magnifications, respectively.

Note: C =Capsul 15%; CM= Capsul 7,5% + Maltodextrina 7,5% e M= Maltodextrina 15%.

Through the images obtained from the Scanning Electron Microscopy of the encapsulated pumpkin seed oil powders, it was possible to analyze the morphology of the particles and how they conform. The microcapsules have a rounded shape and smooth surface, demonstrating that the encapsulation occurred efficiently. Additionally, it was observed that the molecules have a high affinity for each other and tend to agglomerate. From the images, it can be inferred that the CM sample has a greater tendency to

agglomerate compared to the other samples; however, it is noted that its particles are smaller in size.

### 3.7 Principal Component Analysis

Figure 6 refers to the correlation analysis between the solubility parameters, zeta potential, release rate, absolute density, and particle size for the encapsulated oil samples. Component 1 represents 99.99% of the variance of this correlation, indicating that the model can adequately represent the samples' behavior.

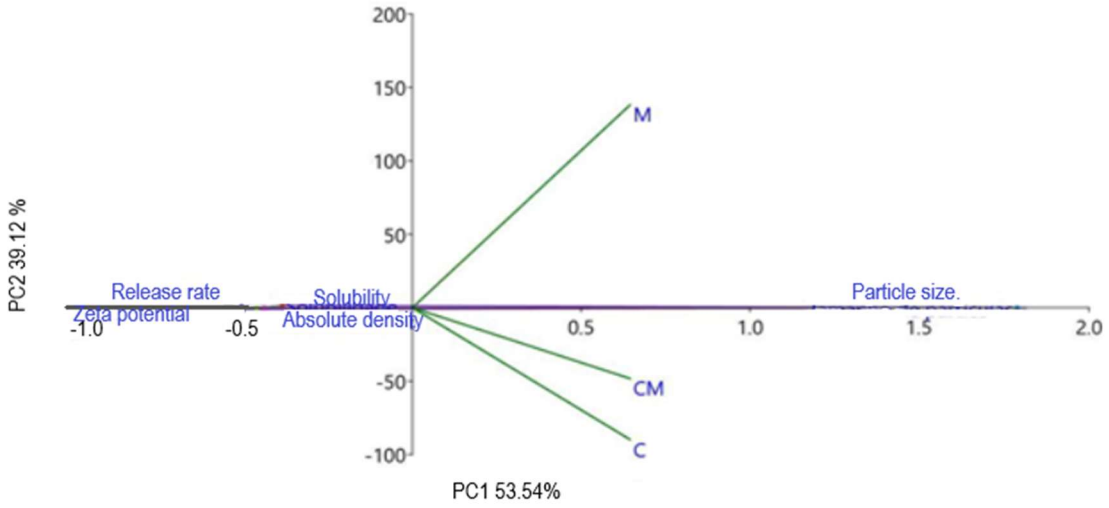



**Figure 6** - Principal Component Analysis (PCA)
Note: C =Capsul 15%; CM= Capsul 7,5% + Maltodextrina 7,5% e M= Maltodextrina 15%.

Analyzing Figure 6, we can infer a negative correlation between particle size and other parameters such as solubility, release rate (in the intestinal phase), zeta potential, and absolute density. In other words, the smaller the particle size, the greater its solubility and release rate. This correlation has been investigated by several researchers, such as Ding et al. (2020), who stated that particle morphology and size are correlated with density. Therefore, smaller powder particles can lead to higher material transport rates, resulting in greater density and lower porosity when mixed with powder (Zhang et al., 2020).

It is also possible to identify the clustering of samples C and CM, which are separate from sample M in component 2. Samples C and CM are closer to the parameters when

analyzing PC1, indicating that the samples exhibit higher solubility, release rate, solubility, and zeta potential than sample M, emphasizing experiment C, which contains only modified maltodextrin and showed superior results.

We can observe that solubility, zeta potential, density, and release rate positively correlate. Hagiya et al. (2022) confirmed this fact, stating that zeta potential is a parameter dependent on surface charge density that can be used to detect modified molecules on the surface of a particle. Thus, it can be inferred that optimizing one parameter can positively influence the others, improving the overall efficacy of encapsulation.

For example, adjusting the surface charge density to increase the zeta potential can improve the active compound's solubility and release rate, optimizing the encapsulated material's delivery and efficacy. Therefore, understanding and controlling these parameters are fundamental for developing encapsulated food products with desired properties, ensuring their functionality, stability, and consumer acceptance.

## CONCLUSIONS

The study concludes that the encapsulation of pumpkin seed oil with different encapsulating agents, such as modified maltodextrin (C), conventional maltodextrin (M), and their combination (CM), significantly impacts the antioxidant capacity and controlled release of the oil during simulated in vitro digestion. The use of Capsul as an encapsulating agent resulted in higher total phenolic content, highlighting the importance of choosing the wall material to maintain the integrity and antioxidant activity of the encapsulated compounds. The analysis of the release of microcapsules in different phases of the in vitro gastrointestinal tract showed that the composition and structure of the microcapsules protect against early degradation, allowing controlled and prolonged release of bioactive compounds. The combination of maltodextrins (CM) showed lower zeta potential values, while sample C (containing only Capsul) showed higher values for this parameter, suggesting a positive influence on the stability and efficiency of encapsulation. This reinforces the importance of the appropriate selection of encapsulating agents to optimize the release and bioavailability of bioactive compounds in food and nutraceutical applications.

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## SUPPLEMENTAL MATERIAL

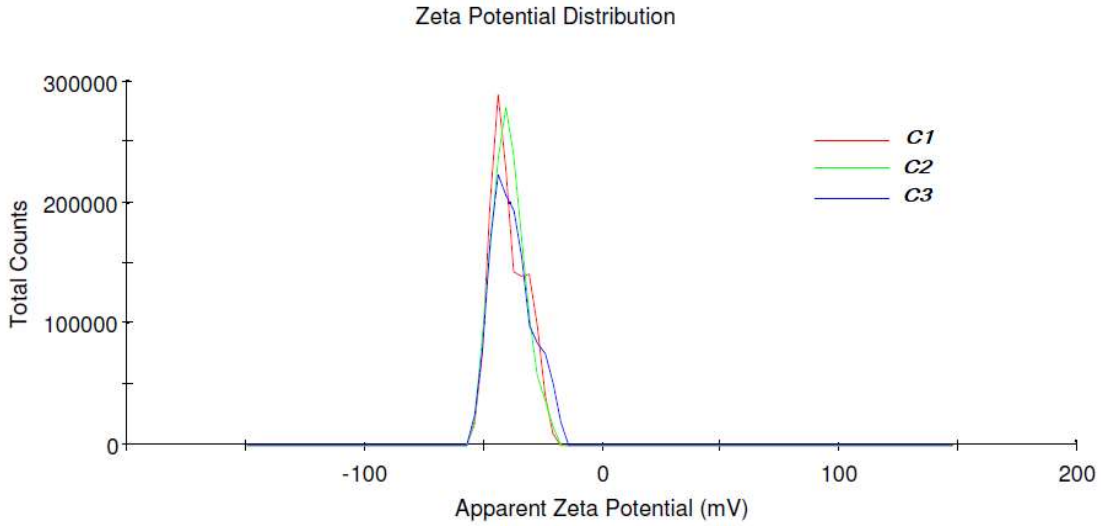


**Figure A1-** Zeta distribution potential of sample C (Oil encapsulated with 15% capsule) and its respective repetitions.

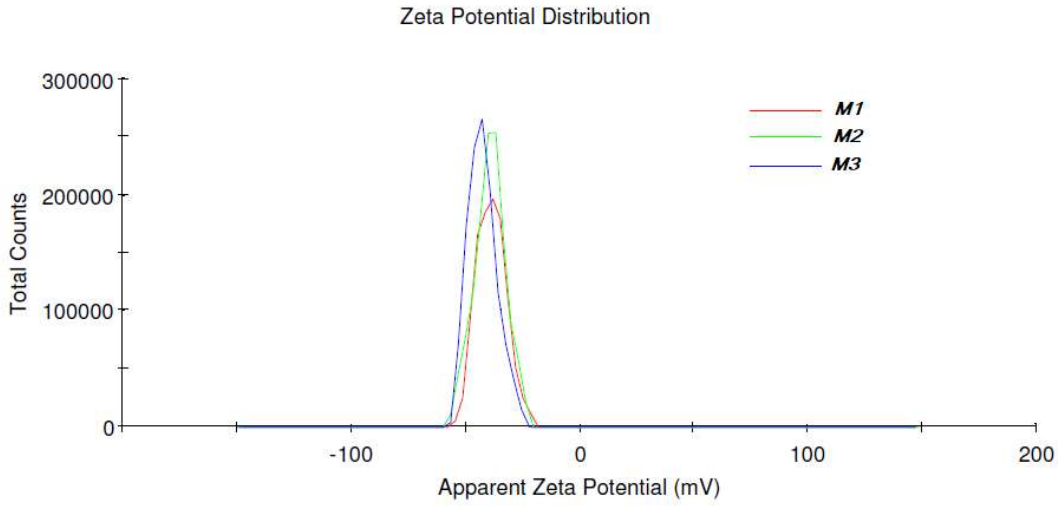


**Figure A2**- Zeta distribution potential of the sample M (Oil encapsulated with 15% maltodextrin) and its respective repetitions.

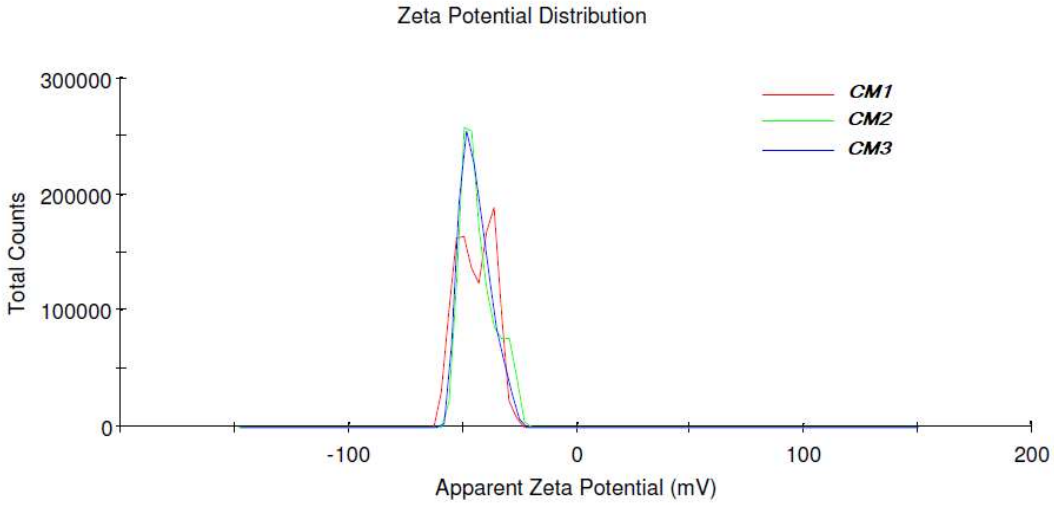


**Figure A3-** Zeta distribution potential of the sample CM (Oil encapsulated with 7.5% maltodextrin and 7.5% capsule) and their respective repetitions.

## FINAL CONCLUSIONS

The economic and health crisis in Brazil has led to an increase in the number of people in vulnerable social conditions, affecting access to food, but it has also resulted in increased consumption of fruits and vegetables. Despite food preservation techniques, food losses and waste continue to exacerbate global hunger, with billions of tons of food wasted annually. Pumpkins can significantly contribute to sustainable development goals, particularly the eradication of hunger (SDG 2) and the reduction of food waste (SDG 12). Achieving these goals requires quality education (SDG 4), interdisciplinary partnerships (SDG 17), and shared infrastructure and innovation (SDG 9). Pumpkin pulp is rich in micronutrients such as polyphenols and carotenoids and can be used in various products, including bread, yogurt, sauces, and chips. Pumpkin peels have nutritional value and technological applications, including the extraction of carotenoids for natural pigments, pectin-rich biodegradable films, and use as thickening agents in food products.

Pumpkin seeds are nutrient-rich but underutilized in human consumption. They possess antifungal and antimicrobial properties, and their oil has nutritional and nutraceutical value, useful in the pharmaceutical and agri-food industries. Pumpkin seed oil can enhance the nutritional value and stability of foods and be used in active packaging to reduce waste. The defatted flour from pumpkin seed oil extraction is protein-rich and can serve as a functional food ingredient with beneficial physiological functions, such as inhibiting bacterial growth and

reducing inflammation. The study demonstrated that the use of ultrasound in the drying process makes the utilization of pumpkin seeds feasible. This method is cost-effective and provides better storage conditions. The encapsulation of pumpkin seed oil with different encapsulating agents, such as modified maltodextrin (C), conventional maltodextrin (M), and their combination (CM), significantly impacts the antioxidant capacity and controlled release of the oil during simulated in vitro digestion. This underscores the importance of selecting appropriate encapsulating agents to optimize the release and bioavailability of bioactive compounds in food and nutraceutical applications.