Ph.D. Thesis

Effect of the inclusion of strawberry by-products as a source of phenolic compounds and dietary fiber on the technofunctional properties of a puffed snack obtained by extrusion

Efecto de la inclusión de subproductos de fresa como fuente de compuestos fenólicos y fibra dietética sobre las propiedades tecnofuncionales de un snack expandido obtenido por extrusión

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A mi familia,
raíz firme y refugio amoroso.

A quienes caminaron a mi lado en los días de luz
y no me soltaron en las noches largas.

A los silencios que me sostuvieron,
a las palabras que me devolvieron la fe.

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fuente amorosa de fuerza,
que me habló en el silencio
y me recordó que la sabiduría no se mide en conocimiento,
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Contents

Chapter 1. Introduction

The snack food market has experienced robust growth in recent years. In 2025, it has been valued at USD 269.45 billion and is projected to grow at a compound annual growth rate (CAGR) of 6.2% through 2030. This expansion has been driven by shifting consumer lifestyles, demand for convenience and portability, flavor and variety innovations, globalization, and a growing interest in natural and organic snacks (Statista, 2025). One of the most dynamic segments within this category is extruded snacks, which reached USD 67.13 billion in 2024 and is expected to exceed USD 110.80 billion by 2034, growing at a CAGR of 5.14% between 2025 and 2034. This growth is largely fueled by rising consumer awareness of nutrition and the demand for healthier snack alternatives (Pandey, 2025).

In Colombia, the snack sector has also undergone sustained growth and transformation since 2020, shaped by global trends and changing consumer preferences (García, 2024). By 2023, the Colombian snack market reached an estimated value of COP 2.5 trillion, growing at an annual rate of approximately 7% (Hurtado, 2024). Per capita consumption of snacks reached nearly 4 kilograms per person in 2023, with extruded and fried snacks among the fastest-growing segments (Cámara de Comercio de Cali, 2020; García, 2024). There has also been a marked shift toward healthier and functional snack options, with an annual per capita expenditure of about USD 47 (García, 2024; Gómez Guasca, 2021).

Extruded snacks are primarily starch-based. During processing, starch undergoes structural transformations that influence expansion and final texture (Neder-Suárez et al., 2024). These changes also increase the product's glycemic load, a common feature of foods rich in gelatinized starch (Nayak et al., 2014; Parada et al., 2019). To mitigate this effect, various strategies have been explored, including processing adjustments, starch modification (Pinky et al., 2015), and the incorporation of ingredients rich in dietary fiber, protein, and phenolic compounds (Chi et al., 2019; Ciudad-Mulero et al., 2018; Kaur et al., 2017; Leoro et al., 2010; Miao et al., 2015). Phenolic compounds, in particular, have attracted attention for their potential to prevent chronic diseases such as cancer, diabetes, cardiovascular conditions, and neurodegeneration. They can interact with carbohydrates, reducing starch digestibility and glycemic response (D. B. Amoako & Awika, 2016; B. Zhao et al., 2018). Fruits, especially berries such as grapes, strawberries, and blueberries, are notable dietary sources of these compounds (Jaroslawska et al., 2011).

Among berries, strawberries are among the most widely cultivated worldwide (Jaroslawska et al., 2011). In Colombia, strawberry production is prioritized within agricultural development programs, with increasing access to international markets. In 2023, national production reached 114,895.2 tons, with the departments of Cundinamarca, Antioquia, and Cauca accounting for the highest volumes (63,033.3, 19,093.9, and 15,178.5 tons, respectively) (Agronet, 2023). These regions have shown consistent growth in recent years (MinAgricultura, 2018, 2025). Approximately 55% of harvested strawberries are consumed fresh, while 45% are processed into products such as pulp and juice (INVIMA, 2022; MinAgricultura, 2017). Juice production generates significant by-products, particularly press cake or pomace consisting of skins, seeds, and residual pulp, representing approximately 4–11% of the total fruit weight (Pukalskienė et al., 2021). These by-products are rich in bioactive compounds, including anthocyanins, proanthocyanidins, ellagic and other phenolic acids, ellagitannins, minerals, and dietary fiber (Cubero-Cardoso et al., 2021; Felix et al., 2018; Pukalskienė et al., 2021; Villamil-Galindo et al., 2022). Despite their high nutritional potential, these materials are often discarded, underscoring the need for innovative valorization strategies. Their incorporation into

extruded snacks offers an opportunity to enhance nutritional and functional properties while contributing to sustainability. Previous studies have explored the use of fruit-derived ingredients and their by-products in extruded snacks to improve their health benefits (da Silva Alves et al., 2018; Khanal et al., 2009; Korkerd et al., 2016; Méndez-García et al., 2011; Oniszczuk et al., 2019; Wójtowicz et al., 2019).

Hypoglycemic effects have also been reported, primarily through reduced starch digestibility via enzyme inhibition (Bahadoran et al., 2013; C. Zhao et al., 2020). In addition, phenolic compounds and fiber may interact with starch to form complexes that modify its structure and digestibility (Chi et al., 2019; Parada et al., 2019). These interactions are complex and depend on factors such as food composition, starch type, phenolic structure, and processing conditions (Parada et al., 2019). Extrusion further modifies the availability and structure of these bioactive compounds, either degrading or enhancing them depending on processing parameters (Arribas et al., 2019; Brennan et al., 2011; Höglund et al., 2018; Leyva-Corral et al., 2016; G. Liu et al., 2019). A deeper understanding of these mechanisms is essential for designing healthier snacks with functional benefits.

Developing healthier snacks is a promising trend that can integrate functional ingredients while reducing the environmental burden of agricultural waste. However, incorporating by-products into extruded matrices presents challenges related to preserving bioactivity and achieving desirable sensory qualities. Therefore, understanding the mechanisms of transformation and interaction of these materials is key to supporting product innovation and development.

Despite growing interest in the development of functional extruded snacks, important knowledge gaps remain regarding the structural transformations and interactions of phenolic compounds, dietary fiber, and starch during extrusion processing. In particular, there is limited understanding of how fruit by-products, with their complex composition, behave during extrusion and influence the technological and nutritional quality of the final product. In the Colombian context, although strawberry production is increasing and generates substantial by-products, their industrial use remains incipient, and studies addressing their effective incorporation into extruded formulations are scarce. Unlocking this potential requires not only characterizing their behavior within the matrix but also evaluating formulation feasibility and the suitability of these materials for product development. Such assessments are essential to determine whether by-products can be successfully scaled up and integrated into commercially viable snacks, aligning nutritional enhancement with market expectations and sustainable innovation. The valorization of fruit by-products into functional ingredients has been identified as a promising strategy to reduce the environmental footprint of food processing and promote resource efficiency, especially when coupled with clean-label trends and circular economy principles (Capozzi, 2022; Santos et al., 2022). These efforts are consistent with international sustainability agendas, notably Sustainable Development Goal 12.3, which calls for a substantial reduction in food losses across production and supply chains, while also contributing to the broader transition toward more resilient and resource-efficient food systems (United Nations, 2025).

In light of the above, this doctoral research aimed to deepen the understanding of how strawberry by-products, rich in phenolic compounds and dietary fiber, can be integrated into starch-based extruded snacks, and how their inclusion affects key technological and nutritional properties relevant to product quality and health potential. The study is structured around three main objectives, which are addressed in Chapters 3 to 5. Chapter 2 presents a comprehensive review of literature related to the inclusion of plant-derived materials in extruded snacks, focusing on the effect of the extrusion on phenolic compounds, dietary fiber, and starch. Chapter 6 provides an integrated discussion of the findings across the experimental chapters, highlighting the main contributions, limitations, and perspectives for future research.

Research question

How does the inclusion of strawberry by-products impact the technofunctional and structural properties of an extruded puffed snack, contributing to their potential valorization as a sustainable ingredient?

General objective

To evaluate the effect of including strawberry by-products as a source of phenolic compounds and dietary fiber on the technofunctional properties of a puffed snack obtained by extrusion-cooking, with specific focus on potential interactions among matrix constituents.

Specific objectives

- To optimize extrusion parameters for the development of starch-based puffed snacks enriched with strawberry by-products, assessing their effects on polyphenol composition, textural attributes, and physical quality.
- To investigate the behavior of dietary fiber and phenolic compounds from whole and fractionated strawberry by-products during extrusion to elucidate their transformation and interaction in extruded systems.
- To analyze extrusion-induced transformations in starch structure and its *in vitro* digestibility, glycemic response, and the release of phenolic compounds in model systems containing strawberry by-products.

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Chapter 2. Plant-derived ingredients as a source of phenolic compounds in extruded snacks: Functional insights and the role of dietary fiber and starch

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Abstract

The incorporation of plant-derived ingredients into extruded snacks offers a promising strategy for enhancing nutritional profiles and delivering health-promoting compounds. These materials serve as natural sources of phenolic compounds and dietary fiber, complementing the starch-rich base that supports product structure and expansion. This review provides a comprehensive analysis of how extrusion processing, through variables such as temperature, moisture content, and screw speed, modulates the stability, extractability, and bioaccessibility of phenolic compounds. It also examines the structural modification of both dietary fiber and the underlying starch matrix during high-temperature, short-time treatment, highlighting their interdependent effects on product characteristics. Optimization studies frequently target techno-functional properties such as expansion ratio, firmness, and crispness, balancing these with the desire to preserve or even enhance the functional potential of bioactive compounds. By integrating insights on ingredient composition, matrix behavior, and process dynamics, this review outlines pathways to design extruded snacks that achieve superior nutritional value, structural integrity, and sensory appeal.

Keywords: functional foods, extrusion, polyphenols, polysaccharides, interactions, bioaccesibility

1. Introduction

Extrusion is a versatile and sustainable food processing technology based on high temperature–short time (HTST) treatment. It is considered cost-effective due to its lower energy consumption and continuous processing capability when compared to conventional cooking and forming methods (Alam et al., 2016; Altan et al., 2009). Before extrusion, raw materials are typically conditioned and blended with various functional ingredients to produce a broad range of ready-to-eat products, including breakfast cereals, baby foods, instant soups, and snacks (Ruiz-Gutiérrez et al., 2018).

Among these applications, extruded snacks represent one of the most dynamic and widely consumed product categories, owing to their sensory appeal, portability, and long shelf life (Tas & Shah, 2021). The formulation of such products is often based on carbohydrate-rich matrices, particularly those containing starch, which plays a central role in shaping key quality attributes such as texture, color, and expansion (Alam et al., 2016; Mironeasa et al., 2023). Starch not only contributes to energy content but also governs the rheological behavior of the melt and the structural formation of the extrudate (Alam et al., 2016). The incorporation of plant-derived ingredients, including fruits, vegetables, legumes, seeds, and their by-products, has gained increasing attention as a means to enhance the nutritional value and functional potential of these products (Grasso, 2020; Patil & Kaur, 2018). These ingredients are valued for their micronutrients, fiber content, and especially for their richness in phenolic compounds, which are associated with a wide range of health-promoting effects (Šárka et al., 2021).

Phenolic compounds represent a broad class of secondary plant metabolites that include flavonoids, phenolic acids, coumarins, stilbenes, xanthones, quinones, and lignans, among others (Giuberti et al., 2020; Pinarli et al., 2020). These compounds are synthesized by plants in response to stress conditions such as physical damage, pathogen attack, or UV radiation (Pinarli et al., 2020). Phenolic compounds have been extensively studied for their potential health benefits, including preventive and supportive effects in cancer, diabetes, and disorders affecting the skin, bones, and metabolism (Dangles, 2020; Pinarli et al., 2020). These effects are largely attributed to their antioxidant and anti-inflammatory activities, which may involve various biological mechanisms beyond direct radical scavenging (Dangles, 2020). Phenolic compounds interact with macronutrients in the food matrix, contributing

to reduced glycemic load, lower caloric availability (D. Amoako & Awika, 2016; B. Zhao et al., 2018; C. Zhao et al., 2020), and hypolipidemic effects (Benítez et al., 2021; Gong et al., 2020).

In addition to their phenolic content, plant-derived ingredients are also valuable sources of dietary fiber, which plays a particularly important role, not only for its well-documented health benefits (Anderson et al., 2009; Stephen et al., 2017), but also for its effect on processing and product texture. Extrusion can cause significant modifications in the structure and physicochemical properties of fiber. These changes depend on several factors, including the type of fiber, the presence of other components in the matrix, and extrusion parameters like moisture content, temperature, and screw speed (H. Chen et al., 2018; Redgwell et al., 2011; M. Zhang et al., 2011). It is reported that extrusion can increase the content of soluble fiber, which may improve digestibility and contribute to health-related outcomes such as glycemic control and gut health (M. Zhang et al., 2011). At the same time, these structural transformations in fiber can influence the expansion ratio, density, and crispness of the final extruded product, highlighting the role of fiber as a relevant component in balancing both the nutritional and textural attributes of functional snacks (Robin et al., 2012).

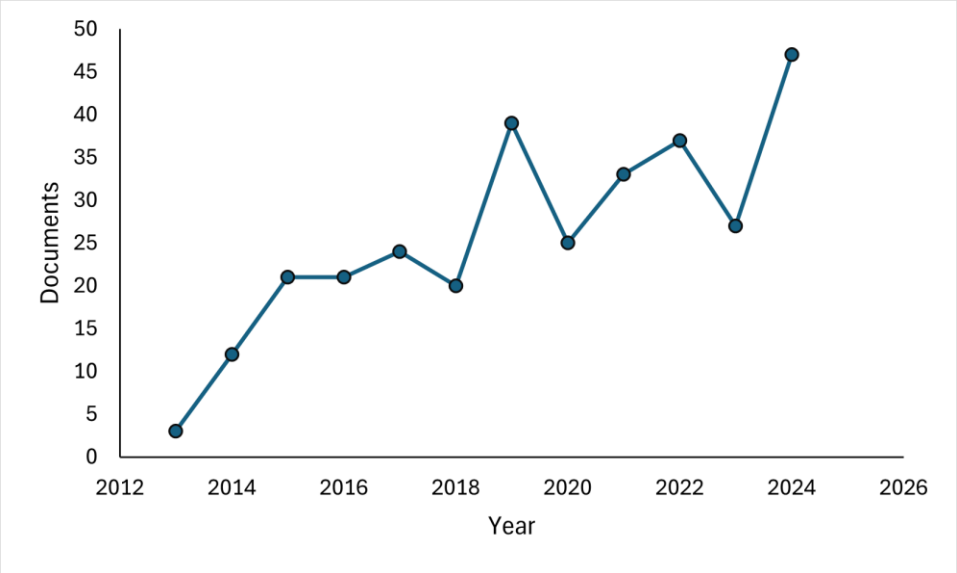

Fig. 1. Scientific publication trend on functional extruded foods (2013–2024).
Number of documents retrieved from Scopus using the query: TITLE-ABS-KEY ((extrusion OR extruded) AND ("functional food*" OR "functional ingredient*")) AND PUBYEAR > 2009 AND PUBYEAR < 2026 AND (LIMIT-TO (DOCTYPE , "ar"))

Several reviews have examined the incorporation of plant-derived materials into extruded foods, emphasizing their nutritional, functional, technological, and environmental potential. Some focused on specific ingredients, such as brewers' spent grain (Beltrán-Borbor et al., 2025), while others addressed broader sources like cereals and legumes (Cotacallapa-Sucapuca et al., 2021; Orozco-Angelino et al., 2023), highlighting the effects of extrusion parameters on protein digestibility, fiber solubility, and the bioavailability of phenolic compounds. Other works reviewed the use of both fresh and processed plant materials in extrusion (Grasso, 2020; Leonard et al., 2020; Offiah et al., 2019), or reported advances in the formulation of cereal-, millet-, and pulse-based functional ingredients (Patil & Kaur, 2018). Šárka et al. (2020) specifically addressed the inclusion of phenolic-rich ingredients in extrudate formulations and their resulting phenolic content.

Collectively, these reviews highlight the versatility of extrusion as a processing technology and its potential to enhance the nutritional and functional quality of plant-based foods. They support the development of new formulations aligned with health promotion and sustainability goals. However, most of these reviews focus on general compositional and techno-functional aspects, while few have explored in detail the fate of phenolic compounds during extrusion, including changes in their content and composition, their bioaccessibility, and their interactions with macromolecules in complex food matrices.

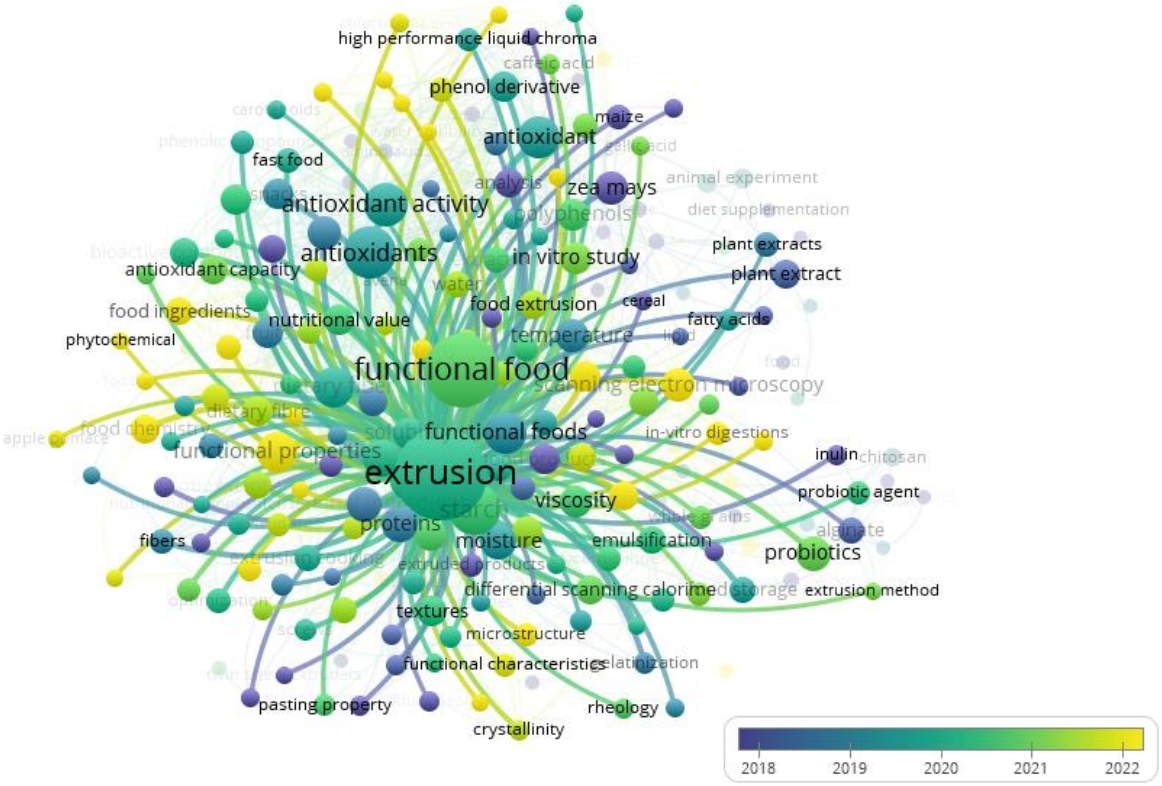

Fig. 2. Co-occurrence network of keywords in research on plant-derived ingredients in functional extruded foods. Visualization generated with VOSviewer based on Scopus data using a refined query focused on extrusion and plant-derived ingredients. Node size represents keyword frequency, edge thickness indicates co-occurrence strength, and the color gradient reflects the average year of appearance, highlighting recent trends in yellow.

This growing scientific interest in extrusion for functional food development is also evident in the increasing number of related publications over the last decade (Fig. 1). While these studies cover a broad spectrum of ingredients and objectives, a more focused view (Fig. 2) reveals the consolidation of a research niche dedicated to the inclusion of plant-derived materials in extruded functional foods. The co-occurrence network illustrates frequent associations of the term extrusion with functional food, snacks, antioxidants, fruits, dietary fiber, and starch. Moreover, the color gradient indicates a shift in recent years toward emerging topics such as flavonoids, functional ingredients, digestion, and extruded snacks, reflecting growing interest in the health-promoting potential of specific bioactive compounds and their behavior during extrusion.

Therefore, the aim of this review is to provide a comprehensive and focused analysis of the inclusion of plant-derived materials in extruded snack-type products, addressing the impact of extrusion on functional constituents of the food matrix, including phenolic compounds, dietary fiber, and starch. Particular attention is given to changes in their content and composition, as well as their interactions

with other matrix components, considering both their health-related functions and their influence on the nutritional and technological quality of the final products. Additionally, this review examines current optimization strategies used to maximize phenolic retention and enhance techno-functional properties, highlighting the role of process parameters and formulation factors in achieving a balance between bioactive preservation and product quality.

2. Methodology

A targeted literature search was carried out primarily through the Scopus database, complemented with additional screening via Google Scholar to ensure comprehensiveness. The search aimed to identify original experimental research articles focused on the behavior or modification of phenolic compounds, dietary fiber, or starch during the extrusion of plant-derived ingredients in extruded snacks. Boolean operators and combinations of keywords such as extrusion, extruded snacks, phenolic compounds, polyphenols, dietary fiber, starch, functional snacks, plant-based ingredients, by-products, bioaccessibility, structure, and optimization, were used to construct the queries. Although no time filter was applied during the database search, the final selection comprised mainly studies published between 2015 and 2025, with only a few exceptions from earlier years. The search was conducted up to February 2025.

Articles not related to extruded snacks, or those using isolated or purified compounds (e.g., extracts or concentrates) rather than whole or minimally processed plant-derived ingredients, were excluded. The final review includes 215 references, of which 108 are studies that specifically evaluated the effect of the extrusion process on physical and textural properties and/or key components such as polyphenols, fiber, or starch. Additional sections in this review provide a broader context by addressing structural and functional characteristics of phenolic compounds and polysaccharides, as well as a general overview of extrusion technology.

3. Structure and functionality of polysaccharides constituents: Starch and dietary fiber

3.1 Starch

Starch is the major polysaccharide found in starch-based extruded snacks and constitutes the primary structural and energy component of many formulations. It is a semicrystalline biopolymer composed of two glucose homopolymers: amylose, a mostly linear molecule linked by α-1,4-glycosidic bonds, and amylopectin, a highly branched polymer with both α-1,4 and α-1,6 linkages. These two components differ in molecular weight, structure, and functionality, with amylopectin typically representing 70–80% of starch in most plant sources (F. Zhu, 2015).

The behavior of starch during extrusion is largely determined by its physicochemical properties, such as granule size and shape, amylose-to-amylopectin ratio, and degree of crystallinity, as well as by processing parameters like temperature, moisture, shear rate, and residence time. Under the high-temperature and high-shear environment of extrusion, starch undergoes gelatinization and partial dextrinization, processes that disrupt crystalline regions and result in an amorphous structure capable of forming new molecular configurations (Alam et al., 2016; Babatunde et al., 2023; V. T. Huang & Perdon, 2020; Neder-Suárez et al., 2024; Qiu et al., 2024). The extent of this transformation increases with the contribution of mechanical energy during processing, commonly expressed as specific mechanical energy (SME). This has been shown to significantly enhance starch conversion compared to conventional cooking at similar temperature and moisture levels (Wolf, 2010). These structural modifications are essential for extrudate formation and directly influence key physical attributes such as expansion, porosity, crispness, and mechanical strength (Neder-Suárez et al., 2024; Qiu et al., 2024).

In terms of technological functionality, starch plays a key role in texture development. The extent of gelatinization and fragmentation during extrusion affects water absorption, melt viscosity, and expansion index, which in turn determine whether the final product will be airy and crispy or compact and dense (Alam et al., 2016; Varsha & Mohan, 2016). Amylopectin-rich starches typically promote greater expansion and lighter textures, while amylose-rich starches may yield denser structures due to lower swelling capacity (Hellemans et al., 2020). Furthermore, the formation of starch-protein or starch-fiber matrices can influence the rheology of the melt and the uniformity of cell structures within the extrudate (Alam et al., 2016).

From a nutritional perspective, extruded starch may retrograde during post-extrusion cooling and storage, leading to the formation of ordered regions that are more resistant to enzymatic digestion (Qiu et al., 2024). This retrogradation, along with the formation of amylose–lipid or amylose–phenolic compound complexes, contributes to the generation of slowly digestible starch (SDS) or resistant starch (RS), thereby modifying the glycemic response of the final product (D. Amoako & Awika, 2016; Huo et al., 2025; Mohamed, 2023; Qiu et al., 2024).

3.2 Dietary fiber

Dietary fiber refers to a complex and heterogeneous group of indigestible plant-derived components, primarily composed of carbohydrate-based substances such as non-starch polysaccharides (NSPs), but also including non-carbohydrate constituents like lignin (Suresh et al., 2024). These compounds resist digestion and absorption in the human small intestine and reach the colon, where they may be partially or fully fermented (S. Li et al., 2023; Stephen et al., 2017). NSPs include cellulose, hemicelluloses, pectins, β-glucans, and arabinoxylans (Waldron & Faulds, 2007). These constituents differ in monomeric composition, linkage types, branching patterns, and molecular weight, which directly influence their physicochemical behavior and physiological effects (Kumar et al., 2012; Waldron & Faulds, 2007).

The main structural components of dietary fiber are the polysaccharides of the plant cell wall, primarily cellulose, hemicelluloses, and pectins, along with other elements such as lignin, gums, starch remnants, and oligosaccharides (Núñez-Gómez et al., 2023). Cellulose consists of linear chains of D-glucose that form microfibrils, which are embedded in a matrix composed of hemicelluloses and pectins or lignin (Gibson, 2012). Hemicelluloses associate with cellulose microfibrils through hydrogen bonding, contributing to the flexibility and mechanical strength of the cell wall. They are generally classified into four structural groups: xyloglycans, mannoglucans (mannans), xyloglucans, and mixed-linkage β-glucans (Loix et al., 2017). Pectins, which are rich in galacturonic acid, provide porosity and firmness to plant tissues, playing a central role in intercellular adhesion and mechanical resistance, as well as in the regulation of cell development (Lara-Espinoza et al., 2018). During plant maturation or secondary growth, some cells develop a secondary cell wall, where lignin is deposited within the polysaccharide network. Lignin is a phenolic polymer composed mainly of guaiacyl (G), syringyl (S), and p-hydroxyphenyl (H) units, conferring rigidity, hydrophobicity, and resistance to microbial degradation (Yao et al., 2022).

From a technological standpoint, dietary fiber significantly influences the behavior of plant-derived ingredients during food processing. In extrusion systems, NSPs contribute not only to the nutritional value but also to the structural, rheological, and textural characteristics of the final product (Robin et al., 2012; Wolf, 2010). In terms of physiological functionality, soluble fibers like pectins and β-glucans are well-known for forming viscous solutions that slow gastric emptying, reduce nutrient absorption rates, and increase satiety. They are also associated with reductions in glycemic response and plasma cholesterol levels (Dragan et al., 2021). Insoluble fibers such as cellulose and lignin

primarily contribute to increased fecal bulk and promote intestinal motility (Suresh et al., 2024). Furthermore, both soluble and insoluble fibers can interact with other dietary components, such as starch and phenolic compounds, either by physical entrapment or through molecular interactions, thereby modulating their digestibility, glycemic response, and bioactive potential (Giuberti et al., 2020; C. M. G. C. Renard et al., 2017).

Thus, dietary fiber plays a dual role in extruded food matrices: it contributes to both nutritional value and processing functionality. A thorough understanding of its structural organization within the plant cell wall and its behavior under extrusion conditions is essential for developing functional, fiber-rich extruded products with optimized health and textural attributes.

4. Structure and functionality of phenolic compounds

Phenolic compounds are a large and diverse group of plant secondary metabolites found in a wide range of plant-based foods such as fruits, vegetables, whole grains, cereals, legumes, tea, coffee, wine, and cocoa. They consist of phenolic units (Fig. 3) and are more commonly found in conjugated forms, where sugar residues are attached to hydroxyl groups. Over 8,000 phenolic compounds have been identified, broadly categorized into phenolic acids, flavonoids, stilbenes, lignans, and polymeric phenols (Bahadoran et al., 2013; H. Zhang et al., 2014). These compounds play a protective role in plants, acting against ultraviolet radiation, oxidative stress, and microbial pathogens (Bahadoran et al., 2013).

Phenolic acids are among the simplest phenolic compounds, composed of a single aromatic ring with one or more hydroxyl groups. They are typically classified as hydroxybenzoic acids (e.g., gallic acid) and hydroxycinnamic acids (e.g., ferulic, caffeic, and p-coumaric acids), and are commonly found in foods such as berries, kiwi, cherry, apple, pear, chicory, and coffee (Bahadoran et al., 2013; Nahar et al., 2021).

Flavonoids, characterized by a C6-C3-C6 structure, represent one of the most abundant phenolic compound groups (Dias et al., 2021). Subclasses include anthocyanins, flavonols, flavanols, flavanones, flavones, and isoflavones. Anthocyanins such as cyanidin, pelargonidin, delphinidin, and malvidin are found in berries, red wine, red cabbage, cherries, black grapes, and strawberries. Flavonols like quercetin, kaempferol, and myricetin are present in onions, kale, leeks, broccoli, and blueberries. Isoflavones (e.g., daidzein, genistein, glycitein) are predominantly found in soy products and are noted for their estrogenic activity (Bahadoran et al., 2013; Dias et al., 2021; Nahar et al., 2021).

Stilbenes, with a 1,2-diphenylethylene core (Sirerol et al., 2016), are less abundant in the diet. The most studied example is resveratrol, primarily found in grapes and red wine (Bahadoran et al., 2013).

Lignans consist of two phenylpropanoid units joined at their central carbon atoms. They occur mainly in seeds, whole grains, and vegetables and are recognized for their phytoestrogenic properties, with potential benefits for hormone-related conditions (J. Chen et al., 2021).

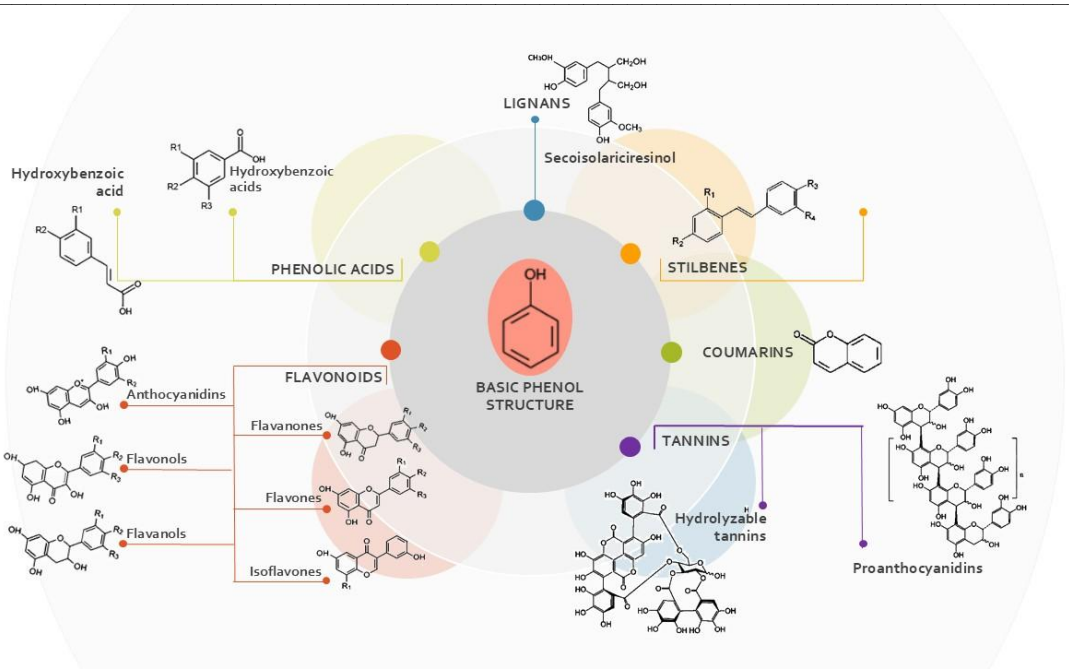

Fig. 3. Overview of the chemical diversity of phenolic compounds in plant-derived foods

Coumarins are a class of phenolic compounds characterized by a benzopyrone backbone. They are widely distributed across plant species, particularly within the Apiaceae, Asteraceae, and Rutaceae families (Nahar et al., 2021; Nan et al., 2025). Common dietary sources include apricots, carrots, celery, cherries, citrus fruits, parsnips, and strawberries, as well as aromatic spices such as aniseed, caraway, cinnamon, coriander, dill, and fennel. Structurally, coumarins are classified into several types, including simple coumarins, prenylated and geranylated forms, as well as furano-, pyrano-, sesquiterpenyl-, and oligomeric derivatives (Nahar et al., 2021).

Tannins are high-molecular-weight phenolic compounds, divided into hydrolyzable tannins and condensed tannins. Hydrolyzable tannins yield gallic or ellagic acid upon hydrolysis (Nahar et al., 2021), whereas condensed tannins, also known as proanthocyanidins (PACs), are composed of flavan-3-ol units (e.g., catechin, epicatechin). These are the second most abundant class of phenolics after lignin, and are present in fruits, cereals, beans, nuts, and spices (Sieniawska et al., 2021). Their degree of polymerization (mDP) affects solubility, antioxidant potential, and reactivity (M. H. Chen et al., 2016; Hellström et al., 2009). Although highly polymerized forms exhibit lower solubility and bioaccessibility (M. H. Chen et al., 2016; X. Liu, Le Bourvellec, et al., 2021), they may interact more with matrix macronutrients such as proteins and carbohydrates, affecting both nutritional and techno-functional aspects of food (Jakobek, 2015; C. M. G. C. Renard et al., 2017).

The structural diversity of phenolics is key to their biological function. Attributes liken hydroxylation patterns, glycosylation, and polymerization influence physicochemical properties and bioactivity (Z. Chen et al., 2024). Antioxidant activity, for instance, is associated with the number and position of hydroxyl groups, which enhance radical scavenging by donating hydrogen atoms or electrons. Conjugation and electron-donating substituents further stabilize phenoxyl radicals, increasing antioxidant efficacy (Dias et al., 2021).

Beyond antioxidant effects, phenolics demonstrate anti-inflammatory, antimicrobial, and anticancer activities through enzyme modulation, gene expression, and signaling pathway regulation (Dias et al., 2021). They also positively modulate the gut microbiota, promoting beneficial microbes while suppressing pathogens (Catalkaya et al., 2020). Additionally, hypoglycemic and hypolipidemic effects have been reported, involving inhibition of digestive enzymes such as α-amylase and lipase, modulation of glucose transporters, and regulation of lipid metabolism (Benítez et al., 2021; C. Zhao et al., 2020).

In food systems, especially under processing methods like extrusion, the structure of phenolic compounds significantly influences their stability and functionality. High temperature, pressure, and shear can degrade or transform phenolics or disrupt their interactions with other macromolecules, thereby affecting bioaccessibility and bioavailability (Brennan et al., 2011). These transformations may involve bond cleavage or cell wall disruption, facilitating phenolic release (Ribas-Agustí et al., 2018).

Understanding these structural characteristics and their behavior under processing conditions is critical for improving the functional role of phenolic compounds in food design. This knowledge enables the development of strategies to preserve or enhance the health-promoting properties of phenolics in processed foods.

5. Extrusion technology in snack production

Snack products are widely consumed convenience foods appreciated for their sensory appeal, portability, and long shelf life. They include a broad range of products such as chips, crackers, puffs, bars, and extruded cereals, which may be savory or sweet, fried or baked, and formulated with various base ingredients. Common production technologies include frying, baking, roasting, puffing, and extrusion. Among these, extrusion stands out for its versatility, scalability, and efficiency (Ali et al., 2024; Bhattacharya, 2023)

Extrusion cooking is a high-temperature, short-time (HTST) process that combines mechanical shear and thermal energy to transform raw materials into shaped and/or expanded products with desired structural and sensory characteristics (Offiah et al., 2019). This continuous process allows for simultaneous cooking, texturizing, shaping, and drying of food mixtures, making it highly suitable for the manufacture of ready-to-eat snacks. It also enables the incorporation of a wide variety of functional ingredients, offering opportunities for product innovation (Alam et al., 2016; Offiah et al., 2019; Sule et al., 2024). A schematic representation of a food extruder system is shown on Fig. 4.

Key extrusion parameters include feed moisture content, barrel temperature, screw speed, screw configuration, die shape, and specific mechanical energy (SME). These parameters directly affect starch gelatinization, protein denaturation, fiber solubilization, and lipid transformations, which in turn determine the expansion, density, crispness, porosity, and texture of the final product (Alam et al., 2016; Offiah et al., 2019; Ruiz-Gutiérrez et al., 2018; Sule et al., 2024). For instance, by reducing the moisture, SME increases, which increases product expansion and breaking strength (Alam et al., 2016).

The physical quality attributes of extruded snacks, such as expansion ratio, hardness, bulk density, and surface morphology, are largely governed by starch behavior and its interaction with other matrix components under thermal-mechanical treatment (Alam et al., 2016; Varsha & Mohan, 2016). Texture, a key factor for consumer acceptance, is closely linked to bubble formation and stabilization in the melt, which are influenced by ingredient composition and process conditions (Alam et al., 2016; Sule et al., 2024).

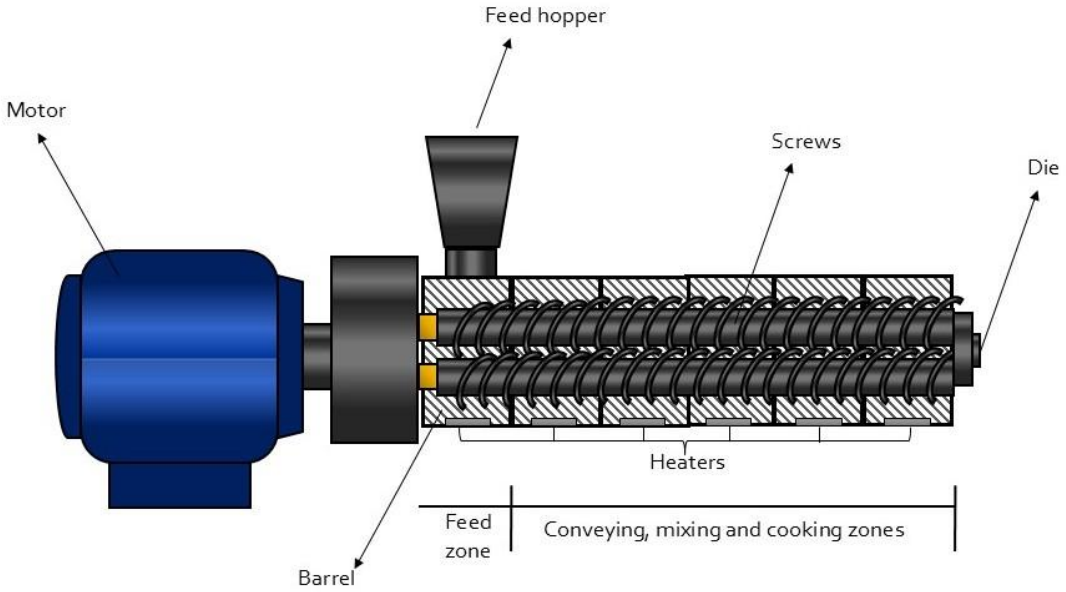

Fig. 4. Schematic representation of a typical food extruder system

From a nutritional perspective, extrusion offers advantages such as the inactivation of antinutritional factors, increased digestibility of starch and protein, and the ability to encapsulate or retain certain bioactive compounds. However, it may also lead to losses in heat-sensitive micronutrients or bioactives if not properly optimized (Alam et al., 2016; Ruiz-Gutiérrez et al., 2015; Sule et al., 2024). Despite these challenges, extrusion is recognized as a powerful technology for developing functional snacks, especially when combined with the inclusion of plant-derived materials rich in dietary fiber, antioxidants, and phytochemicals (Ruiz-Gutiérrez et al., 2018).

Extrusion technology plays a central role in modern snack production by offering a flexible, energy-efficient, and cost-effective method for manufacturing diverse, shelf-stable, and health-oriented products. Its adaptability to incorporate novel ingredients and modify product structure makes it a strategic platform for meeting consumer demands for both indulgence and nutrition.

6. Plant-derived ingredients in extruded snacks

Plant-derived ingredients encompass a wide range of materials originating from parts of plants, such as fruits, vegetables, legumes, cereals, seeds, and leaves, that are incorporated into food formulations either in raw, dried, powdered, or otherwise minimally processed forms. This category also includes by-products generated during the processing of these plant parts, such as pomace, peels, press cakes, and other fiber-rich fractions (Offiah et al., 2019). These by-products often represent between 10% and 70% of the original fresh material and, in many cases, exhibit higher concentrations of nutritional or functional components than the final products (Rațu et al., 2023). The increasing incorporation of plant-based materials into extruded snacks is largely motivated by their ability to enhance both the nutritional value and functional properties of foods, particularly in the development of health-oriented products (Arribas, et al., 2019c; Jozinović et al., 2021). However, it is important to acknowledge the inherent compositional differences between whole plant materials and their derived by-products. Processing can either concentrate or deplete specific components; for instance, peeling fruits and

vegetables generally reduces fiber content, although dietary fiber is still present in the pulp and seeds (Timm et al., 2023).

The type and content of phenolic compounds also differ significantly between whole plant materials and their by-products. In whole fruits, phenolics are predominantly found in vacuoles as free forms. In contrast, fruit by-products, particularly those generated after juice extraction, contain phenolics both in free form and bound to the cell wall matrix of the pomace (Danilov et al., 2024; Sójka et al., 2013). For example, strawberry press cake has been shown to retain high levels of ellagitannins and proanthocyanidins, likely due to their strong affinity for cell wall components. Anthocyanins, typically vacuole-localized, are substantially removed during juice extraction and transferred to the juice phase (Sójka et al., 2013). Other factors such as ripeness can also affect phenolic distribution and extractability. Brahem et al. (2017) observed that in pears, the overall phenolic composition of the fruit did not vary significantly during overripening. However, the efficiency of phenolic extraction into juice declined, falling from 38% and 28% in ripe fruit to 26% and 15% in overripe fruit for different cultivars. Notably, procyanidins exhibited greater affinity for cell walls during the overripening stage.

Comparative studies on the phenolic and fiber profiles of primary plant fractions (e.g., pulp, juice, or whole edible parts), and their corresponding by-products (e.g., peels, hulls, pomace) have consistently shown compositional differences (Table 1). These differences have been documented in fruits (Diamanti et al., 2017; Garcia-Amezquita et al., 2018; Gurak et al., 2014; Santiago et al., 2018; Sir Elkhatim et al., 2018), vegetables (Kim et al., 2024; Ying et al., 2021), legumes (Manco et al., 2023). These studies have demonstrated that both types of materials can enrich food formulations, however, given this compositional heterogeneity, careful evaluation of agri-food by-products is crucial when designing strategies for their valorization in functional snack production (Rațu et al., 2023).

The relevance of plant-derived ingredients lies in their natural content of bioactive compounds, such as dietary fiber and phenolic compounds, which have been associated with a range of health benefits, including improved glycemic response, antioxidant effects, and gut health (Mironeasa et al. 2023). Beyond their biological value, plant-derived materials also influence the techno-functional properties of extruded products, contributing to aspects such as expansion, crispness, and color (Wójtowicz et al. 2019; Wang et al. 2019; Mohamad Mazlan et al. 2020).

Table 1. Comparative studies of the composition of different fruits, vegetables and legumes and their by-products

Plant material	**Findings**	**Reference**
Pomegranate (*Punica granatum*)	Higher phenolic compound content was found in the peel (150.6 mg GAE/g dry weight) compared to arils and seeds (5.8 mg GAE/g); antioxidant activity followed the same trend (46.24 vs. 0.8 mg Trolox/mL).	(Diamanti et al., 2017)
Jaboticaba (*Myrciaria cauliflora*)	Jaboticaba pomace showed total and insoluble dietary fiber contents of 20.54 and 16.42 g/100 g, respectively (2.2-fold higher than whole fruit), and phenolic compound content of 43.39 mg GAE/g (2.5-fold higher than whole fruit).	(Gurak et al., 2014)
Banana (*Musa paradisiaca*), mamey sapote (*Pouteria sapota*), mango (*Mangifera indica*), Meyer lemon (*Citrus meyeri*),	Fruit peels presented higher total fiber levels than pulps, especially fruits rich in pectin.	(Garcia-Amezquita et al., 2018)

Table 1. Comparative studies of the composition of different fruits, vegetables and legumes and their by-products

Plant material	Findings	Reference
orange (*Citrus sinensis*), prickly pear (*Opuntia ficus-in-dica*), tamarind (*Tamarindus indica*), and watermelon (*Citrullus lanatus*)		
Citrus fruits (lemon, orange and grapefruit)	Peels contained higher amounts of phenolics, flavonoids, vitamin C, and antioxidant activity than pulp and seeds; grapefruit peel had the highest phenolic content (77.3 mg GAE/g), followed by lemon (49.8 mg GAE/g) and orange (35.6 mg GAE/g).	(Sir Elkhatim et al., 2018)
Baru (*Dipteryx alata Vog*.)	Peel exhibited the highest dietary fiber content (24.1 g/100 g), followed by pulp (18 g/100 g) and roasted almond (16 g/100 g); raw almond had the highest phenolic content (1,107.0 mg GAE/100 g).	(Santiago et al., 2018)
Broccoli (*Brassica oleracea*)	Pomace powders showed higher fiber content, but lower total carbohydrate levels compared to whole-vegetable powder.	(Ying et al., 2021)
Onion (*Allium cepa* L.)	Total flavonoid and phenolic contents were significantly higher in onion peel and roots compared to bulbs; caffeic acid and quercetin were predominant in the peel, while methyl gallate was more abundant in the bulb.	(Kim et al., 2024)
Lentil (*Lens culinaris* Medik)	Phenolic compounds with antioxidant activity preferentially accumulated in hulls over cotyledons; delphinidin and cyanidin dominated in hulls, while epicatechin and catechin prevailed in cotyledons.	(Manco et al., 2023)

Numerous studies have investigated the incorporation of these plant-derived ingredients into extruded snacks, revealing their potential to support the formulation of functional products. For example, fruits such as açaí (Lucas et al., 2022), goji berries (Ménabréaz et al., 2021), chokeberry (Wójtowicz et al., 2023), tomato (Wójtowicz et al., 2018), elderberry or strawberries (Wójtowicz et al., 2019), mango and passion fruit pulps (Sarmiento-Torres et al., 2025) have been successfully added to corn or rice-based snacks. These additions have enchanced phenolic compound content and antioxidant activity while also modifying expansion and color characteristics. Vegetables such as broccoli, kale, carrot, and spinach have been incorporated into extruded snacks, either in fresh form or as dehydrated powders, to enhance their nutritional value and antioxidant profile (Bisharat et al., 2013, 2015; Kasprzak et al., 2018; Mitrus et al., 2023; Shevkani et al., 2019; Ying et al., 2021).

Legume-based ingredients, such as chickpea seed coats (Bresciani et al., 2023), mung bean protein (Bernin et al., 2024), common beans (Félix-Medina et al., 2021), and fenugreek (Wani et al., 2022), have been explored for their impact on nutritional value, antioxidant activity, and texture. Legumes are particularly appealing for their protein content and functional bioactives, contributing to the development of gluten-free or protein-enriched extruded snacks (Pasqualone et al., 2020).

Cereal-based additions and composite formulations have also been explored to enhance the nutritional and functional value of extruded snacks. Examples include jackfruit seed flour blended with nixtamalized maize (Juárez-Barrientos et al., 2025), amaranth–soycake–shallot blends (Omoba et al.,

2024), corn combined with cocoa (Ondo & Ryu, 2013) or sesame (Hashempour-Baltork et al., 2018), sorghum flour enriched with roasted coffee powder (Chávez et al., 2017), and rice extruded with turmeric powder (Ribeiro Oliveira et al., 2020), beans, or carob fruit (Arribas et al., 2019a, 2019b). These combinations often result in snacks with improved sensory acceptability, higher protein and fiber contents, and enhanced antioxidant properties.

In parallel with the incorporation of primary plant-derived ingredients, increasing attention has been directed toward the use of plant-based by-products in extruded snack formulations. These materials, generated from fruit, vegetable, cereal, and legume processing, have emerged as valuable sources of dietary fiber, phenolic compounds, and other bioactive components. Their use offers new opportunities to enhance the nutritional quality and functional properties of snacks while promoting sustainable food systems. Several studies have explored their integration, highlighting their role in functional snack development.

By-products from fruits have been extensively investigated for their enrichment potential. Tamarind shells have been incorporated as fiber-rich ingredients in extruded formulations, where extrusion processing enhanced the soluble dietary fiber content while retaining significant antioxidant capacity (Aguilar-Ávila et al., 2023). Similarly, fruit peel blends such as papaya and banana have been valorized through their inclusion in extruded macaroni products, providing additional sources of fiber and phytonutrients with acceptable sensory profiles (Mishra et al., 2022). Mango and papaya peels have also been successfully incorporated into corn-based extrudates. Their inclusion led to a significant increase in bioactive compounds such as catechins, mangiferin, and carotenoids, while maintaining favorable sensory acceptance and improving the bioaccessibility of phenolic compounds during digestion (Fontes-Zepeda et al., 2023). Other fruit-derived materials, such as tomato pomace, have similarly contributed to higher phenolic content and enhanced bioaccessibility of key antioxidants in extruded snacks (Yagci et al., 2022). Apple pomace, alone or in combination with oat or rice flour, has been incorporated to produce extruded snacks with high antioxidant potential and good retention of individual phenolic compounds despite processing at elevated temperatures (Hashemian et al., 2025; Leyva-Corral et al., 2016). Additionally, bilberry press cake has been successfully used to produce cereal-based extruded snacks with increased phenolic content and promising sensory acceptance (Höglund et al., 2018).

Vegetable-derived by-products have also demonstrated valuable applications. Black carrot pomace, for instance, has been utilized to fortify starch-based extruded snacks, resulting in significant enrichment in phenolic acids and antioxidant capacity (Uzun et al., 2025). Onion skin powder, a by-product rich in quercetin and fiber, has been incorporated into extruded wheat-based products. Its inclusion increased the levels of accessible phenolics and antioxidant activity without negatively impacting sensory properties (Tonyali et al., 2020). Similarly, the use of carrot pomace in extruded corn snacks has contributed to improved fiber content and β-carotene enrichment (Kaisangsri et al., 2016).

By-products from cereals, legumes, and other plant sources have further broadened the spectrum of fortification strategies. Brewer's spent grain, sugar beet pulp, and wheat bran have been utilized in expanded corn snacks, resulting in products with greater fiber content and antioxidant activity (Hashemian et al., 2025; Jozinović et al., 2021). Coffee silverskin, a by-product of coffee roasting, has demonstrated its potential as a source of fiber and protein when incorporated into extruded cereals (Beltrán-Medina et al., 2020).

Across these studies, the integration of fruit, vegetables, and cereal by-products consistently contributed to increasing the content of dietary fiber, phenolic compounds, and antioxidant capacity in extruded snacks. The variability in the origin and processing of these by-products highlights the

need for careful selection and optimization of extrusion conditions to balance nutritional enrichment with desirable sensory and structural properties. Collectively, these findings reinforce the role of plant-based by-products as valuable, sustainable ingredients for the development of functional extruded snacks aligned with health and environmental goals.

Overall, the incorporation of plant-derived ingredients, including both primary plant materials and by-products, into extruded snacks offers promising avenues for nutritional enhancement and functional food development. However, the successful integration of these ingredients into extruded matrices not only depends on their compositional attributes but also on their interactions with the extrusion process and with other matrix components. In this context, a deeper understanding of how plant-derived ingredients influence the physical, structural, and biochemical properties of extrudates is essential. The following sections address these aspects in detail, examining the impact of ingredient inclusion and processing on techno-functional properties, polysaccharide composition, and phenolic compound behavior.

7. Effect of processing and plant-derived ingredients inclusion on the physical and textural properties of extruded snacks

Physical and textural characteristics are crucial parameters when evaluating the quality of extruded products, as they significantly influence consumer acceptability (Alam et al., 2016). Extrusion parameters such as feed moisture, barrel temperature, and screw speed, along with indirect response parameters like specific mechanical energy (SME), torque, and melt temperature and pressure, are critical control points to ensure product quality (Ribeiro et al., 2024). Specifically, SME determines the extent of macromolecular transformations and the rheological behavior of the mixture during processing (Ondo & Ryu, 2013). The incorporation of plant-derived ingredients often affects SME, which in turn impacts the final product's physical characteristics. Several studies have reported a reduction in SME with increasing inclusion levels of plant-derived materials (Mohamad Mazlan et al., 2020; Pitts et al., 2016; Shevkani et al., 2019), directly influencing properties such as expansion, bulk density, color, hardness, and crispness. In particular, fiber addition tends to reduce expansion volumes, increase product density, harden texture, and decrease crispness (Robin et al., 2012; S. Wang et al., 2019).

Table 2 summarizes the effects of incorporating various plant-derived ingredients and processing conditions on the physical and textural attributes of extruded snacks. Expansion is a highly desirable trait in extruded snacks, as consumers associate expanded structures with lightness and superior eating quality (Yagci et al., 2022). Expansion, generally expressed as the ratio of the extrudate diameter to the die diameter, reflects the extent of puffing as the product exits the extruder. Higher expansion results in increased surface area, larger pore diameters, and greater overall porosity (Ačkar et al., 2018; Aghajanzadeh et al., 2024; Ali et al., 2024; Aussanasuwannakul et al., 2022; Falfán Cortés et al., 2014; Mitrus et al., 2023; Neder-Suárez et al., 2024; Ribeiro Oliveira et al., 2020; Yagci et al., 2022; Ying et al., 2021). Expansion behavior is influenced by multiple factors. An increase in barrel temperature enhances the superheating of internal water, promoting steam formation and greater expansion. Conversely, higher feed moisture tends to plasticize the melt, reducing dough elasticity, limiting superheating, and thereby decreasing expansion and increasing density (Alam et al., 2016; Ali et al., 2024).

Consistently, most studies report that increasing the inclusion level of plant-derived ingredients, whether fruits (Blejan et al., 2025; Falfán Cortés et al., 2014; Hashemian et al., 2025; S. Wang et al., 2019; Wójtowicz et al., 2019), vegetables (Mitrus et al., 2023; Yagci et al., 2022; Ying et al., 2021), cereals, or legumes (Ačkar et al., 2018; Aussanasuwannakul et al., 2022; Neder-Suárez et al., 2024), leads to a decrease in expansion parameters. Consequently, bulk density tends to increase across

nearly all cases analyzed. Bulk density is a critical property, influencing not only packaging material requirements and costs but also the consumer's perception of product freshness, volume, and mouthfeel (Ali et al., 2024). Products with higher density generally exhibit a more compact and firmer texture, which, if not carefully controlled, can negatively impact consumer acceptance, as minimum hardness and high crispness are highly preferred (Bisharat et al., 2013; Varsha & Mohan, 2016).

A clear trend observed is the increase in hardness with higher inclusion levels of plant-derived ingredients (Ačkar et al., 2018; Hashemian et al., 2025; Wójtowicz et al., 2019; Yagci et al., 2022) often accompanied by a reduction in crispness (Aussanasuwannakul et al., 2022) or fracturability (Ačkar et al., 2018; Blejan et al., 2025). Crispness, an essential quality attribute, is related to the rapid force drop during mastication, linked to the fracture propagation in brittle materials. When brittle extruded snacks are chewed, the rupture of the cellular structure generates an audible sound that enhances the sensory perception of crispness (Varsha & Mohan, 2016).

Regarding color, changes are driven by both the natural pigments (such as carotenoids, anthocyanins, and flavonoids) introduced by plant ingredients (Aussanasuwannakul et al., 2022), and by reactions occurring during extrusion. These include pigment degradation, hydrolysis, oxidation, and non-enzymatic browning reactions like caramelization and the Maillard reaction, all of which substantially influence the final color (Ačkar et al., 2018; Aussanasuwannakul et al., 2022; Falfán Cortés et al., 2014). Consequently, reductions in lightness and increases in redness (a*) and yellowness (b*) values, reported in multiple studies, reflect a combination of pigment transfer and chemical transformations during processing.

Although parameters like the water absorption index (WAI) and water solubility index (WSI) are influenced in some formulations (Neder-Suárez et al., 2024; Yagci et al., 2022), their impact on the perceived quality of extruded snacks is generally less critical than changes in expansion, density, texture, and color.

Overall, the evidence indicates that while the inclusion of plant-derived ingredients effectively enhances the nutritional profile of extruded snacks, it inevitably affects their physical and textural properties. Thus, achieving successful functional products requires a balanced strategy that integrates both nutritional enrichment and preservation or improvement of structural and sensory quality. A comprehensive understanding of formulation and processing interactions is crucial to develop extruded snacks that satisfy both health and consumer acceptance demands.

Table 2. Effect of processing and plant-derived ingredients on the physical and textural properties of extruded snacks

Evaluated ingredient	Inclusion levels	Extrusion parameters	Findings	Reference
Fruits and vegetables-derived ingredient				
Bilberry pomace powder (BPP)	0, 2, 4, and 6% of total mixture (cornmeal base)	T: 180 °C FM: 14% SS: 250 rpm	↑BPP → slight ↑H, ↓cohesiveness, gumminess, resilience, chewiness, Fr 2% BPP → ↓ER; 4–6% BPP → ER ≈ control ↑BPP → ↑BD (at 4–6%) ↑BPP → ↓WAI, ↑WSI ↑BPP → ↓lightness, ↑redness, ↓yellowness, ↓hue angle, ↑ΔE > 5, ↓chroma (at 2–4%), ↑chroma (at 6%)	(Blejan et al., 2025)
Cranberry (CP), blueberry (BP), grape (GP) and apple (AP) pomaces	0, 50, 150, 300 g of pomace /Kg mixture (corn starch base)	T in the barrel: 140°C FM: 150 and 200 g/Kg SS: 150-250 rpm	↑IL →↓ER ER values: AP > CP > BP > GP (at 200 g/kg moisture; not consistent at 150 g/kg) TDF and IDF negatively correlated with expansion; SDF positively correlated at 300 g/kg pomace	(S. Wang et al., 2019)
Passion fruit pulp	0, 1.4, 3.5, 5.5, 7% of total mixture (corn starch base)	T (in third zone extruder): 80-110 °C FM: 16-30 %	↑T+↓FM+↓IL → ↑ER, ↓PF ↑ER → ↓PF FM x IL interactions influenced color parameters	(Falfán Cortés et al., 2014)
Dried black elderberry, chokeberry and strawberry	0, 10, 15, 20% of total mixture (corn grits base)	T: 132 °C (section I), 142 °C (section II), 135 °C (forming die) FM: 14% SS: 80 and 120 rpm	↑IL → ↓ER, ↑BD ↑IL → ↓lightness, ↑redness IL >15% → ↑H beyond desired level	(Wójtowicz et al., 2019)
Apple pomace (AP) and wheat bran (WB)	50% WB:AP (25:75, 50:50 or 75:25) + oat flour:broken rice (1:1)	FM: 14-22% SS: 120-200 rpm	↑IL of AP, ↑SS, ↑FM → ↓ER ↑IL of AP → ↑BD AP/WB supplementation → No effect on H ↑SS (120 to 160 rpm) → ↑H SS > 160 rpm → ↓H ↑IL of AP → ↑lightness, ↓redness, ↓yellowness	(Hashemian et al., 2025)

Table 2. Effect of processing and plant-derived ingredients on the physical and textural properties of extruded snacks

Evaluated ingredient	Inclusion levels	Extrusion parameters	Findings	Reference
			↑Screw speed → ↓lightness, ↑redness, ↑yellowness	
Freeze-dried carrot and broccoli powders	100:0, 80:20, 60:40, 40:60, 20:80, 0:100 vegetable powder:rice flour ratio	T from feed to die: 30, 60, 100, 140 °C FM: 20% SS: 230 rpm	↑IL → ↓ER	(Ying et al., 2021)
Fresh broccoli	0, 10, 20, 30% of total mixture (potato base)	FM: 32-36 % SS: 60, 80, 120 rpm	↑IL → ↓ER ↑SS → ↑ER ↑FM (at 20% broccoli) → ↓ER 32% moisture + 100 rpm SS (control, no broccoli) → highest BD 36% moisture + 60 rpm SS + 20% broccoli → lowest BD ↑SS (at 10% broccoli) → ↓BD ↑SS (at 20 to 30% broccoli) → ↑BD	(Mitrus et al., 2023)
Tomato pomace powder	0, 5, 10, 15, 20% (d.m.) of total mixture (cereal mix base: 45% corn semolina, 45% wheat semolina, 10% corn starch (d.m.))	T from feed to die: 40, 50, 70, 90, 100, 130°C FM: 18% SS: 450 rpm	↑IL → ↓ER, ↑BD, ↑H, ↓WAI, ↓WSI, ↓lightness, ↑redness	(Yagci et al., 2022)
Cereal, legume and other plant-derived ingredients				
Brewer's spent grain (BSG), sugar beet pulp (SBP), and apple pomace (AP)	0:100, 5:95, 10:90 and 15:85 by-products:corn grits ratio	T: 135 °C (dosing zone), 170 °C (compression zone) and 170 °C (ejection zone) SS: 100 rpm	↑IL → ↓ER, ↑BD, ↑H, ↓Fr	(Ačkar et al., 2018)
Soybean residue (Okara)	Okara (0-50% w/w), mung bean (20-70% w/w), and rice (20-80% w/w) mixture	T from feed to die: 37, 55, 121, 132, 164, 111 °C FM: 15-17% SS: 400 rpm	↑IL → ↓lightness, ↑redness, ↓ER, ↑BD, ↓Cr	(Aussanasuwannakul et al., 2022)
Black bean flour	11, 22, and 33% black bean flour (based on blue corn:spinach 99:1 mixture)	T: 80 °C (Feed zone), 122 °C (cooking zone), 65 °C (third section) FM: 29% SS : 255 rpm	↑IL → ↓ER slightly, ↑BD, no differences in WAI, ↑WSI, ↑H	(Neder-Suárez et al., 2024)

Table 2. Effect of processing and plant-derived ingredients on the physical and textural properties of extruded snacks

Evaluated ingredient	**Inclusion levels**	**Extrusion parameters**	**Findings**	**Reference**
Turmeric powder	0, 2, 4, 6, 8, 10% w/w (broken rice grains base)	T: 41 °C (section I), 61 °C (section II), 84 °C (section III) FM: 12.5% SS: 3600 x g	↑IL → ↓ER, ↓SV, ↑H, ↑redness and ↑yellowness Lower lightness vs commercial snack (darker)	(Ribeiro Oliveira et al. 2020)

Processing variables abbreviations: **IL**: Inclusion level of the evaluated ingredients; **T**: Temperature; **FM**: Feed moisture; **SS**: Screw speed.
Physical and textural properties abbreviations: **ER**: Expansion-related variables (Expansion Ratio; Expansion Index; Radial Expansion Index); **BD**: Bulk density; **WAI**: Water absorption index; **WSI**: Water stability index; **SV**: Specific volume; **PF**: Penetration force; **H**: Hardness; **Fr**: Fracturability; **Cr**: Crispness.
Other abbreviations not defined within the table: **TDF**: Total dietary fiber; **IDF**: Insoluble dietary fiber; **SDF**: Soluble dietary fiber

8. Effect of extrusion on polysaccharides constituents: Starch and dietary fiber

Extrusion cooking imposes intense thermal and mechanical energy on plant-based matrices, leading to significant modifications in their polysaccharide components. In extruded products enriched with fruits, vegetables, legumes, cereals, or other plant ingredients, both non-starch polysaccharides (dietary fibers) and starch undergo substantial chemical and structural transformations. The process tends to convert a portion of insoluble dietary fiber into soluble forms, while simultaneously disrupting the crystalline structure of starch granules, resulting in important nutritional and functional consequences (Cotacallapa-Sucapuca et al., 2021; X. Huang et al., 2022; Robin et al., 2012).

During extrusion, starch undergoes severe thermal and shear stress, which disrupts hydrogen bonds between polymer chains, particularly in the highly branched amylopectin fraction (X. Huang et al., 2022). Studies using purified starches, such as genetically modified maize and potato starch, have shown significant reductions in molecular weight after extrusion, with preferential cleavage of long amylopectin chains and a narrowing of the molecular size distribution (dos Santos et al., 2019; M. Li et al., 2014; W. C. Liu et al., 2010). Although these analyses were conducted on isolated starches, they provide a mechanistic basis for understanding starch fragmentation in extruded matrices containing plant-based ingredients, even though such analyses (e.g., via HPSEC-MALLS) remain limited in composite systems.

Nutritionally, extrusion increases starch digestibility by gelatinizing the granules and converting semi-crystalline regions into amorphous ones, which are more accessible to enzymatic hydrolysis (Qiu et al., 2024). Consequently, this enhances the glycemic potential of extruded products. However, the inclusion of plant-derived ingredients has shown promise in modulating this effect. For instance, the incorporation of grape pomace, amaranth-shallot-soycake blends, watermelon seed flour, and optimized cereal–legume combinations has led to extruded snacks with significantly lower glycemic indices. These outcomes highlight the capacity of certain plant constituents (e.g., phenolic compounds, fiber) to delay starch digestion and glucose release (Oladiran & Emmambux, 2018; Omoba et al., 2024; Renoldi et al., 2021; Sanusi et al., 2023; Wani et al., 2021).

At a physicochemical level, extrusion reduces starch crystallinity by transforming its semi-crystalline native granules into amorphous forms through gelatinization and mechanical fragmentation (X. Huang et al., 2022). This transition is reflected in lowered gelatinization enthalpy and melting temperatures, as observed through DSC analysis in composite snacks based on barley, rice–bean blends, pineapple stem starch, and mushrooms (Altan et al., 2009; Neder-Suárez et al., 2024; Tangsrianugul et al., 2023). Additionally, extrusion promotes the formation of amylose–lipid complexes, evidenced by V-type crystalline patterns in X-ray diffraction studies, which can influence textural behavior and retrogradation during storage (X. Huang et al., 2022).

In parallel to starch transformations, the extrusion process also induces profound alterations in dietary fiber. A recurrent outcome across studies is the partial solubilization of fiber components, which shifts the balance between insoluble and soluble fractions and significantly alters functional behavior. Redgwell et al. (2011) reported that high specific mechanical energy (SME > 400 kJ/kg) applied to citrus fiber during extrusion led to 30% solubilization, while lower SME values (∼200 kJ/kg) achieved only 8–12% solubilization and induced a broad spectrum of viscosity responses. Similarly, extrusion of lemon residues increased the soluble fiber content from 38.6% to over 50% under conditions of high temperature and low moisture (Méndez-García et al., 2011).

Modifications at the polysaccharide level have been documented as well. In chokeberry pomace, extrusion altered the distribution of pectic polysaccharides, specifically rhamnogalacturonan I, between soluble and insoluble fiber fractions (Schmid et al., 2021). Arabinans in apple pomace were

highly sensitive to thermo-mechanical stress, while xylans proved more resistant. Notably, the degree of methylation of pectins declined from 50% to 15% after extrusion, which could affect gelling behavior and fiber–matrix interactions (Schmid et al., 2020).

These compositional shifts are often accompanied by improvements in hydration and emulsification properties. Studies on tamarind Shell (Aguilar-Ávila et al., 2023), oat bran (M. Zhang et al., 2011), lotus root nodes (H. Chen et al., 2018), and lupin kernel fiber (Naumann et al., 2021) have shown that extrusion increases soluble fiber content and enhances water solubility, swelling capacity, emulsifying potential, and viscosity.

Structurally, these changes reflect partial degradation of cellulose and hemicellulose and reductions in polymer size, as revealed by FTIR, GC, and SEM analyses (H. Chen et al., 2018). The resulting fiber fractions are often more fragmented and exhibit greater solubility but may have diminished water retention and binding capacity (Redgwell et al., 2011; Schmid et al., 2020), factors that are critical for maintaining texture and structural cohesion in extruded formulations.

Overall, the structural and compositional transformations experienced by both starch and dietary fiber during extrusion play a central role in shaping the nutritional, functional, and sensory attributes of extruded snacks. These changes are particularly relevant in formulations enriched with plant-derived ingredients, where the interplay between process conditions and matrix composition determines not only the digestibility and glycemic impact of starch but also the hydration capacity, solubility, and techno-functional behavior of the fiber fraction.

9. Effect of extrusion on phenolic compounds in extruded snacks

The incorporation of plant-derived ingredients into extruded snacks has been widely explored as a strategy to improve the content of phenolic compounds in cereal-based matrices. Numerous studies have demonstrated that the inclusion of materials such as fruit pomace, vegetable powders, and press cakes contributes to the enrichment of extruded products with total and individual phenolic compounds, even when the effect of extrusion itself has not been explicitly assessed. For example, the addition of bilberry press cake to rye flour extrudates led to increased total phenolic content in the final product (Höglund et al., 2018). Similarly, the inclusion of Jerusalem artichoke, amaranth, and pumpkin flours enriched corn-based snacks with phenolic compounds and carotenoids (Kolniak et al., 2017). Other studies include tomato pomace, blackcurrant pressings, kale, goji berries, and apple pomace, which consistently enhanced phenolic content and antioxidant activity compared to control snacks (Drozdz et al., 2014, 2019; Kasprzak et al., 2018; Ménabréaz et al., 2021; Reis et al., 2014; Yagci et al., 2022).

While such formulations demonstrate the potential of extrusion to yield phenolic-enriched products, studies that compare phenolic content before and after extrusion provide more direct insights into the effects of the process on phenolic stability and transformation. Historically, extrusion has been associated with a reduction in phenolic compound content, often attributed to thermal degradation, oxidation, or polymerization under high temperature and shear conditions (Brennan et al., 2011; Wani & Kumar, 2016). However, increasing evidence reveals that the impact of extrusion on phenolic compounds is more complex, showing variability based on compound structure, process parameters, and the matrix composition (Ménabréaz et al., 2021; Wójtowicz et al., 2018).

Several mechanisms have been proposed to explain both the degradation and release of phenolic compounds during extrusion. Thermolabile compounds such as anthocyanins are prone to degradation at high die temperatures and low moisture, while more stable phenolic acids like ferulic or chlorogenic acid may resist thermal breakdown or even increase in extractable concentration due

to matrix disruption (Leyva-Corral et al., 2016; Morales et al., 2015). Structural breakdown of lignocellulosic material under thermal and mechanical stress can promote the release of cell wall-bound phenolics, including hydroxycinnamic acids, often esterified to arabinoxylans or lignin (Cervantes-Ramirez et al., 2022; Leyva-Corral et al., 2016; Ruiz-Armenta et al., 2019; Shahidi & Yeo, 2016).

Indeed, the response of phenolic compounds to extrusion is highly compound-specific. Anthocyanins often show significant losses, as reported by Arribas et al. (2019a), who found reductions of up to 50% in extruded rice–bean–carob blends, whereas flavonols increased nearly threefold in the same formulations. Similarly, Ménabréaz et al., (2021) observed a 40% decrease in rutin content in goji berry-enriched extrudates, but the authors noted that bioaccessibility remained stable, suggesting that the transformation may not impair functionality. Studies such as that by Morales et al. (2015) documented a five-fold increase in total phenolics and significant rises in hydroxybenzoic and hydroxycinnamic acids in lentil-based extrudates, explained by the cell wall hydrolysis in the case of fiber-bound phenolic compounds. Additionally, extrusion has shown the potential to modify the polymerization profile of proanthocyanidins (PACs), shifting the distribution from poorly absorbed high molecular weight polymers toward more bioavailable monomers and dimers. In grape seed and pomace, extrusion significantly increased the content of these low-molecular-weight PACs, with monomer levels rising by up to 120% and dimers by 80%, concurrent with a reduction of up to 54% in polymeric forms (Khanal et al., 2009b). Similar effects were observed in blueberry pomace, where extrusion enhanced the levels of monomers, dimers, and trimers, especially under high-temperature, low-shear conditions (Khanal et al., 2009a). These transformations are attributed to the breakdown of interflavan bonds under thermal and mechanical stress, promoting the release and extractability of smaller PACs fractions, which are considered more biologically active and better absorbed in the human gastrointestinal tract (Qi et al., 2016).

The results compiled in Table 3 reinforce this dual nature of extrusion effects. While some studies confirm the degradation of phenolic compounds, others report increases in either free or total content. In extrudates containing fermented and non-fermented Jabuticaba pomace, brewer's spent grain, sugar beet pulp, apple pomace, and artichoke leaf, total phenolic contents decreased, likely due to thermal decomposition (Asquieri et al., 2021; Guven et al., 2018; Jozinović et al., 2021; Leyva-Corral et al., 2016). Conversely, in extrudates with tomato pomace, increases of up to 118% in total phenolic content were reported, attributed to the release of bound compounds during processing (Wójtowicz et al., 2018).

Moreover, changes in the phenolic profile are not limited to concentration shifts. Chemical modifications during extrusion can convert compounds into more or less active derivatives. For example, chlorogenic acid may be degraded into caffeic and ferulic acids, altering antioxidant activity (Chávez et al., 2017). Likewise, heat-induced decarboxylation of phenolic acids can reduce functionality, but in some cases, these reactions also enhance extractability from the matrix (Morales et al., 2015).

Matrix composition is another key determinant of phenolic fate during extrusion. Ingredients rich in dietary fiber, such as cereal bran, pomaces, or leafy vegetables, may contain phenolic compounds associated with cell wall components through a range of interactions. In cereal-based materials or lignified tissues, phenolic acids such as ferulic and p-coumaric acids are commonly esterified to arabinoxylans or ether-linked to lignin (Cervantes-Ramirez et al., 2022; Nguyen & Beta, 2024; Santos-Zea et al., 2018; Shahidi & Hossain, 2023; Shahidi & Yeo, 2016; Tse & Schendel, 2023). These covalent bonds can be disrupted under high temperature and shear, increasing extractability (Cheng et al., 2024; Ménabréaz et al., 2021; Morales et al., 2015). In contrast, in fruit- or vegetable-based matrices with lower lignin content, phenolic compounds are predominantly retained through

non-covalent associations, such as hydrogen bonding or hydrophobic interactions, with polysaccharides or proteins (Fernandes et al., 2023; Le Bourvellec et al., 2011; X. Liu et al., 2020; Shahidi & Hossain, 2023; X. Zhang et al., 2022). These interactions may also be weakened during extrusion, contributing to the release of phenolic compounds. In addition, differences in phenolic compounds behavior can be observed even among cultivars of the same plant species. For instance, Arribas et al. (2019a) reported a decrease in anthocyanins and an increase in flavonols in extruded rice–bean–carob blends. However, when the same processing conditions were applied using a different bean cultivar, anthocyanin content increased while flavonol levels remained unchanged (Arribas et al., 2019b), highlighting the influence of varietal differences on the phenolic response to extrusion.

Furthermore, the fractionation of phenolics into free and bound forms is gaining importance in extrusion research. Several studies indicate that while free phenolics may decrease during extrusion, the content of bound phenolics increases, or vice versa (Chávez et al., 2017; Félix-Medina et al., 2020, 2021; Ruiz-Armenta et al., 2019). Such changes underscore the need to analyze both fractions to fully understand the functional impact of extrusion.

The effect of extrusion on phenolic compounds in plant-enriched snacks is multifactorial and highly dependent on the specific system. Beyond the composition of the food matrix and the structural characteristics of phenolic compounds, processing parameters, such as temperature, screw speed, and moisture content, play a decisive role in their stability, transformation, or release. A growing body of evidence indicates that extrusion can not only degrade but also enhance the extractability and functional availability of phenolics, particularly those initially bound to cell wall components. Given this complexity, several studies have aimed not only to understand the general effects of extrusion on phenolic compound content but also to optimize processing conditions to maximize phenolic retention while maintaining acceptable techno-functional properties in extruded snacks containing plant-derived ingredients. To this end, response surface methodology (RSM) and factorial experimental designs are commonly employed to evaluate the influence of key variables such as feed moisture, extrusion temperature, and screw speed.

Among the optimization strategies reported in the literature (Table 4), RSM, particularly through central composite design (CCD), has been the most widely used approach. However, some studies have adopted alternative designs, such as Box-Behnken (Lohani & Muthukumarappan, 2017b, 2017a) or full factorial schemes (Promsakha na Sakon Nakhon et al., 2018). While the optimization goals vary across studies, a consistent trend is the prioritization of physical and textural properties, such as expansion ratio, bulk density, and hardness, over the direct maximization of individual phenolic compounds. This reflects the importance of ensuring not only the nutritional quality but also the structural and sensory integrity of the final product.

From the perspective of phenolic content, several studies have shown that increased extrusion temperature does not universally result in phenolic degradation (Félix-Medina et al., 2020; Medina-Rendon et al., 2023; Neder-Suárez et al., 2024; Samyor et al., 2018). In certain matrices, higher temperatures, particularly when combined with lower feed moisture or specific screw speeds, have been associated with increased total phenolic content (TPC), likely due to enhanced matrix disruption and release of bound compounds. These findings highlight the matrix-specific nature of phenolic compound behavior and caution against generalizing processing recommendations across diverse plant ingredients.

In most optimization frameworks, TPC remains the predominant indicator of bioactive compound retention. Although individual phenolics (e.g., cyanidin-3-glucoside, caffeic acid, or ferulic acid) are often profiled (Lohani & Muthukumarappan, 2017a; Medina-Rendon et al., 2023), they are rarely

included as response variables in the optimization process. This preference for TPC is largely due to its practicality as a global indicator of phenolic retention and antioxidant potential. Nevertheless, for applications targeting specific bioactivities or health outcomes, future research could benefit from integrating detailed phenolic compound profiling into multi-objective optimization strategies, allowing for more precise modulation of the functional composition of extruded snacks.

Ultimately, in optimizing processing conditions for plant-based extruded snacks, it is essential to recognize that the inclusion of bioactive-rich ingredients must not come at the expense of techno-functional quality. Physical and textural characteristics influence consumer acceptability. Therefore, optimization must balance the functional potential of plant-derived ingredients with the quality demands of the final product. Given the inherent variability of plant matrices and their dynamic interactions with extrusion parameters, a more holistic and objective optimization approach is needed, one that simultaneously addresses physical, nutritional, and structural dimensions to develop snacks that are both health-promoting and sensorially desirable.

9.1 Effect of the extrusion process on the bioaccessibility of phenolic compounds

To exert their biological properties, phenolic compounds must be at least partially bioavailable in the tissues where they exert their physiological effects. Bioavailability refers to the fraction of an ingested compound that is absorbed and metabolized, and is closely related to bioaccessibility, the proportion of a compound that is released from the food matrix during digestion, becoming available for intestinal absorption through the action of digestive enzymes and gut microbiota. Only the fraction that is bioaccessible can potentially be bioavailable (Güven, 2016; Saura-Calixto et al., 2007).

According to Bohn (2014) several factors influence the bioavailability and bioaccessibility of phenolic compounds, including the administered dose, interactions with other food constituents, the kinetics of release (which differ between solid and liquid matrices), the structure of the food matrix itself, and the technological processes applied during food preparation.

Among these processes, extrusion can play a dual role depending on the matrix composition and process conditions (Salazar-López et al., 2018). On one hand, extrusion may cause the degradation of thermolabile phenolics and promote polymerization reactions that reduce compound stability. On the other hand, the intense thermal and mechanical energy applied can disrupt plant cell walls and cleave covalent bonds linking phenolics to dietary fiber, thereby increasing their release and extractability. The overall effect on bioaccessibility depends on the balance between degradation and release (Reyes Moreno et al., 2018; T. Wang et al., 2014a).

Although still limited, several studies have explored the effect of extrusion on the *in vitro* bioaccessibility of phenolic compounds in snacks enriched with plant-derived ingredients. For instance, Guven et al. (2018) reported enhanced bioaccessibility of cynarin and cinaroside in extrudates formulated with wheat flour and artichoke leaf powder, compared to their unextruded counterparts. Schmid et al. (2020) using a simulated extrusion model on chokeberry pomace powder, found that short treatments (1 min) at temperatures up to 160 °C preserved bioaccessibility, whereas extended treatments (20 min) at the same temperature led to a 14% reduction. Despite observed losses in total phenolic content, particularly anthocyanins, the overall bioaccessibility remained largely unaffected. In a subsequent study, Schmid et al. (2022) evaluated the same ingredient incorporated into extruded corn starch snacks and noted that, although phenolic acids and flavonols were relatively stable, anthocyanin bioaccessibility was highly sensitive to extrusion parameters, especially screw speed. Similarly, Herrera-Cazares et al. (2021) observed decreased bioaccessibility of xanthones and flavonoids, but increased release of phenolic acids, in extruded mango bagasse–corn starch snacks. These findings are consistent with other studies showing that the bioaccessibility of phenolic

compound subgroups is highly dependent on both compound structure and extrusion conditions (G. Liu et al., 2019; Ménabréaz et al., 2021; Tonyali et al., 2020; Yagci et al., 2022).

Overall, the modifications induced by extrusion, including structural changes, oxidation, and matrix disruption, combined with the intrinsic characteristics of the phenolic compounds and their interactions with other food components, significantly influence their stability, bioaccessibility, absorption, and ultimately, their bioavailability (Chandrasekara & Shahidi, 2012; Güven, 2016).

9.2 Interaction of phenolic compounds with other components in extruded products

The interactions between phenolic compounds and other food matrix components, such as proteins, carbohydrates, and lipids, in extruded snack formulations depend on the physical, chemical, and molecular properties of each constituent, which may be altered during processing (Nicolás-García et al., 2021; C. M. G. C. Renard et al., 2017).

Regarding phenolic compound–protein interactions, most evidence suggests a reducing effect on the bioaccessibility and bioavailability of phenolic compounds (Pinarli et al., 2020). These interactions primarily involve reversible non-covalent mechanisms such as hydrophobic interactions and hydrogen bonding, which may later stabilize the association. However, irreversible covalent interactions can also occur, particularly between oxidized phenolic compounds (e.g., flavonoid-derived quinones) and nucleophilic amino acid residues or enzymes (Jakobek, 2015; Kardum & Glibetic, 2018; Lund, 2021). The nature and strength of these interactions are highly dependent on the molecular weight and structural flexibility of the phenolic compounds. Larger, less flexible phenolic compounds often bind more strongly to specific proteins, while smaller or more flexible ones may interact with a wider range of protein targets (Kardum & Glibetic, 2018). Additional factors such as protein physicochemical characteristics, the phenolic compound-to-protein ratio, and synergistic or antagonistic interactions among phenolic compounds can also influence binding. Moreover, external conditions, including pH, temperature, ionic strength, and the presence of other food components, play a critical role in modulating these interactions (Q. Zhang et al., 2020).

Phenolic compounds also interact with carbohydrates through non-covalent forces such as hydrogen bonding, hydrophobic interactions, and ionic interactions (Giuberti et al., 2020; Le Bourvellec & Renard, 2012; X. Liu et al., 2020). These interactions are highly influenced by the molecular size of phenolic compounds and hydrophilicity, as well as the structural complexity of the carbohydrate (D. Amoako & Awika, 2016). Depending on the specific phenolic–carbohydrate complex formed, such interactions may enhance or inhibit phenolic bioaccessibility and bioavailability (Pinarli et al., 2020). In many cases, phenolic compounds associated with carbohydrates may reach the colon intact, where microbial enzymes can release them, contributing to local antioxidant activity and microbial modulation (Jakobek, 2015; Palafox-Carlos et al., 2011; Siemińska-Kuczer et al., 2022). Furthermore, certain polyphenol–carbohydrate interactions have been shown to influence starch functionality by promoting the formation of slowly digestible or resistant starch fractions, which can lower postprandial glycemic responses (Debelo et al., 2020; Nag & Majumder, 2022; F. Zhu, 2015). Recent findings in model systems also suggest that phenolic compounds may form V-type inclusion complexes with starch, protecting them from degradation during processing and digestion and improving their recovery post-digestion (D. B. Amoako & Awika, 2019; Ferruzzi et al., 2020), which reinforces the relevance of starch–polyphenol interactions in functional food development.

Table 3. Effect of processing on the phenolic compound content in extruded snacks

Evaluated plant-derived ingredient	Inclusion levels	Extrusion parameters	Findings	Reference
Fermented and non-fermented Jabuticaba pomace	5-20% + base mix (lentil:rice flour 70 :30)	T from zone 2 to 6: 60, 80, 90, 90, 100 °C FM: 21% SS: 500 rpm	↓TPC	(Asquieri et al., 2021)
Brewer's spent grain, sugar beet pulp, apple pomace	5-15% d. w. (corn grits base)	T profile: 135/170/170 °C FM: 15%	↓TPC	(Jozinović et al., 2021)
Tomato powder	5-30% d. w. (corn grits base)	T in the barrel zone: 125/145/135 °C FM: 15% SS: 120 rpm	↑TPC (increase of 23 – 118%) ↓TPC in snacks without tomato	(Wójtowicz et al., 2018)
Naranjita (*Citrus mitis* B.) bagasse	8.02% + base mix (corn starch:corn flour 60:40 or corn starch:corn flour:milk powder 56.4:37.6:6)	T: 125 °C (cooking/mixing zone), 75 °C (feeding zone), 75 °C (die zone) FM: 23% SS: 75 rpm	↓TPC ↓FP fraction ↑BP fraction	(Ruiz-Armenta et al., 2019)
Onion skin powder	3 – 9% d.m. (wheat flour base)	T in the barrel zone: 70/80/130/150 °C FM: 20% SS: 250 rpm	↓TPC ↑Quercetin (increase of 2.5 – 119%)	(Tonyali et al., 2020)
Apple pomace	14% (+ Oat flour 60.2%, potato starch 25.8%)	T at the die end of the barrel: 104 – 175 °C FM: 21 – 30% SS: 180 rpm	↓TPC (79.9 – 97.1% retention) ↓Chlorogenic acid (57–71% retention) ↓Caffeic acid (55 – 64% retention) ↓*p*-Coumaric acid (38–51% retention) ↓Ferulic acid (25–28% retention) ↓Rutin (56–70% retention) ↓Phloridzin (46–76% retention)	(Leyva-Corral et al., 2016)
Whole carob fruit	5 and 10% + mix base (50-80% of rice + 20 or 40% of bean *Phaseolus vulgaris* var. Almonga)	T at the die end of the barrel: 125 °C Water addition rate: 2.5 – 3.2 Kg/h SS: 900-950 rpm	↑TPC (increase of 36%) ↑Anthocyanins (increase of 24%) Flavonols: no significant change	(Arribas et al., 2019a)
			↓TPC ↓ *p*-Coumanic acid derivative ↓ Tetragalloyl-glucose ↓ Myricetin-O-pentoside	(Arribas et al., 2019c)

Table 3. Effect of processing on the phenolic compound content in extruded snacks

Evaluated plant-derived ingredient	Inclusion levels	Extrusion parameters	Findings	Reference
			↓ Ellagic acid ↓ Quercetin-3-O-rhamnoside	
	5 and 10% + mix base (50-80% of rice + 20 or 40% of bean *Pisum sativum* var. Cartouche)		↑TPC (increase of 40 – 43%) ↓Anthocyanins (decrease of 4 – 50%) ↑Flavonols (~3-fold increase)	(Arribas et al., 2019b)
Lentil flour, wheat bran, apple fiber, corn fiber	Formulations with at least 68% of lentil flour (proportions not specified): Lentil flour + wheat bran + apple fibre Lentil flour + wheat bran + Nutriose® Lentil flour + apple fibre + Nutriose® Lentil + wheat bran + corn fiber	T: 160 °C (forming die) SS: 500 rpm	↑TPC ↑Hydroxybenzoic acids ↑Hydroxycinnamic acids ↓Flavonols	(Morales et al., 2015)
Common bean kernels	White maize + common bean kernels: 175g + 75g	T: 164 °C (in the barrel zone) FM: 18% SS: 187 rpm	*p*-Coumaric hexoside, Sinapic, Biochanin A: ↑in BP fraction *p*-Coumaric, Dimethoxy isoflavone: ↓in BP fraction Syringic hexoside, Methyl isoflavone isomer II, Diferulic: ↓in FP fraction, ↑in BP fraction Ferulic: no changes in FP fraction, ↓in BP fraction Kaempferol hexoside, Naringenin hexoside, Daidzein dihexoside: ↓in FP fraction, no change in BP fraction Methyl isoflavone isomer I: ↓in FP and BP fraction 2-Hydroxycinnamic: no change	(Félix-Medina et al., 2021)
Roasted coffee powder	10-20% (sorghum flour base from two genotypes)	T from feed to die: 60, 120, 140 °C FM: 16 and 20%	↑TPC (increase of 40-48%) ↓TPC in snacks without coffee	(Chávez et al., 2017)

Table 3. Effect of processing on the phenolic compound content in extruded snacks

Evaluated plant-derived ingredient	Inclusion levels	Extrusion parameters	Findings	Reference
		SS: 180 rpm	Sorghum Genotype 1: Gallic acid: ↑in FP fraction, ↓in BP fraction Vanillic acid, *p*-coumaric acid: ↑in FP and BP fractions Chlorogenic acid, Caffeic acid, Sinapic acid, *O*-coumaric acid: ↓in FP and BP fractions Syringic acid, Ferulic acid: ↓in FP fraction, ↑in BP fraction Sorghum Genotype 2: Gallic acid, Chlorogenic acid, Caffeic acid, *p*-coumaric acid, Ferulic acid, Sinapic acid, *O*-coumaric acid: ↓in FP fraction, ↑in BP fraction Vanillic acid: No change in FP fraction, ↓in BP fraction Syringic acid: ↑in FP and BP fractions	

T: Temperature, **FM**: Feed moisture, **SS**: Screw speed, **TPC**: Total polyphenol content, **FP**: Free phenolic compounds, **BP**: Bound phenolic compounds.

Table 4. Optimization of process conditions to obtain extruded snacks containing polyphenolic compounds

Raw materials	Optimized variables	Optimization goal	Experimental design/ Optimization method	Optimal conditions	Findings	Reference
Black bean, blue maize, chard	T: 102-142 °C (intermediate zone) SS: 96-171 rpm FM: 22.2-35.7%	TAC, TPC maximization Hue minimization	CCD/RSM	T: 120 °C SP: 111 rpm FM: 29%	↑T + ↓SS → ↓TAC ↓T + ↓SS → ↑TAC ↑T → ↓C3GC, P3GC, M3GC, P3-5DGC at low SS ↑T → ↓D3GR at low SS, slight ↑D3GR at high SS ↑T + ↓FM → ↑TPC ↓T at low FM + ↑FM at high T → ↑AA	(Neder-Suárez et al., 2021)
White corn flour, mango kernel flour, mango peel flour	T in the die: 100-130 °C SS: 80-120 rpm FM: 17-21%	H, WSI minimization WAI maximization	CCD/RSM	DT: 120.66 °C SS: 66.36 rpm FM: 21.88%	Two trends: ↓DT + ↓FM + ↓SS → ↑TPC (max: <90 °C, 16% FM, 75 rpm) ↑DT + ↑FM + ↑SS → ↑TPC (max: >135 °C, 22% FM, 125 rpm)	(Medina-Rendon et al., 2023)
Germinated brown rice, pumpkin flour	FM: 13, 16, 19% Pumpkin flour content: 10, 20, 30%	TPC, sensory attributes maximization	3 x 3 factorial/RSM	FM: 13-14% Pumpkin flour content: 10-13%	↑Pumpkin flour content + ↓FM→ ↑TPC retained in the extrudates	(Promsakha na Sakon Nakhon et al., 2018)
Corn flour, sorghum flour and apple pomace fermented and hydrodynamic cavited	T near the die section: 80, 110, 140 °C SS: 100, 150, 200 rpm FM: 25, 30, 35%	TPC, AA, ER, brittleness, crispness, WSI, IVSD, TDF maximization H and WAI minimization	Box-Behnken/RSM	T: 132 °C SS: 108 rpm FM: 25 %	↑T (80→140 °C) → ↓TPC (–14% at 10% apple pomace ratio) ↑Apple pomace ratio (10→30%) → ↑TPC (+57%) and ↑AA (+56%) regardless of T, ↑TPC (+47%) regardless of SS ↑SS (100→150 rpm) → ↓TPC (–20% at 10% apple pomace ratio) ↑T (80→140 °C) + ↑SS → ↓TPC (-11% at 100 rpm, -15% at 200 rpm) ↑SS (100→200 rpm) → ↓TPC at both 80 and 140 °C	(Lohani & Muthukumarappan, 2017a)

Table 4. Optimization of process conditions to obtain extruded snacks containing polyphenolic compounds

Raw materials	Optimized variables	Optimization goal	Experimental design/ Optimization method	Optimal conditions	Findings	Reference
Corn flour, sorghum flour, apple pomace	T near the die section: 80, 120, 160 °C SS: 100, 150, 200 rpm FM: 15, 20, 25%	TPC, AA, brittleness, crispness, WSI, TDF maximization H and WAI minimization	Box-Behnken/RSM	T: 97 °C SP: 100 rpm FM: 25%	↑T (80→160 °C) → ↓TPC (-29%) regardless of apple pomace ratio ↑ apple pomace ratio (10→30%) → ↑TPC (+41% at 80 °C; +31% at 160 °C) ↑ apple pomace ratio → ↑AA (+62% at 80 °C; +33% at 160 °C) ↑FM (15→25%) → ↑TPC (+20% at 80 °C); ↓TPC (-20% at 160 °C) ↑T at 25% FM → ↓TPC (-33%); no change at 15% FM	(Lohani & Muthukumarappan, 2017b)
Corn flour, common bean flour	T in the barrel: 130.1-170 °C SS: 50-201.5	ER, AA, TPC and MC maximization H minimization	CCD/RSM	T: 170 °C SS: 200 rpm (For 100% whole corn flour snacks) T: 159 °C SS: 214 rpm (For snacks containing 16% bean flour) T: 164 °C SS: 187 rpm (For snacks containing 30% bean flour) T: 163 °C SS: 165 rpm (For snacks containing 40% bean flour)	↑T + ↓bean flour content → ↑ER, AA, TPC, MC ↑SS → ↑ER, MC; no effect on AA, TPC ↑T + intermediate SS → ↓H	(Félix-Medina et al., 2020)
Corn grit, wheat flour, rice flour and margarine, defatted soybean meal,	T in zone 3: 160, 180, 200 °C SS: 140, 160, 180 rpm	Objective values of ER (3), BD (0.115 g/cm^3), H (340 N), SDF and IDF (3.5 and 2 % wb respectively),	CCD/RSM	T: 160 °C SS: 180 rpm FM: 13%	↑T → ↓TPC, ↑IC_{50}	(Korkerd et al., 2016)

Table 4. Optimization of process conditions to obtain extruded snacks containing polyphenolic compounds

Raw materials	Optimized variables	Optimization goal	Experimental design/ Optimization method	Optimal conditions	Findings	Reference
germinated brown rice meal, mango peel fiber	FM: 13, 15, 17%	TPC (10 mg GAE/g) IC_{50} (5 mg), sensory score for texture and overall acceptance (6 from 9 scale)				
Rice flour, passion fruit powder	T near the die section: 80-150 °C SS : 200-400 rpm FM : 20-30% Passion fruit content : 0-15%	TPC and DPPH maximization, maintain good ER and WAI	CCD/RSM	T : 97.5 °C SS : 250 rpm FM : 25.2% Passion fruit content : 11.25%	↑SS → ↑TPC (max at 300 rpm) → ↓TPC ↑T → ↑TPC (max at 110 °C) → ↓TPC	(Samyor et al., 2018)
Lentil flour, orange peel powder	T in the die: 130-170 °C SS: 150, 200, 250 rpm FM: 14-22%	ER, BD, Porosity, WAI, WSI maximization H minimization	CCD/RSM	DT: 150 °C SS: 200 rpm FM: 16%	↑FM → ↑TPC, ↑TFC at 130-150 °C; ↓TPC, ↓TFC at 170 °C ↑T + ↑FM → ↓TTC ↑T → ↓AA	(Rathod & Annapure, 2017)

T: Temperature, **SS**: Screw speed, **FM**: Feed moisture, **CCD**: Central composite design, **RSM**: Response surface methodology, **TPC**: Total polyphenol content, **TAC**: Total anthocyanins content, **TFC**: Total flavonoids content, **TTC**: Total tannins content, **AA**: Antioxidant activity, **H**: Hardness, **WSI**: Water solubility index, **WAI**: Water absorption index, **ER**: Expansion-related variables (General expansion, Expansion Ratio; Expansion Index), **IVSD**: *In* vitro starch digestibility, **TDF**: Total dietary fiber, **SDF**: soluble dietary fiber, **IDF**: Insoluble dietary fiber, **MC**: Melanodinin content, **C3GC**: Cyanidin-3-glucoside content, **P3GC**: Pelargonidin-3-glucoside content, **P3-5DGC**: pelargonidin-3-5 diglucoside content, **M3GC**: malvidin-3-glucoside content, **D3GR**: retention percentage of delphinidin-3-glucoside chloride

Polyphenolic compounds can also bind to lipids via hydrophobic, hydrogen, or covalent interactions. These interactions may provide antioxidant protection by limiting lipid oxidation and the formation of harmful oxidation products (Jakobek, 2015; Pinarli et al., 2020). Additionally, lipids can form protective micelles or colloidal structures that encapsulate phenolics during digestion, potentially enhancing their stability and absorption along the gastrointestinal tract (Pinarli et al., 2020; H. Zhang et al., 2014).

To study these complex interactions between phenolics and food macromolecules, a wide array of analytical techniques is employed to obtain both qualitative and quantitative data. A comprehensive understanding typically requires the combination of multiple analytical approaches, as summarized by various authors (Le Bourvellec & Renard, 2012; X. Liu et al., 2020; Q. Zhang et al., 2020; F. Zhu, 2018), and illustrated in Fig. 5.

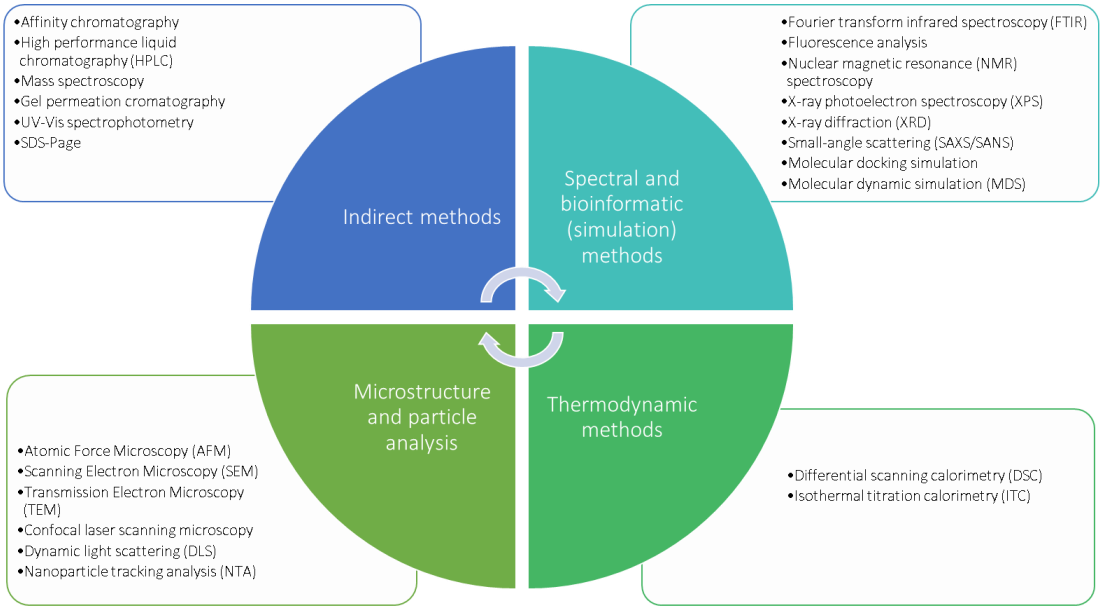

Fig. 5. Mains techniques used for analysis of interaction between phenolic compounds and food macromolecules

To date, most research on polyphenol–macromolecule interactions has been conducted using model systems, particularly focused on proteins (Anigboro et al., 2021; Chamizo-González et al., 2021; Francisco et al., 2021; Miao et al., 2015; Q. Zhao et al., 2020) and carbohydrates (D. B. Amoako & Awika, 2019; Camelo-Méndez et al., 2016; Guzar, 2012; Ribeiro de Barros, 2012; Sun et al., 2018; Xie et al., 2019; B. Zhao et al., 2018). However, investigations in real food matrices, and particularly in extruded products, remain limited due to the compositional and structural complexity of these systems. Nevertheless, a growing body of research has begun to address these interactions in processed food matrices (Table 5). For instance, Ying et al. (2017) showed that the incorporation of olive pomace into extruded rice- and maize-based blends not only enriched the products in fiber and phenolic compounds but also induced conformational changes in carbohydrate and protein structures, as revealed by FTIR analysis. These modifications, in turn, influenced the physical properties of the extrudates and suggested potential interactions between phenolics and other matrix components. Similarly, Sayanjali, (2016) demonstrated that oat fiber, particularly its β-glucan and protein fractions, can interact with curcuminoids, enhancing their solubility and stability during extrusion. Spectroscopic analysis confirmed these interactions, which likely involved both protein and

carbohydrate domains, and resulted in improved curcuminoid retention (~90%) under optimized processing conditions.

Studies by Zeng et al. (2021, 2022) further explored the role of phenolic compounds in modulating starch properties. The introduction of catechins and proanthocyanidins into extruded starch systems inhibited short- and long-term retrogradation, reduced relative crystallinity, and altered rheological behavior, pointing toward direct phenolic–starch interactions. These findings underscore the potential of specific phenolic compounds to interfere with the reassociation of starch chains through hydrogen bonding or hydrophobic interactions.

In protein-rich systems, Oladiran & Emmambux (2018) observed that extrusion of cassava–soy blends with added grape pomace enhanced total phenolic content and antioxidant capacity while simultaneously altering starch digestibility and reducing the estimated glycemic index. The authors also reported structural changes (increased β-sheet content) indicative of phenolic–protein complexation, which may explain the functional shifts in texture and digestibility. The formation of phenolic–protein aggregates was also evident in Lee et al. (2016), where interaction between soy protein isolate and anthocyanin-rich extracts from *Acanthopanax sessiliflorus* reinforced the protein network in gluten-free rice noodles, reducing cooking loss and enhancing tensile properties.

Finally, in bakery systems, (Sivam et al., 2013) demonstrated that the combination of pectin and berry-derived phenolic compounds affected both gluten and starch structures during baking, likely through a mix of hydrogen bonding and hydrophobic interactions. Secondary structure alterations in proteins and the observed loss of extractable phenolic compounds suggest that macromolecule–polyphenol interactions influence both processing behavior and final product functionality.

Altogether, these studies illustrate that polyphenol–macromolecule interactions in food matrices are not only plausible but also impactful in shaping the nutritional and physical properties of the final product. They confirm that extrusion conditions and matrix composition jointly determine whether such interactions lead to enhanced stabilization and retention or diminished bioavailability of phenolic compounds. While the underlying mechanisms vary depending on the polyphenol class and macronutrient involved, analytical tools such as FTIR, X-ray diffraction, and calorimetry have proven effective in capturing these interactions in complex matrices. Further research is warranted to deepen our understanding of these phenomena, particularly in real food systems, and to optimize formulations that balance phenolic stability with desirable product characteristics.

Table 5. Interactions of phenolic compounds and macromolecules in extruded and other food matrices

Food product	Interactions	Applied techniques	Findings	Reference
Rice-oat flour and maize-oat flour based extruded containing olive pomace	Protein,carbohydrates/olive pomace phenolic compounds	FTIR	Interaction between added olive pomace components and significant changes in the carbohydrate components and the structure of the proteins on extrusion, with consequent effects on the expansion and density of the extruded product	(Ying et al., 2017)
Oat fibre extruded products with incorporation of curcuminoids	Protein,oat fibre/curcuminoids	Fluorescence analysis, XRD, FTIR, DSC	Evidence of interaction between proteins and β - glucan fractions of oat fibre. Greater solubility	(Sayanjali, 2016)

Table 5. Interactions of phenolic compounds and macromolecules in extruded and other food matrices

Food product	Interactions	Applied techniques	Findings	Reference
			of curcuminoids and protection against thermal and oxidative degradation	
Extruded cassava soy with grape pomace	Protein/Grape pomace phenolic compounds	FTIR	Grape pomace addition promoted an increase in β-sheet formation and consequent decrease of α-helical conformation, which is attributed to binding between interactions polyphenol-protein	(Oladiran & Emmambux, 2018)
Extruded chesnut starch with incorporation of catechins and proanthocyanidins	Starch/catechins, proanthocyanidins	DSC, low field NMR, XRD	Extrusion of starch with phenolic compounds reduced the recrystallization, flow resistance and gel structure strength of starch paste. Water holding capacity improved on the short/long-term retrogradation compared with extruded chestnut starch.	(Zeng et al., 2022)
Rice starch gel printed by hot extrusion 3D printing with incorporation of catechins and procyanidins	Starch/catechins, procyanidins	XRD, SAXS	Hydrogen bonds were formed between polyphenol molecules and starch. Starch gel network structure was partly disrupted, leading to a decrease in the viscosity and G′ 2 value, which improved the extrudability of the starch gel and laid the basis for successful printing.	(Zeng et al., 2021)
Gluten-free rice noodle	Soy protein/Polyphenol extract of *Acanthopanax sessiliflorus*	UV-Vis spectrophotometry, DSC	Results suggest covalent and non-covalent bonding. Combined treatment of polyphenol extract and protein on rice noodle making would be positively effective on the cooking quality of rice noodles, possibly due to protein–polyphenol interactions	(Lee et al., 2016)
Breads fortified with fibre and phenolic compounds	Protein, carbohydrates/Fruit (apple, kiwifruit, blackcurrant) polyphenol extracts	FTIR, Raman spectroscopy	Both hydrophobic and hydrogen bonding interactions occurred among bread components and the added phenolic compounds and pectin. This caused changes in the molecular	(Sivam et al., 2013)

Table 5. Interactions of phenolic compounds and macromolecules in extruded and other food matrices

Food product	Interactions	Applied techniques	Findings	Reference
			conformations and polymer structure of wheat gluten and starch in the analyzed breads.	
Extruded rice starch with incorporation of caffeic acid, ferulic acid, apigallocatechin gallate, tannic acid and resveratrol	Starch/phenolic compounds	LF-NMR, FTIR, XRD, SAXS, iodine binding, NMR, HPSEC, SEM	Phenolic compounds mainly interacted with starch via hydrogen bonds, which transformed the crystalline structure to V-type and increased the molecular weight of extruded rice starch	(Huo et al., 2025)
Extruded sweet potato starch with matcha, green tea extract, tea phenolic compounds, epigallocatechin gallate	Starch/phenolic compounds	HPSEC, XRD, FTIR, fluorescence quenching, SEM, CLSM	Starch formed larger molecular aggregates with tea products under extrusion, showing a "B + V" type pattern.	(Y. Li et al., 2023)

FTIR: Fourier Transform Infrared Spectroscopy; **XRD**: X-ray Diffraction; **DSC**: Differential Scanning Calorimetry; **NMR**: Nuclear Magnetic Resonance; **LF-NMR**: Low Field Nuclear Magnetic Resonance; **SAXS**: Small-Angle X-ray Scattering; **UV-Vis**: Ultraviolet-Visible Spectrophotometry; **HPSEC**: High-Performance Size-Exclusion Chromatography; **SEM**: Scanning Electron Microscopy; **CLSM**: Confocal Laser Scanning Microscopy

Conclusions

The enrichment of extruded snacks with plant-derived ingredients presents a valuable strategy for enhancing the nutritional and functional profile of widely consumed food products. These ingredients, ranging from fruits, vegetables, and legumes to peels, pomace, and other agro-industrial by-products, are rich in phenolic compounds and dietary fiber, both of which contribute to health-promoting properties. However, the extrusion process exerts a complex influence on these bioactive compounds. Variability in polyphenol retention and bioaccessibility is commonly observed across studies, depending not only on the processing conditions and the structural nature of the phenolics but also on their interactions with matrix macromolecules such as proteins, carbohydrates, and lipids.

The reviewed literature underscores that phenolic behavior during extrusion is matrix- and system-dependent. Some phenolic compounds may degrade under high temperature and shear, while others become more extractable due to cell wall disruption or depolymerization. Likewise, interactions with starch and fiber can modulate their stability and functionality, influencing properties such as glycemic response or antioxidant activity. Despite advances, a deeper understanding of these interactions is still needed, particularly regarding their contribution to structural integrity, nutritional potential, and sensory attributes of the final product.

Ultimately, the successful development of functional extruded snacks requires an integrative optimization approach that considers the composition and structure of plant-derived ingredients, the behavior of phenolic compounds and dietary fiber under extrusion, and the resulting impact on product quality. Future research should aim to bridge the gap between nutritional enrichment and consumer-acceptable textures by adopting multi-objective optimization models and advancing the mechanistic understanding of food matrix–compound interactions. Such an approach will be essential for leveraging the full functional potential of plant-based ingredients within sustainable and health-oriented snack innovations.

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Chapter 3. Optimizing extrusion parameters to develop puffed snacks enriched with strawberry by-products: impacts on phenolic compounds composition and content, textural properties and physical quality

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Abstract

Strawberry (*Fragaria × ananassa*) by-products (SBP), generated during industrial fruit processing, are rich in phenolic compounds, dietary fiber, and other functional components. However, these by-products are often underutilized, highlighting the need for innovative valorization strategies. This study evaluates the feasibility of using strawberry by-products as functional ingredients in tapioca starch-based extrudates, employing extrusion-cooking technology. Moreover, it aims to optimize processing conditions to improve both physical properties and the content of free and bound phenolic compounds. Using a central composite design and Response Surface Methodology (RSM), the effects of die temperature (DT), feed moisture content (FM), and SBP inclusion level (SBPC) on key response variables were analyzed. These variables included expansion index (EI), bulk density (BD), specific mechanical energy (SME), textural attributes, and polyphenol content. The aim was to identify processing conditions that maximize phenolic compound retention and expansion index, while minimizing bulk density and crispness-related hardness, in order to develop nutritionally enhanced and texturally appealing extruded snacks.

The optimization process identified optimal extrusion parameters (DT: 192 °C, FM: 18.6%, SBPC: 16.5%) that maximized the expansion index and the polyphenol content while minimizing the bulk density and the crispness work. Additionally, the proanthocyanidin (PACs) content and mean degree of polymerization (mDP) were evaluated, revealing that higher inclusion levels of SBP increased proanthocyanidin content, while elevated feed moisture levels promoted depolymerization, leading to a reduction in mDP and potential shifts in their structural profile. These results provide insights into the potential of extrusion to improve the functional properties of the final product. This study highlights the feasibility of utilizing SBP as a sustainable ingredient in snack formulations, demonstrating the role of extrusion as a tool for transforming by-products into innovative, value-added food products with improved physical, nutritional and functional properties.

Keywords: Functional ingredients, expansion index, polyphenols, proanthocyanidins, valorization, response surface methodology

1 Introduction

Strawberries (*Fragaria × ananassa*) are among the most widely cultivated berries globally, with high demand driven by their exceptional nutritional and sensory properties. However, their highly perishable nature results in a substantial portion of the harvest being processed into various products, including jams, purees, juices, wines, and frozen goods. During such processing, significant by-products are generated, particularly in juice production, where press cake or pomace, composed of seeds, skins, and residual pulp, accounts for approximately 4–11% of the total fruit weight (Pukalskienė et al., 2021). These by-products are rich in valuable nutrients such minerals, dietary fiber, and phyto-microconstituents such as phenolic compounds (e.g., anthocyanins, proanthocyanidins, ellagic and other phenolic acids, ellagitannins), and other bioactive components (Cubero-Cardoso et al., 2021; Felix et al., 2018; Pukalskienė et al., 2021; Villamil-Galindo et al., 2022) which are linked to different beneficial functions in the human body, including antioxidant properties (Bohn, 2014b). Despite their nutrient density and potential applications, strawberry by-products are often underutilized. Current uses are mainly limited to low-value applications such as animal feed or composting, or they are simply discarded as waste (Cubero-Cardoso et al., 2021), underscoring the need for innovative strategies to valorize these residues within the food industry.

These nutrient-rich by-products can serve as functional ingredients in the development of value-added food products such as snacks. As convenient and ready-to-eat options, snacks fit well into modern

lifestyles. When formulated with ingredients rich in dietary fiber and phenolic compounds, they offer a healthier alternative to conventional snack products and can contribute to improved nutritional intake and overall well-being (Hashemian et al., 2025). Several studies have explored the incorporation of fruits and their by-products into extruded snacks to leverage their nutritional and functional properties (da Silva Alves et al., 2018; Khanal et al., 2009; Korkerd et al., 2016; Méndez-García et al., 2011; Oniszczuk et al., 2019; Wójtowicz et al., 2019). However, the integration of by-products into food matrices presents notable challenges, particularly in maintaining the bioactive properties of the components and achieving desirable physical and sensory characteristics in the final products.

Extrusion, a widely employed industrial process for snack production, provides a promising approach for incorporating by-products into value-added foods. This technique not only enhances the digestibility and bioavailability of nutrients but also performs multiple functions, including mixing, cutting, pressing, expanding, and sterilizing (Khanal et al., 2009). The high-temperature and shear conditions inherent in extrusion facilitate starch gelatinization and the formation of expanded structures, which are critical for producing snacks with desirable textures. However, these conditions can also alter the bioactive compounds present in the ingredients (Šárka et al., 2021). Phenolic compounds are for the most part present in cell fruit vacuoles as free forms. However, in fresh fruits, phenolic compounds are mostly present in the vacuoles as free forms. However, in fruit by-products, especially those obtained after juice extraction, they are distributed between free forms and bound forms associated with the cell wall of the pomace (Danilov et al., 2024; Sójka et al., 2013). While free phenolic compounds are more readily bioavailable, bound phenolic compounds attached to the cell wall matrix require specific processing, such as thermal treatments like extrusion, to release them (Patil & Kaur, 2018). Proanthocyanidins, one of the most significant phenolic compound classes, also have limited bioaccessibility and bioavailability in their bound form. Extrusion has the potential to influence the release of bound phenolic compound and modify their structure, resulting in either enhanced or diminished bioactive properties depending on the specific process conditions applied (Khanal et al., 2009).

To maximize the nutritional benefits of incorporating strawberry by-products into extruded snacks, it is essential to assess their feasibility as functional ingredients in extrusion-based formulations and to optimize the processing parameters. Key variables, such as die temperature, feed moisture, and inclusion levels of by-products, must be carefully controlled to achieve the dual goals of preserving bioactive compounds and producing snacks with desirable physical properties, thereby allowing the development of a healthy product with attractive sensory characteristics. For instance, conditions that promote the release of bound phenolic compounds could significantly enhance the functional quality of the final product while ensuring consumer-acceptable texture and expansion.

This study employs Response Surface Methodology (RSM), a powerful statistical and mathematical tool for multivariable process optimization, to investigate the effects of extrusion parameters on the physical properties and phenolic compound content and composition of tapioca starch-based extrudates enriched with strawberry by-products (SBP). RSM is particularly effective in modeling the relationships between independent variables (e.g., die temperature, feed moisture, and SBP inclusion) and response variables (e.g., expansion index, free and bound phenolic compound content, bulk density, and work of crispness) (Natabirwa et al., 2018). Using a central composite design, this study explores the combined effects of these parameters to identify optimal conditions for extrusion. In addition to the optimized variables, selected treatments were further analyzed to characterize the structure of proanthocyanidins and the profile of individual phenolic compounds, providing deeper insight into the compositional transformations induced by extrusion. The findings provide critical insights into the development of nutritionally enhanced, value-added snack products. Furthermore,

this research highlights the potential of extrusion as a sustainable food innovation tool, offering a pathway to valorize agricultural residues, enhance functional properties, and reduce food waste.

2 Materials and methods

2.1 Raw materials

Strawberry (*Fragaria × ananassa*) by-products (SBP) used in this research were obtained from pulp extraction process and provided by Monkeyfruit S.A. (Popayán, Colombia). The fruits before processing were sourced from local cultivars in the municipality of Sotará, Cauca, and were of the Sabrina variety. The SBP were frozen at -80 °C for 12 h. Then, the frozen SBP were dried in a vacuum freeze dryer (YR05188, Kalstein, Paris, France) for 72 h. The dried SBP were grinded (MF 10 basic Molino IKA, Germany) rapidly and the powder was vacuum sealed and stored at -20 °C until use. Tapioca (*Manihot esculenta*) starch was supplied by Ingredion Colombia S.A. (Cali, Colombia). The SBP had a moisture content of 6.62%, protein 1.91%, fat 0.79%, ash 3.51%, and dietary fiber 29.56% d.m. The free and bound phenolic compound content was 6.86 and 1.71 mg GAE/g d.m, respectively. Tapioca starch had a moisture content of 13.48%, protein <0.1%, fat 1.47%, ash 0.24%, and dietary fiber <0.5% d.m. and total phenolic content was negligible.

2.2 Standards and chemicals

All reagents were of analytical grade, HPLC solvents were of chromatographic purity, and water was purified through deionization using a Milli-Q system (Millipore, Bedford, MA, USA). Folin-Ciocalteu reagent and sodium carbonate were obtained from Panreac (Barcelona, Spain). Acetonitrile of HPLC grade was obtained from Fisher Scientific (Strasbourg, France). Acetone was supplied by Merck (Darmstadt, Germany). Methanol, formic acid, and standards of (+)-catechin, (−)-epicatechin, quercetin, kaempferol, pelargonidin-3-O-β-glucoside, cyanidin-3-O-β-glucoside, coumaroylhexoside acid, as well as other standards and reagents, were sourced from Sigma-Aldrich (Steinheim, Germany).

2.3 Preparation of mixtures

The mixtures were prepared with tapioca starch and SBP powder at varying inclusion levels and moisture contents, as specified in the experimental design (Table 1). The amount of water required to reach the target moisture content was calculated considering the initial moisture of the raw materials and then added by spraying onto the dry mixture using a spray bottle. Hydration was carried out at room temperature, followed by mixing for ten minutes at medium speed in a KP26M1XER commercial mixer (KitchenAid, MI, USA). Finally, the mixtures were packed in polyethylene bags and stored overnight (12–14 hours) at room temperature to ensure homogeneity before processing.

2.4 Extrusion process

Extrusion cooking was carried out using a laboratory-scale co-rotating twin-screw extruder (HAAKE™ Rheomex OS, ThermoFisher Scientific, Germany) with a screw diameter of 11 mm, a length-to-diameter ratio of 40:1, and a die opening of 3 mm. The barrel temperatures (140/160/180 °C), screw speed (140 rpm), and feed rate (20 g/min) were kept constant throughout the process and the strawberry by-products content (SBPC), die temperature (DT) and feed moisture (FM) were established based on 20 experimental runs (Table 1). The extrudates were dried at 60 °C for 30 minutes, packed in polyethylene bags, and stored in an airtight container until physical tests were performed. For chemical and color analyses, the samples were ground using a laboratory grinder (A 11 basic Molino IKA, Germany) and passed through a mesh-60 sieve.

Table 1. Experimental design for the extrusion process

	Coded levels			Actual levels		
Run				**SBPC (%)**	**DT (°C)**	**FM (%)**
1	-1	-1	-1	15	180	20
2	1	-1	-1	25	180	20
3	-1	1	-1	15	200	20
4	1	1	-1	25	200	20
5	-1	-1	1	15	180	24
6	1	-1	1	25	180	24
7	-1	1	1	15	200	24
8	1	1	1	25	200	24
9	-1.68179	0	0	11.6*	190	22
10	1.68179	0	0	28.4*	190	22
11	0	-1.68179	0	20	173.2*	22
12	0	1.68179	0	20	206.8*	22
13	0	0	-1.68179	20	190	18.6*
14	0	0	1.68179	20	190	25.4*
15	0	0	0	20	190	22
16	0	0	0	20	190	22
17	0	0	0	20	190	22
18	0	0	0	20	190	22
19	0	0	0	20	190	22
20	0	0	0	20	190	22

SBPC: Strawberry by-products content; DT: Die temperature; FM: Feed moisture. *Values were rounded up to the nearest whole number to set extrusion conditions

2.5 Determination of physical properties and system parameters

2.5.1 Expansion index (EI) and bulk density (BD)

The expansion index was determined by measuring the diameter of the extrudate using a Vernier caliper (Mitutoyo Co., Kawasaki, Japan) and dividing it by the internal diameter of the extruder die. Ten measurements were taken for each extrudate sample (Medina-Rendon et al., 2023).

The bulk density was calculated based on the extrudate's volume, derived from its dimensions, and its measured weight. The length and diameter of the extrudate were measured using a digital Vernier caliper (Mitutoyo Co., Kawasaki, Japan), while an analytical balance (KERN ABJ 220-4NM, Balingen, Germany) was used to determine its weight. The volume and weight of 10 extrudates were recorded, and the bulk density was calculated as mass per unit volume (g/cm^3) (Mohamad Mazlan et al., 2020).

2.5.2 Textural properties

The textural properties of the extrudates were evaluated using a texture analyzer (EZ Test Texture Analyzer, Shimadzu Corporation, Japan) equipped with Trapezium X software (Shimadzu Corporation, Japan). Twenty-five extrudates from each treatment were equilibrated in a humidity

chamber (30% RH) for 94 hours. The samples were compressed perpendicular to the extrusion direction to 50% of their original diameter using a 1 mm compression punch at a test speed of 0.5 mm/min. The hardness (H) values were directly obtained from the software data. A force-deformation curve was obtained using Origin software (version 2018, OriginLab Corporation, Northampton, MA, USA), and the number of peaks (*n*) above 1.5 N, integral of the curve (*S*) (or area below the curve), and distance of compression (*x*) were computed. Using *n, S,* and *x* values, spatial frequency of ruptures (NSr), average crushing force (FCr) and crispness work (WCr) were calculated using Equations (1), (2) and (3) (Devi et al., 2013; Karkle et al., 2012).

$$NSr\ (mm^{-1}) = n/x \quad (1)$$

$$FCr\ (N) = S/x \quad (2)$$

$$WCr\ (N.mm) = FCr/NSr \quad (3)$$

2.5.3 Color

The color parameters *L*, a** and *b** were measured using tristimulus colorimetry with a Konica Minolta CR-400 colorimeter (Minolta Co., Osaka, Japan). The samples were placed in 5 cm diameter Petri dishes, and 15 readings were recorded for each treatment. The mean values and standard deviations were then calculated. Color index (CI), Chroma (*C**) and Hue (*h*) were determined using Equations (4) to (6) (Neder-Suárez et al., 2021; Téllez-Morales et al., 2022).

$$CI = \frac{1000\ x\ a^*}{L^*\ x\ b^*} \quad (4)$$

$$C^* = \sqrt{\left(a^{*2} + b^{*2}\right)} \quad (5)$$

$$h = \arctan\ (b/a) \quad (6)$$

2.5.4 Specific mechanical energy (SME)

The total mechanical energy input, commonly referred to as specific mechanical energy (SME), was calculated using Equation (7) (Reißner et al., 2022; Ribeiro et al., 2024). The average mass flow rate, Q (g/min), was determined based on the mass throughput measured over 30 seconds in quadruplicate. Energy input was calculated using the mean values of torque (T) and screw speed (SS), which were recorded every 15 seconds during extrusion. Each test condition included at least 15 recorded values.

$$SME = (2\pi * T * SS)/Q \quad (7)$$

2.6 Effect of extrusion on phenolic compounds

2.6.1 Free and bound phenolic compound content

Phenolic compounds are categorized into free phenolic compounds (FPP), which are extracted with aqueous-organic solvents, and bound phenolic compounds (BPP) attached to the cell wall after processing which need an acid hydrolysis to be extracted because they are bound to macromolecules like proteins or polysaccharides from the cell wall.

The FPP extraction and analysis was based on the method described by Pico et al. (2020) with any modifications. 2 g (±0.0500 g) of powder sample was weighed in a centrifuge tube and 8 mL of 80 % methanol in 0.1% formic acid were added for the first extraction during 15 min using a wrist shaker. The sample was centrifuged for 5 min at 3500 rpm and 20 °C and 40 µL of 2 % EDTA were added to

the supernatant for the stabilization of flavan-3-ols. The sample solution was kept on ice. 8 mL of 70 % acetone in 0.1 % formic acid were added to the pellet for a second extraction for 15 min using the wrist shaker. After centrifugation, the supernatant was combined with the methanolic extract from the first extraction and made up to 20 mL with deionized water. The extract was kept at −80 °C for the colorimetric determination by Folin-Ciocalteu reaction.

The pellet was also kept at −80 °C for further sequential determination of BPP. The extraction of this fraction was based on the method described by Pico et al. (2019). 0.8 g (± 0.0050 g) of sample from the FPP pellet were weighed in a glass tube and 10 mL of methanol/sulfuric acid (90/10) were added. The hydrolysis was carried out for 22 h at 85 °C, with magnetic stirring. The sample was then centrifuged at 3500 rpm for 20 min and the supernatant was made up to 25 mL with deionized water. The extract was kept at −80 °C for the colorimetric determination by Folin-Ciocalteu reaction.

For the Folin-Ciocalteu reaction, 260 µL of Milli-Q water, 26 µL of 7.5 % (v/v) Na_2CO_3, 20 µL of extract and 20 µL of Folin–Ciocalteu reagent were mixed in a 96-well plate. The samples were incubated for 1 h at room temperature in the dark and the spectrophotometric determination was performed at 765 nm in a multimode microplate reader Varioskan™ LUX (ThermoFisher Scientific, Germany). For the blank, 20 µL of deionized water were used. The total content of FPP and BPP were expressed as mg of gallic acid equivalents (GA)/g dry matter, based on a GA calibration curve. All analyses were carried out in quadruplicate and the handle of the reagents was performed in conditions as dark and cold as possible.

2.6.2 Identification and quantification of phenolic compounds

Identification of phenolic compounds was performed using HPLC coupled to ElectroSpray Ionisation Mass Spectrometry (HPLC/ESI-MS^2) analysis was performed on an Acquity Ultra performance LC (UPLC) apparatus from Waters (Milford, MA, USA), equipped with a photodiode array detector coupled with a Bruker Daltonics (Bremen, Germany) HCT ultra ion trap mass spectrometer with an electrospray ionization source. Separations were achieved using a Luna Omega Polar C18 column (50 mm × 2.1 mm × 3 µm column, Phenomenex, Torrance, USA) operated at 30 °C. The mobile phase consisted of water/formic acid (98,2, mL/mL) (eluent A) and acetonitrile (eluent B). The flow rate was 1 mL/min. The elution program was as follows: 3–9 % B (0–5 min); 9–16 % B (5–15 min); 16–50 % B (15–45 min); 50–90 % B (45–48 min); 90–90 % B (48–52 min). 10 µL of samples (“furanolysis” and “crude” extracts) were injected. For polyphenol characterization, a capillary voltage of 2 kV was used in the negative ion mode. N2 was used as drying and nebulizing gas with a flow rate of 12 L/min. The desolvation temperature was set at 365 °C and the nebulization pressure at 0.4 MPa. The ion trap was operated in Ultrascan mode from m/z 100 to 1000. For anthocyanin characterization, a capillary voltage of 1.8 kV was used in the positive ion mode under the same conditions. Phenolic compounds were analyzed and quantified by HPLC-DAD (Prominence system, Shimadzu, Kyoto, Japan) and separations were achieved as described in Rincon et al. (2025). Analyses of phenolic compounds were carried out with or without the acidic depolymerization of proanthocyanidins (PACs) in the presence of menthofuran (named furanolysis). PACs were thus characterized by their subunit composition, average degree of polymerization (mDP) and contents. HPLC-DAD analyses of crude methanolic extracts (not submitted to furanolysis) were performed to titrate monomeric catechins.

Furanolysis and methanol extract were obtained as described in Rincon et al. (2025). The most prevalent compounds were quantified in the UV–visible region by chromatographic analysis conducted at wavelengths of 280 nm (flavanols), 320 nm (phenolic acids), 360 nm (flavonols), and 520 nm (anthocyanins). Individual compounds were quantified in mg/100 g of fresh weight (FW) by comparison with external standards: (+)-catechin and (−)-epicatechin for flavanols, caffeic and para-

coumaric acids for phenolic acids, quercetin and kaempferol for flavanols, and pelargonidin-3-*O*-β-glucoside and cyanidin-3-*O*-β-glucoside for anthocyanins.

The mean degree of polymerization (mDP) of proanthocyanidins was calculated as the molar ratio of all flavan-3-ol units (i.e., furan adducts plus terminal units, minus monomers from crude methanol extract) to the sum of terminal units, minus monomers from crude methanol extract (X. Liu et al., 2021b).

2.1 Experimental design, statistical analysis and optimization

An orthogonal and rotatable central composite design 2^3 with three independent variables (SBPC, DT and FM) was used, resulting in a total of 20 experimental runs (Table 1). The dependent variables measured included expansion index, bulk density, crushing force, hardness, spatial frequency of ruptures, color, specific mechanical energy, free polyphenol content, and bound polyphenol content. Response surface methodology (RSM) was applied to determine the optimal combination of conditions and to explore the relationships between the selected process variables for extrusion. Experimental data were evaluated through analysis of variance (ANOVA), Tukey's test, and multiple regression analysis at a significance level of 0.05 ($p < 0.05$). The data were modeled using Design-Expert® 13 (Stat-Ease, Inc., Minneapolis, MN, USA) and analyzed using IBM® SPSS® Statistics (Version 27, IBM Corp., Armonk, NY, USA). The behavior of each response variable concerning the independent parameters was fitted to a polynomial model. The quality and adequacy of the models were assessed by evaluating the coefficient of determination (R^2) and the lack of fit.

Optimization was performed by maximizing the expansion index, a key variable for an expanded extrudate, while also maximizing polyphenol content and minimizing bulk density and the crispness work (WCr). Derringer's desirability function methodology was employed to generate optimal conditions for the extrusion process variables, considering all the properties of the extruded snacks. The optimal conditions were validated by applying them in the process and determining all associated variables. Validation was verified through a one-sample t-test performed for each variable, comparing the experimental values with those predicted by the model.

Beyond the variables included in the optimization study, additional compositional analyses were performed to provide a deeper understanding of the effects of extrusion on the bioactive profile of the extrudates. These included the profiling of individual phenolic compounds by HPLC as well as the quantification of total content of proanthocyanidins, their mean degree of polymerization, and constitutive units. These analyses were carried out on a subset of nine treatments corresponding to the factorial points of the central composite design and one replicate of the central point (run 15), selected to represent the experimental design space. Although not used in the construction of the predictive models, these data offered complementary insights into the compositional changes induced by the processing conditions and contributed to a more comprehensive evaluation of the extrudates.

3 Results and discussion

The impact of processing conditions on the different variables is shown in Figs. 1–5, while the complete dataset is provided in Supplementary Material S1. The results of the regression models for each variable are summarized in Table S1.

3.1 Effect of extrusion conditions on physical properties

3.1.1 Expansion index and bulk density

During the extrusion process, biopolymers undergo structural modifications due to shear forces and pressure, resulting in the gelatinization and dextrinization of starch components (Babatunde et al., 2023; V. T. Huang & Perdon, 2020; Neder-Suárez et al., 2024). This leads to increased expansion. The expansion index (EI) and bulk density (BD) are important parameters in extruded snacks, with a higher EI being a key physical quality indicator for these products (Neder-Suárez et al., 2024). The EI ranged from 1.6 to 2.3 (Table S1), depending on the extrusion conditions, consistent with the findings of Wójtowicz et al., (2019), who report similar results for strawberry inclusions above 15%. EI was significantly influenced by all factors, particularly by feed moisture (FM) and the inclusion level of strawberry by-products (SBPC). Additionally, the quadratic effects of FM and temperature (DT) impacted its behavior ($p < 0.05$) (Table 2).

BD, other index of puffing extent, was recorded between 0.2 and 0.6 g/cm³, similar values were registered by Wójtowicz et al., (2019) at 20 % of strawberry inclusion. BD was also primarily affected by FM and DT, with observable effects from all quadratic terms and the combined influence of DT and FM ($p < 0,05$) (Table 2). The inverse behavior observed with respect to the EI was expected, as the literature often suggests that a reduction in expansion is inversely proportional to the measurement of bulk density (Wójtowicz et al., 2019). Likewise, the difference in size of the effect of the evaluated factors on these two variables could be attributed to the fact that bulk density accounts for expansion in all directions (Singh et al., 2014).

Feed moisture had an inverse effect on EI and a direct effect on BD, indicating that greater expansion was achieved with lower feed moisture levels (Fig. 1 a, b). Earlier studies on the development of snacks with inclusion of fruits materials (Falfán Cortés et al., 2014; Rathod & Annapure, 2017; Samyor et al., 2018) reported the same effect. This behavior could likely be due to the role of water as a plasticizer in the amorphous regions of starch granules, where it directly participated in gelatinization and contributed to the rheological properties of the melt and the formation of gas bubbles (Oliveira et al., 2017). In this context, additional water acted as a plasticizer, reducing dough viscosity and lowering resistance to flow, which led to decreased gas retention and, consequently, reduced expansion (Natabirwa et al., 2018; Sahu et al., 2022).

Furthermore, increasing the incorporation of strawberry by-products reduced the expansion of the extrudates. Similar effects have been reported for extrudates with incorporation of fruits such as chokeberry, elderberry and strawberry (Wójtowicz et al., 2019) and materials derived from them such as apple pomace (Ačkar et al., 2018), passion fruit shell (da Silva Alves et al., 2018) and mango peel (Mohamad Mazlan et al., 2020). This behavior could be due to the higher dietary fiber content introduced by the fruits and their by-products, which bound water more effectively than starch, leading to less water loss at the die and reduced expansion capacity. Additionally, the fiber structures may interfere with the formation and stability of the gas cells during expansion, limiting bubble growth (Ačkar et al., 2018; Bisharat et al., 2013). Moreover, the inclusion of fruit materials had been reported to act as a lubricant, reducing friction inside the extruder and lowering shear forces, which in turn decreases extrusion pressure and limits expansion (Ménabréaz et al., 2021).

The expansion of extruded products is a complex process that occurs under specific conditions of high temperature and low feed moisture, driven by events such as structural transformations of biopolymers, phase transitions, and formation of air bubbles (Falfán Cortés et al., 2014). The die temperature had a positive effect on expansion up to 180 °C, beyond which expansion decreased (Fig 1 a). This observation is consistent with findings by R. P. Rathod & Annapure (2016, 2017), who reported that expansion depends on starch gelatinization, and die temperature is a critical factor influencing starch modification during extrusion. However, excessive starch degradation can lead to decreased expansion, which may explain the decline in expansion beyond 180 °C.

Table 2. Regression coefficients of the effects of feed moisture, die temperature and strawberry by-product contents, quadratic and interaction terms on extrudate properties

Source	EI	BD	H[b]	FCr[b]	NSr	WCr[b]	L*	a*	b*	C*	h	CI	SME	FPP[b]	BPP
Intercept	1.94	0.34	3.67	2.95	20.53	-0.07	56.48	3.07	5.95	6.79	63.08	8.99	2457.01	1.28	2.62
SBPC	-0.13[a]	0.03[a]	0.05[a]	0.06[a]	0.45	0.04[a]	-1.83[a]	0.10[a]	0.22[a]	0.24[a]	0.22	0.22[a]	-189.85[a]	-0.19[a]	0.22[a]
DT	-0.05[a]	0.07[a]	0.11[a]	0.08[a]	-0.72	0.14[a]	-0.29	-0.05	0.12[a]	0.09	0.92[a]	-0.34[a]	12.49	-0.004	0.27[a]
FM	-0.31[a]	0.17[a]	0.47[a]	0.44[a]	-2.77[a]	0.63[a]	-2.87[a]	-0.45[a]	-0.66[a]	-0.79[a]	1.04[a]	0.03	-424.21[a]	0.09[a]	-0.39[a]
SBPC*DT	0.04	-0.02	0.05[a]	0.03[a]	0.88	-0.02	0.16	0.04	0.01	0.03	-0.15	0.06	48.67	-	0.34[a]
SBPC*FM	0.03	-0.01	-0.03	0.01[a]	-0.86	0.06	0.02	-0.16[a]	-0.13	-0.19[a]	0.83[a]	-0.29[a]	173.42[a]	-	-0.39[a]
DT*FM	0.02	0.09[a]	0.26[a]	0.19[a]	-2.21[a]	0.34[a]	-0.29	-0.18[a]	-0.19[a]	-0.24[a]	0.67[a]	-0.27[a]	-101.15[a]	-	-0.5
$SBPC^2$	0.02	-0.02[a]	0.04[a]	0.04[a]	-2.35	0.19[a]	-0.23	-0.03	-	0	0.54[a]	-0.05	96.42[a]	-	0.37
DT^2	-0.08[a]	0.05[a]	0.10[a]	0.08[a]	-0.66	0.12[a]	-0.68[a]	-0.02	-	-0.1	-0.33	0.20[a]	36.67	-	-0.2
FM^2	-0.12[a]	0.08[a]	0.16[a]	0.14[a]	-1.95[a]	0.27[a]	-0.90[a]	-0.11[a]	-	-0.11[a]	0.68[a]	-0.16[a]	-4.77	-	0.24
p of F (model)	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001
Lack of fit	0.40	0.49	0.41	0.54	0.12	0.11	0.26	0.06	0.07	0.06	0.43	0.41	0.14	0.07	0.09
R^2	0.98	0.99	0.99	1.00	0.95	1.00	0.96	0.96	0.95	0.97	0.94	0.93	0.98	0.75	0.97
Adjusted R^2	0.96	0.98	0.99	1.00	0.91	0.99	0.93	0.93	0.92	0.94	0.88	0.87	0.97	0.70	0.94
Predicted R^2	0.89	0.94	0.97	1.00	0.69	0.97	0.79	0.76	0.84	0.81	0.70	0.66	0.89	0.58	0.79

SBPC: Strawberry by-products content; **DT**: Die temperature; **FM**: Feed Moisture; **EI**: Expansion index; **BD**: Bulk density; **H**: Hardness; **FCr**: Crushing force; **NSr**: Spatial frequency of ruptures; **WCr**: Crispness work; **L***: Luminosity; **a***: redness; **b***: yellowness; **C***: Chroma; **h**: Hue; **CI**: Color index; **SME**: Specific mechanical energy; **FPP**: Free phenolic compounds; **BPP**: Bound phenolic compounds.

[a] Statistically significant at $p<0.05$

[b] Transformed model: H (Natural Log), FCr (Natural Log), WCr (Natural Log), Free phenolic compounds (Lambda of -0.6).

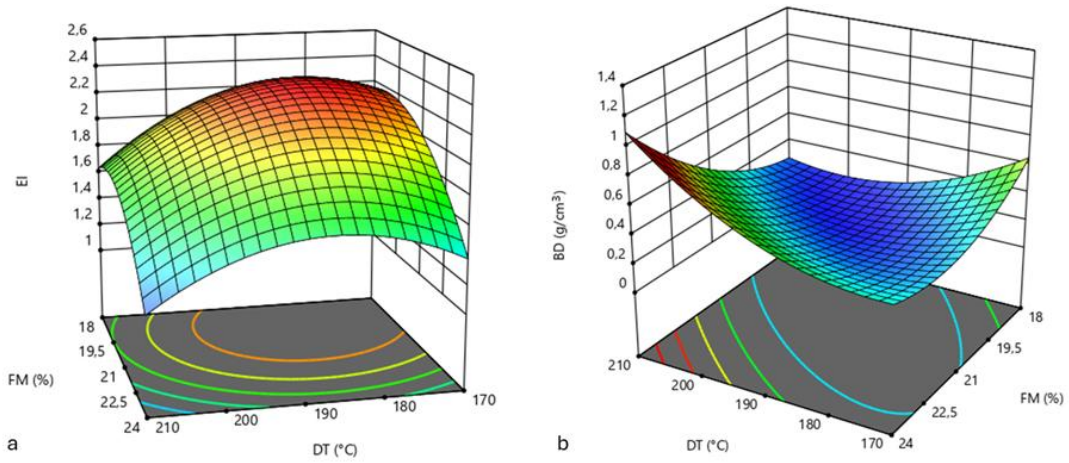

Fig. 1 Response surface curve showing the effect of extrusion process variables on (a) expansion index (EI) and (b) bulk density (BD).
DT: Die temperature; FM: Feed moisture.

3.1.2 Color

The color parameters (*L*, a*, b**) were significantly influenced by both SBPC and FM, while DT only affected the *b** value, and the interactions and quadratic terms affected the color profile in different ways ($p < 0.05$) (Table 2). *L** values ranging from 50.16 to 59.20, *a** values from 2.07 to 3.82 and *b** values from 5.21 to 7.25. According to the results, higher feed moisture and inclusion of strawberry by-products decreased lightness (*L**). The behavior of lightness *L** aligned with the expansion results as a strong correlation between the two parameters ($r = 0.95$) was observed. As discussed previously, lower moisture levels resulted in more expanded extrudates, likely due to the formation of less compact air cells and a greater number of bubbles during extrusion cooking. This structural change, particularly the thinner cell walls, may affect porosity and consequently influenced the degree of light reflection (Chan et al., 2019). Additionally, extrudates with higher levels of by-products appeared darker, which was not only attributed to the progressive darkening induced by increasing amounts of by-products (Aussanasuwannakul et al., 2022), but also to chemical reactions that occurred during extrusion cooking. These reactions, including pigment degradation, hydrolysis, oxidation, and non-enzymatic browning processes such as sugar caramelization and the Maillard reaction could significantly affect the final color (Ačkar et al., 2018; Aussanasuwannakul et al., 2022; Falfán Cortés et al., 2014). Similar results were observed for extruded snacks enriched with strawberry by Wójtowicz et al. (2017), who found *L** values ranging 44.73 to 63.24 with decreasing addition from 20 to 5 %. This same range of inclusions was evaluated by Gumul et al. (2023), who observed similar behavior with values varying from 30.2 to 53.43, 50.31 to 66.16 and 45.2 to 60.73 depending on inclusion level of chokeberry, cherry and blackcurrant by-products respectively.

On the other hand, redness (*a**) and yellowness (*b**) were significantly affected by FM and its interaction with DT. The *a** value was also influenced by the interaction of SBPC with FM, as well as the quadratic term of FM (Table 2). Lower moisture and higher by-product content increased both redness (*a**) and yellowness (*b**), whereas low temperature combined with high moisture decreased these values. This behavior highlighted the impact of moisture on these physical parameters, with a strong negative correlation between moisture content and both *a** ($r = -0.86$) and *b** ($r = -0.88$). Higher moisture levels are typically associated with increased gelatinization and reduced energy dissipation within the system due to lower viscosity. Consequently, the energy required to process the

melt in the screw channel would primarily come from thermal input from the barrel walls, rather than from molecular friction, potentially intensifying thermochemical changes (Akdogan, 1999; Chiodetti et al., 2024). These changes may affect pigments related to color expression, as flavonoids have been associated with both reddish and yellowish hues (Lu et al., 2021), particularly anthocyanins in relation to redness (Neder-Suárez et al., 2021). However, yellow coloration has been more consistently linked to carotenoids (Xiao-Dong et al., 2023), which were not within the scope of the present study.

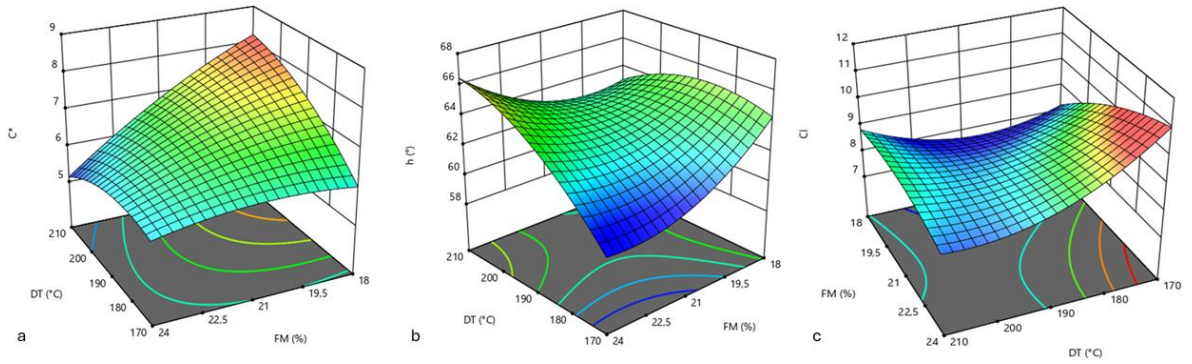

Fig. 2 Response surface curve showing the effect of extrusion process variables on color parameters: (a) Chroma (C*), (b) Hue (*h*) and (c) color index (CI).
DT: Die temperature; FM: Feed moisture.

It was not surprising that *a** and *b** values increased with higher levels of by-product content, given the presence of pigments in strawberries. A similar trend was observed by Wójtowicz et al. (2017), who report values ranging from 5.34 to 15.61, increasing with higher strawberry content.

Furthermore, *C** and *h* values were in the range of 5.20 to 7.81 and 60.51 to 67.84 ° respectively. Extrudates were shifting away from the red color characteristic of strawberries (Fig. S1), showing a greater tendency towards orange or yellow hues (*h* values), particularly at high moisture and temperature levels, but with low intensity (*C** values) (Fig. 2 a, b). In contrast to the findings of this study, Neder-Suárez et al. (2021) reported higher *h* and **C* values at high temperature (142 °C) but under low moisture conditions (22%). Respect to CI values, which ranged from 8.25 to 10.15 (Fig. 2 c), indicating desaturated yellow and orange hues with low intensity (Vignoni et al., 2006). Téllez-Morales et al. (2022) reported similar values and trends for corn flour-based extrudates containing other types of biological material. They suggest that this observed behavior could be attributed to previously mentioned reactions, such as sugar caramelization and Maillard reactions, potentially leading to the development of yellowish tones. Another factor contributing to this behavior, as mentioned earlier, could be the degradation of pigments responsible for red color, such as anthocyanins.

3.2 Effect of extrusion conditions on textural properties

Crushing, hardness, and crispness are important parameters in determining the quality and consumer acceptance of expanded extruded snacks. These textural attributes significantly influence the sensory experience and overall satisfaction of the product (Yadav et al., 2018). Crushing force (FCr) measures the average force required to crush the foam structure, analogous to the sensory parameter of hardness (H), which is defined as the force needed to completely crush the sample with the molars. On the other hand, spatial frequency of ruptures (NSr) describes the number of fractures events during puncture (Uribe-Wandurraga et al., 2020). It refers to the frequency of fractures or breaks in the snack's structure during mastication and it is a measure of crispness (Sahu, 2020). Fracture work

(WCr) measures the average work required to fracture a cell or a group of cells simultaneously, similar to the sensory parameter of fracturability, which is defined as the force with which the sample fractures on the first bite down with the molars (Devi et al., 2013). The values of the textural properties ranged from 22 to 134 N for H, 13 to 59.5 N for FCr, 9.9 to 23.3 mm^{-1} for NSr and 0.6 to 5.5 N·mm for WCr (Table S1). All individual factors had a significant effect on most textural properties ($p < 0.05$) (Table 2).

In the case of H, DT and FM were the factors with the greatest influence, and the interaction effects between these two factors were also observed, as well as the effects of all quadratic individual factors ($p < 0.05$) (Table 2). The H values were comparable to those observed by Wójtowicz et al. (2019) who reported values between 44 and 105 N depending on the level of strawberry inclusion, with higher values at greater inclusion levels. Similarly, Gumul et al. (2023) found higher hardness values with increased cherry and chokeberry pomace content. Based on the observed behavior, the highest hardness values were obtained with combinations of high moisture and temperature levels (Fig. 3 a), which aligns with findings by Sahu (2020) for maize-millet extrudates fortified with soy. In the same way, Falfán Cortés et al. (2014) observed a similar effect by increasing of moisture, although they observed no significant effect of temperature. Medina-Rendon et al. (2023) also observed a similar effect of temperature in extrudates enriched with mango by-products, but they reported an inverse effect for moisture. However, the moisture range evaluated (17-21%) was lower than in the present study.

The effect observed in this study may be due to the influence of the temperature and moisture on the expansion of the extrudates, which was negatively related to the H ($r = -0,821$). Increased expansion resulted in lower fracture resistance, which was expected because of a lower compaction of extrudates (Uribe-Wandurraga et al., 2020). This was mainly because of a more expanded extruded product had longer but thinner cell walls, making it easier to crush under compression (Kesre & Masatcioglu, 2022). Based on the results observed, increasing feed moisture at each die exit temperature led to an increase of H levels. This may be attributed to the plasticizing effect of water on the amorphous regions of starch granules, which reduces viscosity and the capacity for mechanical energy dissipation in the extruder. Consequently, the final product becomes denser and harder, with restricted cell growth (Kesre & Masatcioglu, 2022; Natabirwa et al., 2018; Sahu et al., 2022).

The results of FCr and WCr showed high positive correlations with these exhibited for H ($r = 0.99$ in both cases). Two parameters followed a similar pattern, and a high and significant correlation was observed between them ($r = 0.99$). FCr was significatively affected by all factors (lineal and quadratic) and interactions ($p < 0.05$), while WCr was mainly influenced by FM and DT and to lesser extent by SBPC, and a negligible influence was observed of SBPC and DT interactions ($p < 0.05$) (Table 2). The highest values for these variables were observed with a combination of high levels of DT and FM (Fig. 3 b, c). WCr is related with fracturability and a similar behavior was found by Wójtowicz et al. (2019) respect to the low effect of the inclusion level of dried strawberry on the fracturability of expanded snacks and it depended on the screw speed. As for H, a high and inverse association was found with the expansion of the extrudates for both FCr and WCr ($r = -0.86$ and $r = -0.79$, respectively). Same relations were also observed in extruded snacks by Karkle et al. (2012) and Ačkar et al. (2018) between fracturability and the expansion and hardness, which may be related to the changes occurring during the extrusion explained above.

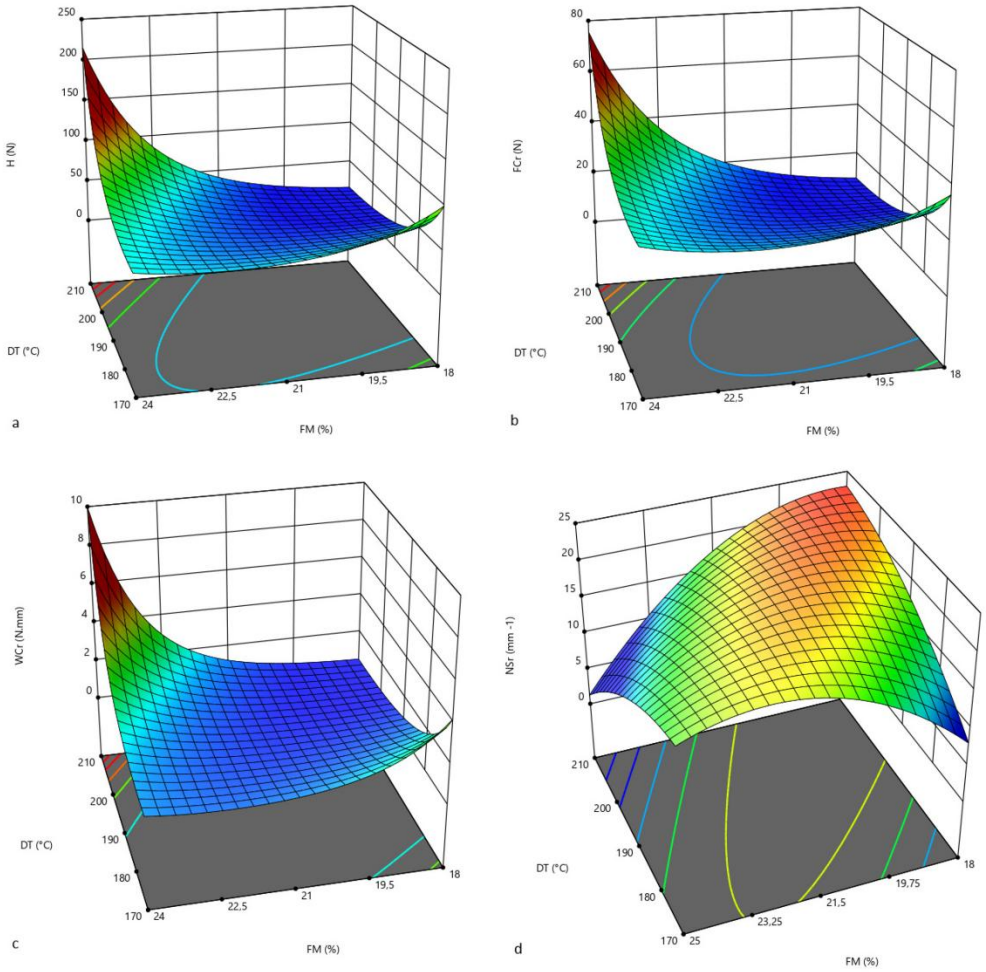

Fig. 3 Response surface curve showing the effect of extrusion process variables on textural properties: (a) hardness (H), (b) crushing force (FCr), (c) fracture work (WCr) and (d) spatial frequency of ruptures (NSr). DT: Die temperature; FM: Feed moisture.

Regarding NSr, a negative association was observed with H (r = -0.78) and no significant effect was found for SBPC, DT and DT^2 or for interactions of SBPC with DT or FM (Table 2). However, the interaction between DT and FM was highly significant. NSr reached its highest values under a combination of low feed moisture and high die temperature, whereas the lowest values were observed when both variables were at low levels or both at high levels (Fig. 3 d). As mentioned above, the spatial frequency of ruptures was an indication of the crispness, and similar to this study, the results shown by Wójtowicz et al. (2019) indicated little or no effect of inclusion level of dried strawberry on the crispness of extruded snacks. In contrast, Karkle et al. (2012) observed a significant effect of the apple pomace content in extruded snacks, but similarly to the findings in this study, a decrease in moisture caused an increase in NSr, indicating a more fragile porous structure and greater crispness (Renoldi et al., 2021). This behavior makes sense since NSr was directly associated with the expansion index. In fact, a positive correlation was observed between these parameters (r = 0,56). Longer walls, caused by a greater expansion in the direction of compression, combined with greater variation in individual wall lengths, increase the sensitivity to applied force and result in a higher number of sequential fractures as the weakest cells buckled first. This allows for the detection of individual fractures, leading to more distinct crushing peaks over the same probe travel distance when the moisture decreases (Karkle et al., 2012). With respect to the temperature factor, unlike this study,

other authors have found a significant increase of crispness with a rise in die or barrel temperature, though they evaluated lower levels of this factor (110 – 150 °C) (Kesre & Masatcioglu, 2022; Sahu, 2020; Zambrano-Moreno et al., 2015).

3.3 Effect of extrusion conditions on system parameters: Specific mechanical energy (SME)

Specific mechanical energy (SME) is an important quantitative descriptor in extrusion processes, as it determines the degree of mechanical energy delivered to the material, which directly influences the extent of macromolecular transformations and the rheological properties of the melt (Guerrero et al., 2014; Ondo & Ryu, 2013). SME measures the energy transferred into the extrusion system and dissipated as heat through shear friction during effective conveying. This heat generation leads to the gelatinization of starch, a process particularly enhanced under high mechanical energy conditions (Mohamad Mazlan et al., 2020). This variable was significantly affected by the linear and quadratic factor of SBPC as well as by the linear factor of FM and its interaction with SBPC and DT ($p < 0.05$) (Table 2). Hence, increased strawberry by-products content led to a significant decrease in SME (Fig. 4 a), indicating that their higher content facilitated better flow through the extruder, thus reducing SME (Karkle et al., 2012). The effect of including different plant materials has been reported by other authors, such as Karkle et al. (2012), who register that increasing apple pomace content has no significant effect on SME, although they note a general trend of decreasing SME with higher apple pomace levels. Other studies have also reported a significant decrease in SME with the inclusion of spinach, citrus fiber, and mango peel (Mohamad Mazlan et al., 2020; Pitts et al., 2016; Shevkani et al., 2019).

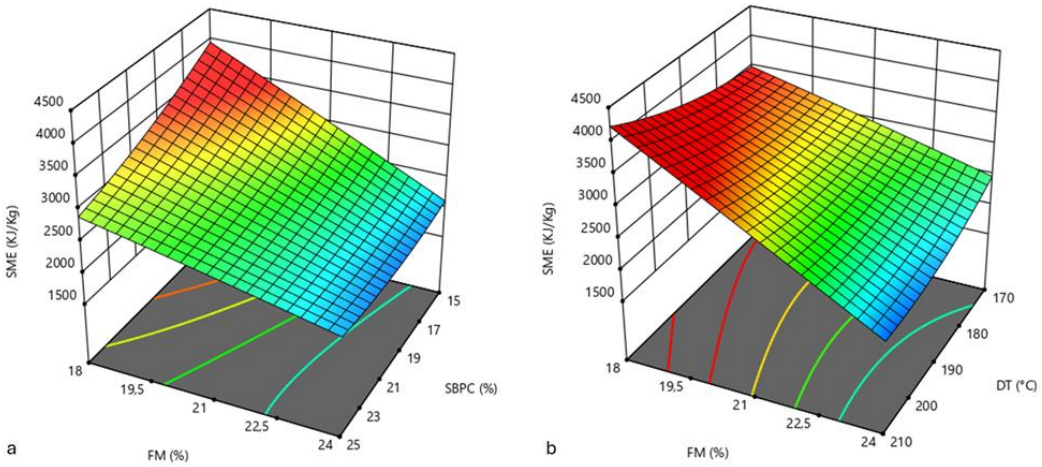

Fig. 4 Response surface curve showing the effect of extrusion process variables on specific mechanical energy (SME): (a) FM *vs* SBPC and (b) FM *vs* DT.
SBPC: Strawberry by-product content; DT: Die temperature; FM: Feed moisture.

On the other hand, lower values were observed at higher moisture levels and higher die temperatures (Fig. 4 b), which is consistent with the results obtained for EI. In fact, a strong correlation was observed between SME and EI ($r = 0.84$), BD ($r = -0.76$) and even L^* ($r = 0.85$), all of which were interrelated, as discussed above. Similar correlations have been reported previously Karkle et al., (2012), indicating that higher moisture content facilitates flow during extrusion, a phenomenon commonly referred to as the lubricating effect (Mohamad Mazlan et al., 2020). This effect aids flow through the barrel but reduces both the pressure and the energy needed for expansion. In contrast, Mohamad Mazlan et al. (2020) found no evidence of a lubricating effect in extrudates containing corn grits and mango peel perhaps due to the use of a lower moisture range (15.5%–21.5%). However, regarding the effect of temperature on SME, they report high values with increasing temperature.

These results confirmed how the different factors influenced starch conversion by disrupting semi-crystalline structures, leading to starch granule rupture (Shevkani et al., 2019), and affecting melt viscosity, thereby influencing the energy input into the system. These changes within the barrel impacted the development of the structure of the matrix in puffed extrudates, as evidenced by the significant correlations observed between SME, EI, BD and *L**.

3.4 Effect of extrusion on phenolic compounds

3.4.1 Free and bound phenolic compound content

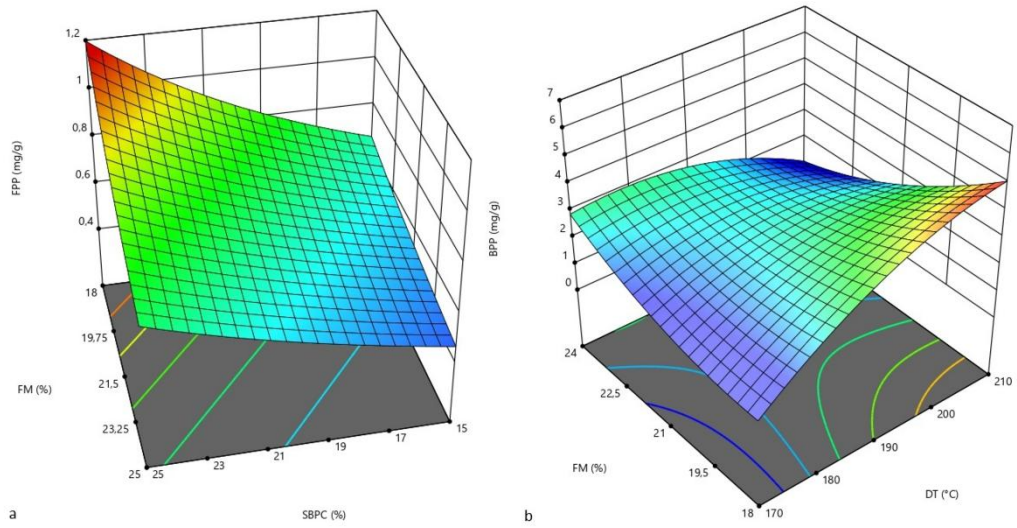

Fig 5. Response surface curve showing the effect of extrusion process variables on textural properties: (a) Free (FPP) and (b) bound (BPP) phenolic compounds.
SBPC: Strawberry by-product content; DT: Die temperature; FM: Feed moisture.

As expected, high inclusion level led to high phenolic compound contents. FPP contents, according to the factors considered by the linear model obtained, were affected significantly by SBPC and FM, while DT did not have a significant effect in the range of the study. FPP increased concomitantly with SBPC, while it decreased as FM increased (Fig. 5 a). Similarly, Medina-Rendon et al. (2023) did not find any effect of temperature on the free polyphenol content. However, they analyzed the highest values of this parameter at greater and lowest levels of this variable. Natabirwa et al. (2018) did not observe any effect of the linear or quadratic terms of temperature, but they observed effect of interaction with moisture. In contrast, some studies have reported significant effect of the extrusion temperature on free phenolic compound content, in some cases, depending on the temperature range evaluated, an increase of free phenolic compounds is observed with the rise of temperature (Bisharat et al., 2015; Leyva-Corral et al., 2016; Neder-Suárez et al., 2021), which is associated to the release of compounds from the cell walls. The increase of temperature leads also to thermal degradation of the phenolic compounds (Lohani & Muthukumarappan, 2017a, 2017b; Ortiz-Cruz et al., 2020). BPP content was significantly influenced by all factors, including interactions and quadratic factors. Lower BPP content was observed at low levels of FM and DT, as well as at high levels of FM and DT. For both FPP and BPP, feed moisture exhibited a negative effect on their content. Bisharat et al. (2015), Leyva-Corral et al. (2016) and Natabirwa et al. (2018) also show a significant drop in the phenolic contents in extrudates with an increase in feed moisture from 14 to 19 %, 25 to 28 % and 15 to 20 % respectively. As was exposed, free phenolic compounds decreased as moisture content increases. Bound phenolic compounds, however, exhibited behavior that depended on the combination of

temperature and moisture. The highest values were observed at low moisture and high temperatures. In contrast, the lowest values occurred under conditions of high moisture as well as under combination of low temperature and low moisture (Fig. 5 b). Depending on the process conditions, during the extrusion cooking the destabilization of phenolic compounds could occur as well as the formation of new compounds leading to a greater extractability (Cao et al., 2021). In this sense, under high-temperature and low-moisture conditions, the energy transferred to the material was significantly higher, as previously analyzed. This increased energy within the system could promote the formation of new bonds between phenolic compounds and matrix components, potentially resulting in a higher content of bound phenolic compounds. In contrast, at lower temperatures or higher moisture levels, the probability of forming bonds between phenolic compounds and the matrix could be low due to insufficient thermal energy to break and form new bonds. Moreover, elevated moisture content may facilitate the extraction of phenolic compounds in their free forms (Ortiz-Cruz et al., 2020), which could lead to their loss or degradation. Additionally, under conditions of high moisture combined with high temperatures, the probability of thermal degradation could increase, as the presence of excess water promotes reactions such as hydrolysis and oxidation, which break down phenolic compounds and reduce their availability to form bonds with the matrix (Brennan et al., 2011; Lohani & Muthukumarappan, 2017b; Natabirwa et al., 2018).

3.4.2 Quantification of phenolic compound

The extrusion process significantly influenced the polyphenolic profile of the extrudates (Table 3) and confirmed by the ANOVA results (Table 5). The highest levels of all compounds were achieved at a die temperature of 180 °C, 20% moisture content, and 25% by-product inclusion.

Processing conditions affected the content, mean degree of polymerization, and constitutive units of proanthocyanidins (PACs) (Tables 3 and 4), as confirmed by the statistical significance of these effects (Table 4), indicating chemical modifications in the polymer structure. As expected, PACs content increased with the inclusion level of strawberry by-products. Moreover, moisture had a significant effect, unlike temperature. However, a significant interaction between moisture and temperature was observed. Higher PACs values were observed at higher moisture levels, consistent with findings reported by Hirth et al. (2015) for total proanthocyanidins in extrudates containing chokeberry extract. This could be attributed to the reduced specific mechanical energy (SME) associated with high water content during processing. Reduced SME may minimize shearing and thermal stress, thereby preventing excessive compound degradation (Lohani & Muthukumarappan, 2017b). Although higher moisture levels favored the retention of PACs, they also led to a reduction in their mean degree of polymerization (mDP). The mDP was influenced by both by-product inclusion (SBPC) and feed moisture (FM), with a strong negative correlation observed between these variables ($r = –0.806$), indicating that increasing SBPC and FM levels were associated with lower mDP values. In contrast, die temperature (DT) did not significantly affect mDP in this study, unlike the findings of Khanal et al. 2009, who reported that higher extrusion temperatures led to a marked depolymerization of procyanidins in blueberry pomace, resulting in increased monomer, dimer, and trimer contents. In the present work, the lowest mDP values were observed at the highest levels of FM and SBPC, with no consistent variation attributable to temperature. This could be linked to PACs degradation due to interflavanic bond disruption (Liang et al., 2024). Higher water content may enhance the solvation effect and increase water polarity, facilitating ionization processes and the formation of new compounds (X. Liu et al., 2021a; Lončarić et al., 2018). Such reactions may involve the release and/or rearrangement of monomers during thermal processing, as proposed by previous studies (De Taeye et al., 2014; La Mantia et al., 2023).

Both (+)-catechin (as a terminal unit) and (–)-epicatechin (as an extension unit) were influenced by all the factors studied, whereas epiafzelechin (as a terminal unit) was affected by SBPC and DT, but

not by FM. However, significant interactions between FM and DT influenced the epiafzelechin content. mDP was positively associated with the percentage of (–)-epicatechin as extension unit (r = 0.89) and negatively associated with (+)-catechin as extension unit (r = -0.79). This suggests that longer proanthocyanidin chains may be more closely linked to (–)-epicatechin incorporation, which could be associated with greater thermal stability of (–)-epicatechin compared to (+)-catechin (Ananingsih et al., 2013; Lončarić et al., 2018), which may contribute to its higher prevalence in more polymerized structures under extrusion conditions.

The extrusion process also affected (+)-catechin and (-)-epicatechin monomers (Table 3). (+)-Catechin was significantly influenced by all factors and their interactions, except for the interaction between DT and FM. Similarly, (-)-epicatechin was affected by all linear factors, however, no significant effects from the interactions were observed. For both compounds, temperature led to a reduction in their content. In contrast, as the moisture level increased, (+)-catechin content decreased, whereas (-)-epicatechin content increased. Several studies have reported changes in the ratio of these compounds during food processing (Fernández-Romero et al., 2020; Hurst et al., 2011; Kofink et al., 2007; Lončarić et al., 2018; Valverde et al., 2020). These changes could not be entirely attributed to thermal degradation but could also be influenced by modifications in proanthocyanidins (Le Bourvellec et al., 2013), changes in the extractability (Cao et al., 2021), epimerization reactions that occur at high temperatures, (+)-catechins undergo epimerization at the C-2 position in hot aqueous solutions. This process converts non-epimerized catechins into their epimerized forms and vice versa (Ananingsih et al., 2013).

Anthocyanins were strongly affected by the extrusion process (Table 3). After extrusion pelargonidin-3-*O*-glucoside was detected in only three treatments, all of them at low temperature level. The level of by-products inclusion did not significantly influence their content, probably due to the high effect of extrusion process conditions. Neder-Suárez et al. (2021) reported a decrease in anthocyanins, including cyanidin-3-*O*-glucoside and pelargonidin-3-*O*-glucoside, in extruded snacks mainly due to temperature and screw speed. Similarly, Hirth et al. (2015) show a reduction in cyanidin glycosides and total anthocyanin levels after extruding a mixture of starch and chokeberry extract. Anthocyanins are polyhydroxy and polymethoxy derivatives of 2-phenylbenzofuran cations with inherently unstable structures, making them particularly susceptible to temperature (Patras et al., 2010; Z. Wang et al., 2024), added to effect of other non-thermal factors including water, light, oxygen and pH (W. Li et al., 2021). Their degradation primarily occurs through oxidation, cleavage of covalent bonds, or accelerated oxidative reactions induced by thermal processing. The thermal breakdown of anthocyanins could lead to the formation of various degradation products, depending on the intensity and nature of the heating (Cunha et al., 2023; Enaru et al., 2021).

Among the compounds analyzed, quercetin-3-*O*-glucuronide consistently exhibited the highest content across all evaluated treatments (Table 3). In contrast, compounds such as kaempferol-3-*O*-malonylglucoside and pelargonidin-3-*O*-glucoside were not detected under certain processing conditions due to their low content and their reactivity and susceptibility during processing. Each compound responded differently to the processing parameters. *p*-coumaroylhexoside acid was significantly influenced by SBPC and its interaction with DT and FM, as well as by the interaction between DT and FM, reaching maximum value of 2.2 µg/g d.m.

Table 3. Profile of phenolic compounds (ug/g d. m.) in extrudates enriched with strawberry by-products

Run	PACs	p-C-HexA	Q	Q-3-GlcA	K-3-GlcA	K-3-MalGlc	Pg-3-Glc	(+)-Cat	(-)-Epi
1	109.0 ± 6.5^{a}	0.6 ± 0.0^{ac}	0.2 ± 0.0^{a}	3.9 ± 0.2^{a}	0.8 ± 0.0^{a}	ND	0.4 ± 0.0^{a}	2.3 ± 0.1^{ab}	1.8 ± 0.1^{a}
2	168.5 ± 8.1^{b}	2.2 ± 0.2^{b}	0.6 ± 0.0^{d}	12.8 ± 0.7^{c}	4.4 ± 0.4^{b}	0.1 ± 0.0^{a}	1.4 ± 0.1^{b}	8.3 ± 0.5^{c}	3.6 ± 0.3^{b}
3	126.8 ± 1.4^{c}	0.6 ± 0.1^{c}	0.2 ± 0.0^{a}	5.4 ± 0.4^{d}	1.0 ± 0.1^{ac}	ND	ND	3.2 ± 0.2^{de}	0.7 ± 0.0^{c}
4	162.4 ± 5.7^{b}	0.6 ± 0.0^{ac}	0.3 ± 0.0^{b}	4.8 ± 0.1^{d}	1.3 ± 0.1^{cd}	ND	ND	2.9 ± 0.1^{de}	2.2 ± 0.2^{a}
5	158.2 ± 6.1^{bd}	0.4 ± 0.0^{a}	0.2 ± 0.0^{a}	2.9 ± 0.1^{e}	1.0 ± 0.1^{ac}	ND	ND	2.7 ± 0.1^{bd}	2.8 ± 0.1^{d}
6	251.4 ± 3.9^{e}	1.3 ± 0.1^{d}	0.5 ± 0.0^{c}	7.6 ± 0.4^{f}	2.7 ± 0.1^{e}	0.1 ± 0.0^{b}	0.8 ± 0.0^{c}	3.5 ± 0.1^{e}	5.4 ± 0.3^{e}
7	160.4 ± 2.5^{bd}	0.7 ± 0.0^{c}	0.3 ± 0.0^{b}	3.8 ± 0.2^{ae}	1.5 ± 0.1^{d}	ND	ND	2.0 ± 0.1^{a}	2.2 ± 0.1^{a}
8	222.7 ± 7.6^{f}	1.4 ± 0.1^{d}	0.5 ± 0.0^{c}	7.9 ± 0.4^{f}	3.1 ± 0.2^{f}	ND	ND	2.1 ± 0.2^{ab}	4.2 ± 0.3^{f}
Central point	146.2 ± 4.4^{d}	1.0 ± 0.1^{e}	0.2 ± 0.0^{a}	3.3 ± 0.2^{ae}	1.0 ± 0.1^{ac}	ND	ND	1.7 ± 0.2^{a}	1.0 ± 0.1^{c}

PACs: Proanthocyanidins; **p-C-HexA**: *p*-coumaroylhexoside acid; **Q**: Quercetin; **Q-3-GlcA**: Quercetin-3-*O*-glucuronide; **K-3-GlcA**: Kaempferol-3-*O*-glucuronide; **K-3-MalGlc**: Kaempferol-*O*-3-malonylglucoside; **Pg-3-Glc**: Pelargonidin-3-*O*-glucoside; **(+)-Cat**: (+)-Catechin; **(-)-Epi**: (-)-Epitechin, ND: Not detected.
Different letters in the same column indicate significant differences ($p < 0,05$) by Tukey test.

Table 4. Content (µg/g d.m.), mean degree of polymerization (mDP), and proportion of constitutive units (%) of proanthocyanidins in extrudates containing strawberry by-products.

Run	mDP	% (+)-Cat T	% (-)-Epi T	% (+)-Cat E	% (-)-Epi E	% Epiafz E
1	7.6 ± 0.5^{ab}	10.3 ± 0.6^{a}	2.9 ± 0.2^{a}	31.7 ± 0.3^{a}	49.2 ± 0.5^{a}	6.0 ± 0.4^{ab}
2	6.2 ± 0.2^{c}	14.2 ± 0.5^{b}	2.0 ± 0.1^{b}	40.1 ± 1.0^{b}	38.5 ± 0.8^{bc}	5.2 ± 0.3^{c}
3	8.3 ± 0.3^{a}	6.8 ± 0.4^{c}	5.3 ± 0.1^{c}	30.6 ± 1.8^{a}	52.5 ± 2.3^{a}	4.9 ± 0.3^{cd}
4	5.6 ± 0.2^{cd}	13.4 ± 0.2^{bd}	4.5 ± 0.4^{d}	38.0 ± 0.8^{b}	41.3 ± 0.4^{b}	2.8 ± 0.1^{e}
5	6.3 ± 0.4^{ce}	12.2 ± 0.9^{d}	3.8 ± 0.3^{e}	37.5 ± 0.6^{b}	41.1 ± 0.6^{b}	5.5 ± 0.2^{bc}
6	5.2 ± 0.1^{d}	18.0 ± 0.2^{e}	1.3 ± 0.1^{f}	39.8 ± 1.8^{b}	36.0 ± 2.1^{c}	4.9 ± 0.2^{cd}
7	5.9 ± 0.2^{cd}	13.0 ± 0.4^{bd}	4.1 ± 0.2^{de}	37.7 ± 1.9^{b}	40.0 ± 1.8^{b}	5.3 ± 0.3^{bc}
8	5.3 ± 0.2^{d}	16.3 ± 0.5^{f}	2.6 ± 0.2^{ab}	38.8 ± 0.7^{b}	38.0 ± 0.9^{bc}	4.3 ± 0.2^{d}
Central point	7.0 ± 0.2^{be}	12.2 ± 0.3^{d}	2.1 ± 0.2^{b}	38.6 ± 0.2^{b}	41.0 ± 0.8^{b}	6.3 ± 0.3^{b}

mDP: Mean Degree of Polymerization; **% (+)-Cat T:** Percentage of (+)-catechin as terminal unit; **% (-)-Epi T:** Percentage of (-)-epicatechin as terminal unit; **% (+)-Cat E:** Percentage of catechin as extension unit; **% (-)-Epi E:** Percentage of (-)-epicatechin as extension unit; **% Epiafz E:** Percentage of epiafzelechin as extension unit.
Different letters in the same column indicate significant differences ($p < 0,05$) by Tukey test.

Table 5. Analysis of variance (ANOVA) *p*-value to significant effects on the individual phenolic compounds, proanthocyanidins and their mDP and constitutive units

Responses	*p*-value by source					
	SBPC	**DT**	**FM**	**SBPC x FM**	**SBPC x DT**	**FM x DT**
p-coumaroylhexoside acid	0.0007*	0.3903	0.2576	0.0014*	0.0025*	< 0.0001*
Quercetin	0.0007*	0.0211*	0.2003	0.0037*	0.0002*	< 0.0001*
Quercetin-3-*O*-glucuronide	0.0058*	0.2348	0.0549	0.0035*	0.0097*	< 0.0001*
Kaempferol-3-*O*-glucuronide	0.0004*	0.3456	0.8119	0.0095*	0.0031*	< 0.0001*
Kaempferol-3-*O*-malonylglucoside	0.0218*	0.0218*	0.0218*	< 0.0001*	< 0.0001*	< 0.0001*
Pelargonidin-3-*O*-glucoside	0.4942	< 0.0001*	< 0.0001*	< 0.0001*	< 0.0001*	< 0.0001*
(+) Catechin	0.0004*	0.0005*	0.0007*	0.0003*	0.006*	0.1617
(-) Epitechin	< 0.0001*	0.001*	< 0.0001*	0.4976	0.2839	0.4869
PACs	< 0.0001*	0.3909	< 0.0001*	0.0037*	0.0017*	0.033*
mDP	< 0.0001*	0.8096	< 0.0001*	0.317	0.0039*	0.5729
% (+)-Cat T	< 0.0001*	0.0024*	< 0.0001*	0.8555	0.3974	0.036*
% (-)-Epicat T	< 0.0001*	< 0.0001*	0.0016*	0.2151	0.0136*	0.0009*
% (+)-Cat E	< 0.0001*	0.0717	< 0.0001*	0.2956	< 0.0001*	0.2747
% (-)-Epicat E	< 0.0001*	0.006*	< 0.0001*	0.2771	< 0.0001*	0.0337*
% Epiafz E	0.0001*	0.0002*	0.2386	0.1014	0.1636	0.0093*

SBPC: Strawberry by-products content; **FM**: Feed moisture; **DT**: Die temperature; **PACs**: Proanthocyanidins; **mDP**: mean degree of polymerization
*Significant at 95 %

In the flavonol group, quercetin was affected by SBPC, DT, and their interactions, particularly between DT and FM, reaching a maximum value of 0.6 µg/g d.m. For quercetin-3-*O*-glucuronide, a conjugated form of quercetin, the maximum content was 12.8 µg/g d.m. Similar factors influenced this compound, except for the linear term of DT, which did not show a significant effect. The stability of quercetin, like other phenolic compounds, depends on environmental conditions. Additionally, the effect of temperature appears related to its molecular structure. Conjugated forms, such as quercetin-3-*O*-glucuronide, may exhibit greater thermal stability due to the presence of the additional conjugated group (Gopalakrishna et al., 2023; S. Lin et al., 2022; W. Wang et al., 2016). A similar structural differentiation was observed between kaempferol-3-*O*-glucuronide and kaempferol-3-*O*-malonylglucoside. The former was influenced by SBPC and all interactions between factors, while the latter was affected by all terms (linear and interactions). Kaempferol-3-*O*-malonylglucoside was detected only in the treatments with high inclusion of by-products and the lowest temperature. The instability of malonyl esters of kaempferol has been previously reported and is attributed to demalonation reactions, given the low stability of the malonyl group. It has been suggested that during storage or processing of vegetables, flavonoid malonylglycosides may lose carbon dioxide, potentially leading to the formation of corresponding flavonoid acetylglycosides (DuPont et al., 2000).

Table 6. Optimum extrusion process conditions and validation of responses

	Importance	Target	Optimum value	Experimental value	Desirability	p-value (*t* test)
Experimental factors						
DT (°C)	3	Optimum	192		0,732	
FM (%)	3	Optimum	18,6			
SBPC (%)	3	Optimum	16,5			
Response variables						
EI	2	Maximize	2,22	2,15 ± 0,05		0,11
BD (g/cm3)	2	Minimize	25,05	25,03 ± 0,02		0,95
H (N)	3	Range	12,77	12,71 ± 2,82		0,99
FCr (N)	3	Range	17,54	19,86 ± 1,26		0,94
NSr (mm-1)	3	Range	0,73	0,7 ± 1,14		0,07
WCr (N.mm)	2	Minimize	0,22	0,25 ± 0,09		0,56
L*	3	Range	59,96	60,61 ± 0,23		0,98
a*	3	Range	3,28	3,23 ± 0,04		0,19
b*	3	Range	6,83	6,87 ± 0,04		0,20
C*	3	Range	7,51	7,51 ± 0,04		0,97
h (°)	3	Range	64,35	65,68 ± 0,18		0,09
CI	3	Range	7,98	7,76 ± 0,1		0,13
SME (KJ/Kg)	3	Range	3584,84	3544,01 ± 429,37		0,88
FPP (mg/g)	3	Maximize	0,69	0,67 ± 0,06		0,71
BPP (mg/g)	2	Maximize	3,67	3,52 ± 0,16		0,24

SBPC: Strawberry by-products content; **DT**: Die temperature; **FM**: Feed Moisture; **EI**: Expansion index; **BD**: Bulk density; **H**: Hardness; **FCr**: Crushing force; **NSr**: Spatial frequency of ruptures; **WCr**: Crispness work; **L***: Luminosity; **a***: redness; **b***: yellowness; **C***: Chroma; **h**: Hue; **CI**: Color index; **SME**: Specific mechanical energy; **FPP**: Free phenolic compounds; **BPP**: Bound phenolic compounds
Significance level of *t*-test for one sample (p>0.05)

3.5 Multi-response optimization and validation

The process optimization was performed using an experimental design approach based on the Response Surface Methodology (RSM). This method enabled the modeling and analysis of the influence of process parameters on the evaluated responses, yielding adequate R^2 values and model fit levels (Table 6), which indicate validity and high predictive capacity.

For multi-response optimization, the desirability function was employed, which combines multiple responses into a single weighted global index. In this study, the maximization of FPP, BPP, and EI was prioritized, with varying degrees of importance assigned. Simultaneously, the minimization of WCr and BD was targeted. Similar to other studies (Lohani & Muthukumarappan, 2017b; Medina-Rendon et al., 2023; Natabirwa et al., 2018), some parameters were identified as critical attributes to obtain extrudates with suitable physicochemical characteristics.

The optimal extrusion conditions identified through the response surface methodology were DT of 192 °C, FM of 18.6%, and SBPC of 16.5%. These conditions were selected to achieve a compromise between physical quality attributes and total phenolic content, rather than maximizing individual responses. Experimental validation confirmed the model's predictive accuracy ($p < 0.05$) across the studied response variables, supporting its suitability for describing process behavior within the experimental range evaluated.

Conclusions

This study demonstrated the feasibility of incorporating strawberry by-products into tapioca starch-based extrudates, achieving improvements in their functional properties. Through the application of Response Surface Methodology (RSM), optimal extrusion conditions were identified (die temperature: 192 °C, feed moisture content: 18.6 %, inclusion level of strawberry by-products: 16.5 %), which successfully balanced desirable physical properties, such as reduced bulk density and enhanced expansion index, with the retention of bioactive compounds, particularly free and bound phenolic compounds.

The findings underscored the significant impact of extrusion parameters on both the textural and functional properties of the extrudates. The optimized combination of processing conditions favored a balance between phenolic compound retention and desirable physical attributes. The incorporation of strawberry by-products contributed to modifying texture, while also influencing the polyphenolic profile. The analysis of proanthocyanidins revealed structural modification influenced by extrusion conditions, where higher inclusion levels increased their content, but elevated moisture levels promoted the breakdown of polymeric chains, leading to a reduction in the mean degree of polymerization (mDP).

From a technological perspective, the optimized conditions allowed for a balanced product with desirable physical characteristics and a high retention of bioactive compounds. Furthermore, the extrusion parameters established in this study are compatible with standard industrial extrusion-cooking operations, facilitating potential scale-up to pilot and production levels. The process conditions identified ensure product consistency, supporting the practical applicability of this formulation within the snack industry. These findings reinforce the role of extrusion as a sustainable technology for the valorization of agro-industrial by-products and contribute to ongoing efforts to integrate functional ingredients into the development of nutritionally enhanced, market-relevant food products. This study contributes to the growing body of research advocating for the use of food by-products, promoting sustainability and circular economy practices.

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Appendix A. Supplementary data

Table S1. Effect of extrusion conditions on extrudate properties

Run	EI	BD (g/cm^3)	H (N)	FCr (N)	NSr (mm^{-1})	WCr (N.mm)	L*	a*	b*	C*	h (°)	CI	SME (KJ/Kg)	FPP (mg/g)	BPP (mg/g)
1	2.28 ± 0.15^{a}	0.25 ± 0.03^{abc}	38.46 ± 4.57^{ade}	17.53 ± 1.28^{abc}	17.09 ± 1.72^{abcdef}	1.03 ± 0.03^{ab}	58.99 ± 0.42^{a}	3.06 ± 0.04^{a}	5.92 ± 0.06^{abc}	6.66 ± 0.07^{ab}	62.64 ± 0.28^{ab}	8.76 ± 0.17^{abc}	3300.43 ± 451.90^{ab}	0.61 ± 0.01^{abc}	2.30 ± 0.11^{ab}
2	1.93 ± 0.17^{bcd}	0.33 ± 0.02^{def}	41.00 ± 5.10^{ade}	18.47 ± 1.42^{abcd}	16.32 ± 1.90^{abcdefg}	1.13 ± 0.05^{a}	55.55 ± 0.24^{b}	3.36 ± 0.01^{bh}	6.45 ± 0.02^{de}	7.28 ± 0.01^{c}	62.50 ± 0.12^{ab}	9.37 ± 0.06^{de}	2554.80 ± 114.04^{cd}	1.06 ± 0.09^{de}	2.76 ± 0.12^{acde}
3	2.10 ± 0.14^{acde}	0.22 ± 0.02^{a}	25.94 ± 3.54^{a}	13.36 ± 2.03^{b}	18.28 ± 1.61^{abcef}	0.73 ± 0.05^{c}	59.20 ± 0.17^{ag}	3.29 ± 0.03^{bc}	6.62 ± 0.05^{e}	7.39 ± 0.06^{c}	63.58 ± 0.11^{cde}	8.40 ± 0.06^{cf}	3469.91 ± 445.14^{a}	0.74 ± 0.03^{fg}	3.06 ± 0.32^{cde}
4	1.83 ± 0.14^{bf}	0.30 ± 0.03^{bcde}	33.16 ± 4.25^{ad}	15.23 ± 1.49^{bc}	23.25 ± 2.66^{f}	0.66 ± 0.01^{c}	55.52 ± 0.45^{b}	3.82 ± 0.03^{d}	7.25 ± 0.13^{f}	8.19 ± 0.13^{d}	62.22± 0.25^{a}	9.49 ± 0.17^{de}	2879.03 ± 288.16^{bd}	1.16 ± 0.07^{d}	5.02 ± 0.59^{h}
5	1.62± 0.16^{fg}	0.41 ± 0.02	62.32 ± 11.92^{b}	28.32 ± 2.99^{ad}	15.99 ± 2.22^{abcdeg}	1.77 ± 0.06^{d}	53.85 ± 0.45^{c}	2.74 ± 0.06^{e}	5.22 ± 0.15^{gh}	5.90 ± 0.15^{ef}	62.26 ± 0.37^{a}	9.76 ± 0.21^{eg}	2252.80 ± 40.33^{cef}	0.57 ± 0.04^{ab}	3.24 ± 0.17^{ce}
6	1.28 ± 0.10^{hi}	0.51 ± 0.04^{g}	56.64 ± 10.84^{bef}	30.74 ± 3.78^{d}	14.01 ± 1.69^{cdeg}	2.19 ± 0.01^{e}	49.62 ± 0.31^{de}	2.47 ± 0.06^{f}	5.26 ± 0.18^{gh}	5.81 ± 0.18^{e}	64.82 ± 0.45^{fg}	9.47 ± 0.26^{de}	2160.95 ± 202.67^{cef}	0.95 ± 0.03^{eh}	2.30 ± 0.19^{ab}
7	1.43 ± 0.13^{gh}	0.80 ± 0.05^{i}	117.65 ± 17.26^{c}	45.43 ± 7.85^{e}	10.59 ± 1.24^{dg}	4.29 ± 0.24^{f}	52.00 ± 0.32^{f}	2.32 ± 0.07^{f}	5.21 ± 0.14^{g}	5.74 ± 0.15^{e}	65.26 ± 0.34^{g}	8.58 ± 0.14^{acf}	1977.76 ± 138.67^{ef}	0.50 ± 0.04^{bi}	2.17 ± 0.04^{ab}
8	1.38 ± 0.10^{h}	0.77 ± 0.05^{h}	133.68 ± 22.76^{c}	56.62 ± 5.97^{ef}	9.89 ± 1.50^{g}	5.73 ± 0.28^{g}	49.29 ± 0.34^{d}	2.15 ± 0.06^{g}	5.27 ± 0.14^{gh}	5.70 ± 0.14^{e}	67.84 ± 0.28^{i}	8.26 ± 0.12^{f}	2120.49 ± 274.22^{cef}	0.82 ± 0.03^{fj}	2.41 ± 0.12^{abd}
9	2.26 ± 0.19^{ae}	0.24 ± 0.02^{ab}	38.18 ± 5.50^{ade}	19.32 ± 2.55^{abcd}	12.35 ± 1.53^{cdg}	1.56 ± 0.01^{h}	59.65 ± 0.41^{g}	2.72 ± 0.06^{e}	5.53 ± 0.11^{hi}	6.23 ± 0.12^{fg}	64.91 ± 0.32^{g}	8.25 ± 0.15^{f}	3112.40 ± 390.53^{ab}	0.42 ± 0.0045^{i}	3.45 ± 0.14^{ef}
10	1.83 ± 0.11^{bf}	0.31 ± 0.02^{cdef}	46.66 ± 3.58^{bdef}	24.11 ± 2.31^{abc}	15.07 ± 2.74^{acdeg}	1.60 ± 0.14^{h}	53.15 ± 0.50^{h}	3.30 ± 0.09^{bc}	6.61 ± 0.12^{e}	7.39 ± 0.15^{c}	64.55 ± 0.21^{fgh}	9.39 ± 0.04^{ce}	2335.18 ± 102.24^{cf}	0.97 ± 0.07^{eh}	4.21 ± 0.23^{g}
11	1.85 ± 0.09^{bf}	0.36 ± 0.05^{bcde}	41.58 ± 4.69^{ade}	21.00 ± 1.82^{abc}	20.98 ± 1.70^{abf}	1.00 ± 0.01^{ab}	55.72 ± 0.23^{b}	3.25 ± 0.07^{c}	5.74 ± 0.13^{aci}	6.60 ± 0.14^{ab}	60.51 ± 0.16j	10.15 ± 0.08^{g}	2557.06 ± 348.51^{cd}	0.54 ± 0.03^{abi}	1.76 ± 0.13^{b}
12	1.66 ± 0.13^{f}	0.58 ± 0.04^{g}	60.15 ± 4.57^{bf}	27.29 ± 4.22^{ac}	15.99 ± 2.32^{abcdeg}	1.71 ± 0.02^{dh}	54.54 ± 0.36^{i}	2.83 ± 0.03^{e}	5.82 ± 0.07^{aci}	6.47 ± 0.08^{ag}	64.04 ± 0.17^{cfh}	8.93 ± 0.06^{abh}	2552.53 ± 136.48^{cd}	0.56 ± 0.03^{ab}	2.70 ± 0.11^{acd}
13	2.17 ± 0.14^{ade}	0.29 ± 0.02^{abcd}	27.27 ± 5.55^{ad}	13.46 ± 1.98^{b}	18.82 ± 2.44^{abcef}	0.72 ± 0.01^{c}	58.86 ± 0.51^{a}	3.47 ± 0.07^{h}	7.00 ± 0.08^{f}	7.81 ± 0.11^{h}	63.65 ± 0.32^{cde}	8.41 ± 0.19^{f}	3062.30 ± 236.41^{ab}	0.87 ± 0.03^{hj}	4.14 ± 0.07^{fg}
14	1.09 ± 0.10^{i}	0.82 ± 0.08^{hi}	133.72 ± 23.11^{c}	59.45 ± 10.45^{f}	10.89 ± 1.65^{dg}	5.46 ± 0.14^{i}	50.16 ± 0.50^{e}	2.07 ± 0.04^{g}	4.78 ± 0.05^{j}	5.20 ± 0.05^{i}	66.62 ± 0.36^{k}	8.62 ± 0.21^{acf}	1812.88 ± 200.09^{e}	0.56 ± 0.04^{ab}	2.78 ± 0.29^{acde}
15	1.92 ± 0.07^{bc}	0.36 ± 0.03^{ef}	42.32 ± 5.59^{adef}	19.35 ± 3.00^{abcd}	20.25 ± 3.00^{aef}	0.96 ± 0.01^{b}	55.53 ± 0.33^{b}	2.94 ± 0.03^{a}	5.99 ± 0.04^{abc}	6.67 ± 0.05^{ab}	63.81 ± 0.25^{cdh}	8.85 ± 0.15^{ab}	2411.34 ± 368.34^{cdf}	0.63 ± 0.02^{acg}	2.65 ± 0.26^{ac}
16	1.95 ± 0.18^{bcd}	0.31 ± 0.03^{cdef}	40.87 ± 4.74^{ade}	19.11 ± 2.44^{abcd}	19.50 ± 2.87^{abef}	0.98 ± 0.02^{b}	55.87 ± 0.20^{b}	3.07 ± 0.03^{a}	6.16 ± 0.11^{bd}	6.88 ± 0.11^{b}	63.53 ± 0.21^{cde}	8.91 ± 0.10^{abh}	2493.66 ± 316.49^{cdf}	0.61 ± 0.02^{abc}	2.44 ± 0.19^{abd}
17	2.04 ± 0.09^{bcde}	0.34 ± 0.02^{def}	37.71 ± 5.60^{ade}	18.72 ± 2.85^{abcd}	20.69 ± 2.18^{abef}	0.90 ± 0.04^{b}	57.00 ± 0.20^{b}	3.11 ± 0.04^{a}	6.17 ± 0.04^{bd}	6.91 ± 0.05^{b}	63.21 ± 0.21^{bde}	8.86 ± 0.05^{ab}	2546.25 ± 258.78^{cd}	0.71 ± 0.004^{cfg}	2.65 ± 0.14^{acd}
18	1.91 ± 0.12^{bc}	0.31 ± 0.02^{cdef}	37.60 ± 6.16^{ade}	18.97 ± 1.70^{abcd}	20.11 ± 2.43^{abef}	0.94 ± 0.03^{b}	57.16 ± 0.07^{b}	3.15 ± 0.01^{a}	6.04 ± 0.01^{abc}	6.82 ± 0.01^{b}	62.46 ± 0.09^{ab}	9.13 ± 0.05^{bch}	2387.92 ± 175.21^{cdf}	0.61 ± 0.02^{abc}	2.78 ± 0.34^{acde}
19	1.95 ± 0.07^{bcd}	0.36 ± 0.03^{ef}	38.99 ± 6.40^{ade}	19.16 ± 3.42^{abcd}	22.04 ± 2.97^{bf}	0.87 ± 0.04^{b}	57.08 ± 0.13^{b}	3.09 ± 0.01^{a}	6.07 ± 0.07^{ab}	6.82 ± 0.07^{b}	62.96 ± 0.30^{abe}	8.93 ± 0.13^{abh}	2467.54 ± 246.37^{cdf}	0.56 ± 0.05^{ab}	2.65 ± 0.15^{acd}

Table S1. Effect of extrusion conditions on extrudate properties

Run	EI	BD (g/cm^3)	H (N)	FCr (N)	NSr (mm^{-1})	WCr (N.mm)	L*	a*	b*	C*	h (°)	CI	SME (KJ/Kg)	FPP (mg/g)	BPP (mg/g)
20	1.86 ± 0.16^{bcf}	0.38 ± 0.02^{f}	39.67 ± 4.44^{ade}	19.09 ± 2.68^{abcd}	20.62 ± 3.17^{abef}	0.93 ± 0.01^{b}	56.05 ± 0.40^{b}	3.07 ± 0.09^{a}	5.90 ± 0.13^{abc}	6.65 ± 0.15^{ab}	62.48 ± 0.17^{b}	9.29 ± 0.02^{ch}	2437.37 ± 125.56^{cdf}	0.60 ± 0.03^{abc}	2.47 ± 0.13^{a}

EI: Expansion Index; BD: Bulk density; H: Hardness; FCr: Crushing Force; NSr: Number of spatial ruptures; WCr: Crispness Work; L*, a*, b*, C*, h: Color parameters; CI: Color Index; SME: Specific Mechanical Energy; FPP: Free Phenolic compounds; BPP: Bound Phenolic compounds. Different letters in the same column indicate significant differences ($p < 0,05$) by Tukey test.

Fig. S1. Color contrast in selected extruded snack samples

Chapter 4. Comprehensive study of the effect of extrusion on fiber and phenolic compounds in whole and fractionated strawberry by-products in tapioca starch-based extrudates

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Abstract

Strawberry by-products (SBP), generated during juice processing, are composed of a complex matrix rich in dietary fiber and phenolic compounds. Their incorporation into starch-based extruded formulations offers an opportunity to develop value-added foods with enhanced functional properties. This study aimed to evaluate the modifications of phenolic compounds and cell wall components from SBP under extrusion processing conditions, as well as their interactions within starch-based matrices. For this purpose, distinct extruded formulations were produced using SBP as a whole or cell wall material and phenolic compound extract isolated from SBP, combined with tapioca starch. Extrusion significantly reduced total phenolic compound content, particularly anthocyanins and flavonoids, although specific compounds such as kaempferol-3-*O*-glucuronide and (-)-epicatechin increased depending on the processing conditions. The presence of fiber favored phenolic compound retention especially proanthocyanidins with high degree of polymerization due to cell wall specificity under specific conditions. Fiber composition analysis revealed structural alterations mainly in pectin rhamnogalacturonans, while hemicelluloses and cellulose were resistant to extrusion. Differences in pectin methylation degree and lignin content suggested matrix-dependent rearrangements and possible phenolic compound -fiber interactions. FTIR and SEM confirmed structural modifications and indicated that both fiber and phenolic compounds influenced starch reorganization and the microstructure of extrudates. Overall, this study highlights the importance of matrix composition in modulating the behavior of SBP components during extrusion and provides a foundation for designing functional extruded products enriched with fruit by-products.

Keywords: processing, polysaccharides, proanthocyanidins, interactions

1 Introduction

Extrusion is a versatile industrial technique that allows the formulation of various ready-to-eat products, such as snacks and breakfast cereals, which are highly consumed, particularly by younger generations (Arribas et al., 2019). It is a continuous cooking and molding process that involves short processing times, high temperatures, and a combination of factors such as moisture, pressure, screw speed, residence time, and mechanical shearing, among others. These conditions facilitate the cooking of starch-rich foods and induce physicochemical modifications of raw materials, resulting in products with new textural, functional, and structural properties (Alam et al., 2016; Medina-Rendon et al., 2023). Extrusion conditions, starch granule characteristics, and the presence of other components such as proteins, fibers, and sugars directly influence the extent of transformation. During extrusion process, structural modifications of fiber, starch gelatinization, interactions among food components, and the degradation or modification of bioactive compounds and/or formation of process-induced compounds may occur (Alam et al., 2016).

The value addition of extruded products involves the incorporation of ingredients to enhance their textural and functional properties. In some cases, extrusion technology may even improve the bioavailability of bioactive compounds. Efforts have been made to enrich extruded snacks with fruit and vegetable by-products to increase their fiber and polyphenol content (Grasso, 2020; Offiah et al., 2019; Patil & Kaur, 2018). Strawberry by-products, which are generated during juice production, have been identified as a promising source of functional ingredients due to their high content of dietary fiber and phenolic compounds, particularly flavonoids such as proanthocyanidins, and phenolic acids, known for their antioxidant and bioactive properties (Pukalskienė et al., 2021). However, the stability and functionality of these compounds within extruded products remain poorly characterized, especially regarding their interactions with other food matrix components. Previous studies have reported modifications in fiber and the stability of phenolic compounds after extrusion

of different plant-based matrices (Hirth et al., 2015; Neder-Suárez et al., 2021; M. Zhang et al., 2011), but most of these studies focus on analyzing changes in raw materials or evaluating individual components without establishing relationships between the main fractions that constitute a given material.

Strawberry by-products consist primarily of cell wall polysaccharides and phenolic compounds. Snack production involves both a formulation step and the extrusion process. Therefore, a deeper understanding of how these components, namely fiber and phenolic compounds, are transformed during extrusion is required. In this context, the aim of this study was to determine whether extrusion conditions, the structural nature and composition of dietary fiber and phenolic compounds, and/or their interactions influence both the processing-induced modifications and the functional behavior of these components within the matrix. The originality of this work lies not only in comparing native and extruded strawberry by-product-based snacks, but also in the use of isolated phenolic compounds and purified cell wall fractions, which were incorporated into extruded formulations both individually and in combination. This experimental approach enabled the assessment of how thermal processing and the presence of these components, either alone or together, affect their structural integrity and interactions during extrusion. To achieve this, cell wall material and phenolic extracts were first isolated and purified from strawberry by-products and then combined with tapioca starch to create composite feed matrices. By gaining a deeper understanding of the modification/degradation that occur in the structural components of strawberry by-products during extrusion, this study provides insights into their stability and functionality in extruded food systems. The findings could contribute to the development of formulations with enhanced structural integrity and added value, guiding future research on fiber- and phenolic compound-rich ingredients in extruded products.

2 Materials and methods

2.1 Raw materials

Strawberry (*Fragaria × ananassa*) by-products (SBP) used in this study were obtained from pulp extraction process and provided by Monkeyfruit S.A. (Popayán, Colombia). The fruits of the Sabrina variety were sourced from local cultivars in the municipality of Sotará, Cauca. The SBP were frozen at -80 °C for 12 h and then were dried in a vacuum freeze dryer (YR05188, Kalstein, Paris, France) for 72 h. The dried SBP were grinded (MF 10 basic Molino IKA, Germany) rapidly and the powder was vacuum sealed and stored at -20 °C until use. Tapioca (*Manihot esculenta*) starch was supplied by Ingredion Colombia S.A. (Cali, Colombia).

2.7 Standards and chemicals

All reagents were of analytical grade, HPLC solvents were of chromatographic purity, and water was purified through deionization using a Milli-Q system (Millipore, Bedford, MA, USA). Folin-Ciocalteu reagent and sodium carbonate were obtained from Panreac (Barcelona, Spain). Ethanol and acetone were from Fisher Scientific (Strasbourg, France). Acetonitrile of HPLC grade was obtained from VWR International (Radnor, USA). Hexane, methanol, hydrochloric acid and acetic acid were from Merck (Darmstadt, Germany). Sugar standards (arabinose, fucose, galactose, xylose, mannose and rhamnose) were from Fluka (Buchs, Switzerland). Formic acid, sodium carbonate, sodium hydroxide, NaBH4, N-methylimidazole, acetic anhydride, (+)-catechin, (-)-epicatechin, caffeic acid, para-coumaric acid, quercetin, kaempferol, pelargonidin-3-*O*-β-glucoside, cyanidin-3-*O*-β-glucoside, acetyl chloride, menthofuran 95%, inositol and galacturonic acid were provided by Sigma-Aldrich (Saint Quentin Fallavier, France). Methanol-d3 was from Acros Organics (Geel, Belgium).

2.2 Preparation of cell wall material and phenolic compound extract

Cell wall (CW) was prepared from strawberry by-products following the method described by Le Bourvellec et al. (2011). Briefly, 100 g of strawberry by-product powder was washed with 70% ethanol and extracted at room temperature until the absence of sugars was confirmed by a negative reaction in the phenol-sulfuric acid test. The samples were then sequentially washed twice with a 60% acetone-water solution, once with an 80% acetone-water solution, and finally with pure acetone until the supernatant became colorless. The residue was dried at 40 °C for 24 h and subsequently weighed, yielding 54.7% on a dry basis. This yield was later taken into account for the formulation of the matrices containing cell wall material.

The washing liquid was recovered, concentrated using a rotary evaporator, and subsequently purified. For purification, the extracts were dissolved in water acidified with 0.01 M HCl at a 1:10 ratio (Hejniak et al., 2019) and passed through a Sep-Pak C18 cartridge. The cartridge was pre-conditioned with 8 mL of methanol, followed by 4 mL of pH 7.0 water and 5 mL of 0.01 M HCl. The sample was then washed with 10 mL of pH 7.0 water and eluted with 12 mL of methanol to obtain the phenolic compound extract (Sarria Villa et al., 2021). The resulting methanolic extract was freeze-dried and subsequently reconstituted in water for use in the extrusion formulations. The concentration of total phenolic compounds in the reconstituted extract was determined using the Folin–Ciocalteu method, yielding 2.64 mg GA/mL. This concentration was later used to determine the volume of extract to be incorporated into the phenolic-enriched matrices.

2.3 Preparation of matrices for extrusion

Different matrices were prepared and used to evaluate the impact of the extrusion process, an extruded snack formulation on both starch, fiber and phenolic compounds (Table 1). Composite matrices consisting of various combinations of tapioca starch, cell wall material, and phenolic compound extract were prepared. The extruded snack (E-SBPTs) was formulated by mixing tapioca starch with 16.46% (d.m.) matter) strawberry by-products and processed at a moisture content of 18.64%, a condition previously identified through optimization studies to improve both processing performance and product quality. In addition, a non-extruded matrix with the same formulation (NEM) was included as a control of the extrusion process.

Table 1. Description of the samples analyzed in the study

Sample Code	Sample description
Ts	Native tapioca starch (control)
SBP	Strawberry by-products (pomace), non-extruded
NEM	Non-extruded mixture of strawberry by-products and tapioca starch
E-Ts	Extruded tapioca starch (control)
E-SBP	Extruded strawberry by-products
E-SBPTs	Extruded snack, composite of strawberry by-products and tapioca starch
E-PPCWTs	Extruded composite matrix: phenolic extract + cell wall (from SBP) + tapioca starch
E-CWTs	Extruded composite matrix: cell wall (from SBP) + tapioca starch
E-PPTs	Extruded composite matrix: phenolic extract (from SBP) + tapioca starch

Composite matrices were formulated using tapioca starch. One matrix consisted of tapioca starch with cell wall material extracted from strawberry by-products (E-CWTs), another combined tapioca starch with a purified extract of phenolic compounds obtained from strawberry by-products (E-PPTs), and a third included both cell wall material and phenolic extract along with tapioca starch (E-PPCWTs). The amount of each component was calculated to replicate the contribution of cell wall and phenolic compounds present in a formulation containing 16.5% (d.m.) strawberry by-products, as used in the E-SBPTs snack. Specifically, the amount of cell wall material was adjusted based on its extraction

yield (54.7%), while the volume of phenolic extract was determined according to its total phenolic concentration (2.64 mg GA/mL), ensuring equivalent polyphenol input across formulations.

Furthermore, strawberry by-products were extruded alone at 18.6% moisture content (E-SBP), enabling the evaluation of structural and chemical changes induced by processing. In parallel a matrix containing only extruded tapioca starch (E-Ts) was prepared as a control to determine the influence of the other components on the evolution of starch during the extrusion process. Due to the fine texture of these materials, the composite matrices were processed at 30% moisture content, as lower moisture levels hindered their proper passage through the extruder. Each formulation was thoroughly mixed and adjusted to the target moisture content, followed by blending for ten minutes at medium speed using a KP26M1XER commercial mixer (KitchenAid, MI, USA). The prepared mixtures were then packed in polyethylene bags and stored for 12–14 hours to ensure moisture equilibration and homogeneity before extrusion.

2.4 Extrusion process

Extrusion cooking was carried out using a laboratory-scale co-rotating twin-screw extruder (HAAKE™ Rheomex OS, ThermoFisher Scientific, Germany) with an 11 mm screw diameter, a length-to-diameter ratio of 40:1, and a 3 mm die opening. The final barrel zones and the die were set at temperatures of 140/160/180/192 °C. Screw speed and feed rate were set at 140 rpm and 20 g/min, respectively. The extrudates were dried at 60 °C for 30 minutes, packed in polyethylene bags, and stored in airtight containers until further physical analysis. For chemical analysis, the extruded samples were ground using a laboratory grinder (A 11 basic IKA mill, Germany) and passed through a mesh-60 sieve.

2.5 Surface morphology analysis by scanning electron microscopy (SEM)

Samples were mounted on SEM specimen stubs using a double-sided carbon-conductive adhesive tape before coating. A 20 nm gold layer was then deposited on the samples by ion sputtering using a Quorum Q300TD sputter coater (Quorum Technologies, Laughton, East Sussex, UK). Imaging was performed with a JEOL JSM-7100F scanning electron microscope (JEOL Ltd., Tokyo, Japan) operated at an accelerating voltage of 20 kV. ImageJ 1.54g software (National Institutes of Health, Bethesda, MD, USA) was used for processing SEM micrographs.

2.6 ATR-FTIR spectra

The spectral data of cell walls and solubilized polysaccharides were acquired using a Tensor 27 FTIR spectrometer (Bruker Optics®, Wissembourg, France). ATR-FTIR spectra were recorded at room temperature on powdered samples using a single-reflectance horizontal ATR cell (Golden Gate equipped with a diamond crystal, Bruker Optics). The measurements were conducted as described by Liu, Renard, Rolland-Sabaté, Bureau, et al. (2021), scanning in the range of 4000 to 600 cm^{-1}, with background correction against air. Each sample was analyzed in triplicate, and each spectrum was obtained by averaging 16 scans. Data were processed using Origin software (version 2018, OriginLab Corporation, Northampton, MA, USA).

2.7 Quantification of phenolic compounds

Identification of phenolic compounds was performed using HPLC coupled to ElectroSpray Ionisation Mass Spectrometry (HPLC/ESI-MS^2) analysis was performed on an Acquity Ultra performance LC (UPLC) apparatus from Waters (Milford, MA, USA), equipped with a photodiode array detector coupled with a Bruker Daltonics (Bremen, Germany) HCT ultra ion trap mass spectrometer with an

electrospray ionization source. Separations were achieved using a Luna Omega Polar C18 column (50 mm × 2.1 mm × 3 µm column, Phenomenex, Torrance, USA) operated at 30 °C. The mobile phase consisted of water/formic acid (98,2, mL/mL) (eluent A) and acetonitrile (eluent B). The flow rate was 1 mL/min. The elution program was as follows: 3–9 % B (0–5 min); 9–16 % B (5–15 min); 16–50 % B (15–45 min); 50–90 % B (45–48 min); 90–90 % B (48–52 min). 10 µL of samples ("furanolysis" and "crude" extracts) were injected. For polyphenol characterization, a capillary voltage of 2 kV was used in the negative ion mode. N2 was used as drying and nebulizing gas with a flow rate of 12 L/min. The desolvation temperature was set at 365 °C and the nebulization pressure at 0.4 MPa. The ion trap was operated in Ultrascan mode from m/z 100 to 1000. For anthocyanin characterization, a capillary voltage of 1.8 kV was used in the positive ion mode under the same conditions. Phenolic compounds were analyzed and quantified by HPLC-DAD (Prominence system, Shimadzu, Kyoto, Japan) and separations were achieved as described in Rincon et al. (2025). Analyses of phenolic compounds were carried out with or without the acidic depolymerization of proanthocyanidins (PACs) in the presence of menthofuran (named furanolysis). PACs were thus characterized by their subunit composition, average degree of polymerization (mDP) and contents. HPLC-DAD analyses of crude methanolic extracts (not submitted to furanolysis) were performed to titrate monomeric catechins.

Furanolysis and methanol extract were obtained as described in Rincon et al. (2025). The most prevalent compounds were quantified in the UV–visible region by chromatographic analysis conducted at wavelengths of 280 nm (flavanols), 320 nm (phenolic acids), 360 nm (flavonols), and 520 nm (anthocyanins). Individual compounds were quantified in mg/100 g of fresh weight (FW) by comparison with external standards: (+)-catechin and (−)-epicatechin for flavanols, caffeic and para-coumaric acids for phenolic acids, quercetin and kaempferol for flavanols, and pelargonidin-3-O-β-glucoside and cyanidin-3-O-β-glucoside for anthocyanins.

The mean degree of polymerization (mDP) of proanthocyanidins as well as their subunits composition was calculated as the molar ratio of all flavan-3-ol units (i.e., furan adducts plus terminal units, minus monomers from crude methanol extract) to the sum of terminal units, minus monomers from crude methanol extract (X. Liu, Renard, et al., 2021).

2.8 Analysis of fiber composition

The matrices containing strawberry by-products and cell wall material were characterized for their cell wall composition, including neutral sugars, galacturonic acid, methanol content, degree of methylation, and lignin content, as described in the following sections. For this purpose, the cell wall material from each sample was re-extracted using the procedure previously described. Additionally, samples containing starch underwent a de-starching process before analysis.

2.8.1 De-starching of cell wall material of the samples

The re-extracted cell wall material was de-starched following the protocol adapted from the starch assay method (Mccleary et al., 1997). Briefly, 8 g of the sample were suspended in 200 mL of deionized water and stirred for one hour at room temperature. Subsequently, 1.6 mL of thermostable α-amylase (60 U) was added, and the mixture was incubated at 95 °C for 12 min. After cooling to 70 °C, the pH was adjusted to 4.5 using 25 mL of 0.2 M acetate buffer (pH 4.5). Then, amyloglucosidase (40 U) was added, and the mixture was incubated at 50 °C for 3 h. After cooling, the pH was adjusted to 7.0 by adding 1 M NaOH, followed by the addition of 1.6 mL of subtilisin A. The mixture was maintained in an oven at 35 °C with continuous stirring for one hour. Subsequently, 480 mL of 96% ethanol were added, and the mixture was stirred overnight at 4 °C. The mixture was centrifuged (6000g, 10 min), and the pellet was washed with 70% ethanol three times. The mixture was

centrifuged (6000g, 10 min), and the pellet was washed three times with 70% ethanol. Finally, the de-starched material was dried overnight in an oven at 37 °C. The complete removal of starch was verified using the Total Starch Assay Kit (AA/AMG) (Megazyme®) before proceeding with further analyses.

2.8.2 Hydrolysis

To determine cellulosic glucose content, neutral sugars, including non-cellulosic glucose (NCGlc) and cellulosic glucose (CGlc), were analyzed as alditol acetates following acid hydrolysis in two different approaches. For cellulose analysis, samples were pre-hydrolyzed with 250 µL of 72% sulfuric acid at room temperature for 1 hour, according to Saeman method (Saeman et al., 1954). After pre-hydrolysis, the mixture was diluted to 2 M sulfuric acid by adding deionized water, followed by the addition of 1 mL of an internal standard (inositol), and incubated at 100 °C for 3 hours. For the characterization of NCGlc, no pre-hydrolysis was performed; instead, a direct hydrolysis approach was applied by adding 2 M sulfuric acid immediately after the internal standard, followed by incubation at 100 °C for 3 hours. The content of cellulosic glucose (CGlc) was determined by calculating the difference between glucose levels obtained with and without pre-hydrolysis (X. Liu, Renard, et al., 2021).

2.8.3 Neutral sugars composition

Neutral sugars were analyzed as alditol acetates (Canteri et al., 2019). The samples were injected into a GC-FID HP 5890 Series II (Agilent, Inc., Palo Alto, USA) equipped with a capillary column (30 m × 0.25 mm i.d., coated with DB-225 MS, 0.25 µm film thickness). The chromatographic conditions were as follows: split mode injection (1:25 ratio), injector temperature set at 250 °C, hydrogen as the carrier gas at 45 cm/s (215 °C), a column flow rate of 1.3 mL/min, and an oven temperature of 215 °C (isothermal).

2.8.4 Galacturonic acid content

Uronic acids were quantified using the meta-hydroxyl-diphenyl assay, as described by (Canteri et al., 2019), following the pre-hydrolysis step. Absorbance was measured at 520 nm using a spectrophotometer (V-530 Jasco, Tokyo, Japan), and concentrations were determined using a calibration curve with galacturonic acid as the external standard.

2.8.5 Methanol and degree of methylation

Samples of 10 mg were suspended in 3.8 mL of distilled water and subsequently saponified by adding 0.8 mL of 1 M KOH containing CD_3OH (7 mM) as an internal standard, followed by incubation at room temperature for 1 hour. A calibration curve was prepared using methanol in the range of 0.6–6 mM, with CD_3OH as an internal standard at 1.4 mM. Methanol content was determined by stable isotope dilution assay using headspace-GC-MS (QP2010 Shimadzu, Kyoto, Japan), as described by (C. M. G. C. Renard & Ginies, 2009). The degree of methylation (DM) was calculated as the molar ratio of methanol to galacturonic acid.

2.8.6 Lignin content

Lignin content was determined spectrophotometrically following the method described by Liu, Renard, Rolland-Sabaté, Bureau, et al. (2021). Briefly, 15 mg of cell wall samples were digested in 1 mL of 25 % acetyl bromide in acetic acid containing 2.7 % (v/v) perchloric acid and incubated at 70 °C for 30 min. After cooling, 10 µL of the digest was transferred into a test tube, followed by the

addition of 570 µL of 17 % 2 N NaOH and 83% acetic acid. To stop the reaction, 20 µL of 7.5 M hydroxylamine hydrochloride was added. The final volume was adjusted to 2 mL with acetic acid, and the absorbance was measured at 280 nm using a V-530 spectrophotometer (Jasco, Tokyo, Japan). Lignin content was quantified based on a linear calibration curve using commercial alkali lignin as a standard.

2.9 Statistical analysis

Statistical analyses were performed with at least three replicates. Results were expressed as mean value ± standard deviation and statistically evaluated for differences between means using either a T-test or Tukey's test, as appropriate, at a 95% confidence interval using IBM® SPSS® Statistics (Version 27, IBM Corp., Armonk, NY, USA).

3 Results and discussion

3.1 SEM

In the non-extruded mixture (NEM) (Fig. 1a-b), spherical and smooth starch granules were observed, with no evidence of gelatinization. Some granules exhibited surface deposits, possibly due to interactions with components from strawberry by-products, similar to what has been reported for starch treated with pectin (Y. Zhang et al., 2021). After extrusion, significant changes in the internal microstructure of the extruded products were observed. These changes could be attributed to the high temperature, pressure, and shear stress inside the extruder, which may induce starch gelatinization and dextrinization, disrupting the granular structure and altering its amorphous state (Neder-Suárez et al., 2024). Although these modifications varied depending on the composition of each matrix, they generally led to the breakdown of starch granules and the formation of heterogeneous, scratched, cracked, and irregular structures, as previously reported in extrudates containing plant-based ingredients (H. Chen et al., 2018; Neder-Suárez et al., 2024; Samyor et al., 2018). The extruded snack (E-SBPTs) (Fig. 1c) exhibited a highly expanded and porous structure, characterized by thin laminar formations and internal cavities, typical of highly expanded extruded materials with extensive starch gelatinization (Hashemi et al., 2017; Wani & Kumar, 2019).

In contrast, the extrudates E-Ts, E-CWTs, E-PPTs, and E-CWPPTs (Fig. 1d-g) displayed denser and more fractured structures, suggesting more limited expansion, which may be attributed to the higher moisture content of these formulations. The extruded tapioca starch (E-Ts) (Fig. 1d) exhibited a compact and dense structure with fractured and collapsed regions and no significant porosity. This may suggest that in the absence of additional structural components, starch underwent complete gelatinization, but its post-extrusion reorganization favored a more compact structure.

The extruded matrix containing cell wall material (E-CWTs, Fig. 1e) exhibited areas with internal cavities and heterogeneous fragmentation, indicating that cell wall polysaccharides might have influenced starch cohesion, leading to a more irregular structure. The extrudate containing phenolic compound extract (E-PPTs, Fig. 1f) displayed a highly compact structure with layers organized into parallel planes and clean fractures, indicating greater structural cohesion. Phenolic compounds may have promoted intermolecular interactions that increased the rigidity of the material, reducing expansion and favoring a denser matrix. Finally, the extrudate containing starch, cell wall and phenolic compounds (E-CWPPTs, Fig. 1g) exhibited a less compact structure compared to E-PPTs, with a more heterogeneous arrangement, increased fragmentation, and the presence of cracks. Despite the potential restriction in expansion due to the interactions between cell wall and phenolic compounds, the structure suggests that internal material cohesion was lower, likely due to the interference of cell wall polysaccharides in the organization of starch and phenolic compounds.

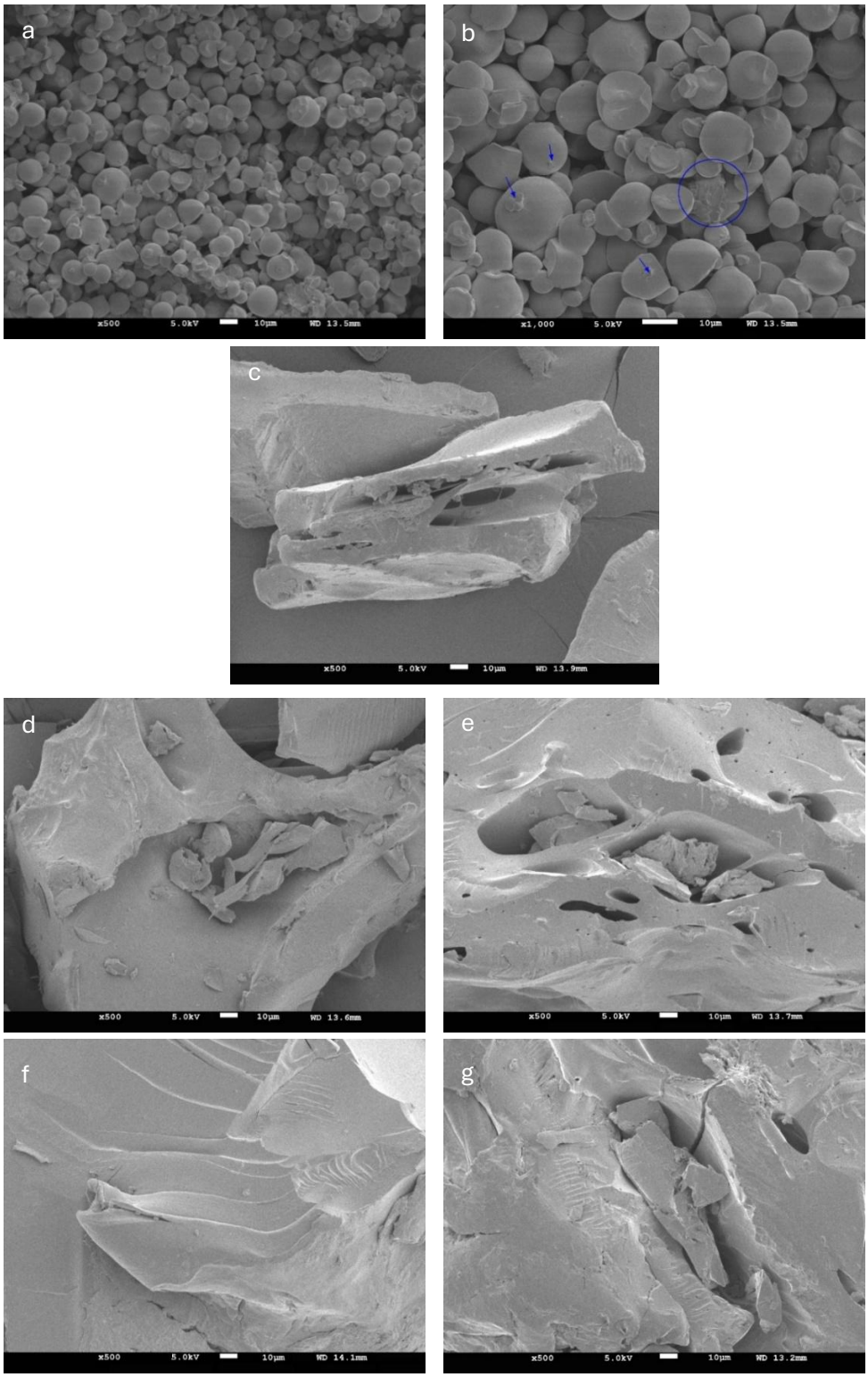

Fig. 1. Scanning electron micrographs of (a, b) non-extruded mixture (NEM) ; (c) extruded snack (E-SBPTs); (d) extruded tapioca starch (E-Ts); (e) extruded with tapioca starch and cell wall material of strawberry by-products (E-CWTs); (f) extruded with tapioca starch and polyphenol extract of by-products strawberry (E-PPTs); (g) extruded with tapioca starch, cell wall and polyphenol extract of strawberry by-products (E-CWPPTs)

3.2 FTIR analysis

The spectrum of SBP was compared with that of E-SBP (Fig. 2a). The extruded snack (E-SBPTs) was analyzed in comparison to both the mixture without extrusion process (NEM) and tapioca starch (Ts) (Fig. 2b). The composite mixtures (E-CWTs, E-PPTs, E-CWPPTs) were compared with NEM, Ts, and extruded tapioca starch (E-Ts) (Fig. 2c).

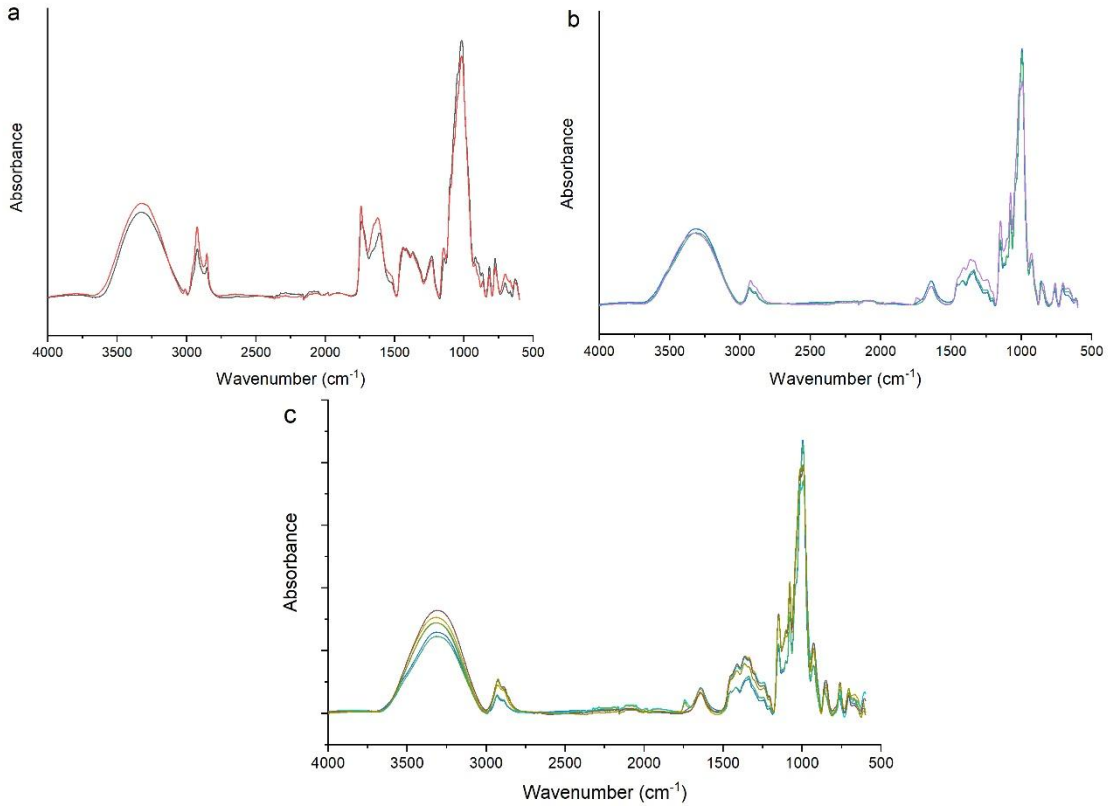

Fig 2. Normalized FTIR spectra of non-extruded and extruded matrices, comparing: (a) – Strawberry by-products (SBP) and – Extruded strawberry by-products (E-SBP); (b) – Native tapioca starch (Ts), – non-extruded mixture (NEM), and – extruded snack (E-SBPTs); (c) – Ts, – NEM and composite matrices containing Ts and cell wall (– E-CWTs) or phenolic compounds extract (– E-PPTs) or both (– E-PPCWTs)

In the comparison between SBP and E-SBP (Fig. 2a), several differences were observed at different wavenumbers within the range of 600–3030 cm^{-1}, indicating matrix structural modifications as reflected by changes in the intensity of various bands. A decrease was observed in the 770 cm^{-1} band, associated with phenyl groups (Canteri et al., 2019), which could be related to the reduction in phenolic compounds detected by HPLC-DAD. Similarly, a reduction in vibrational bands associated with C-H out-of-plane bonds in the p-hydroxyphenyl unit of lignocellulosic polymers (817 cm^{-1}) was noted (D. Chen et al., 2022), which may be linked to lignin content.

A decrease was also observed in the bands corresponding to glycosidic bonds in cellulose and hemicelluloses (860–940 cm^{-1}) (D. Chen et al., 2022; Szymanska-Chargot & Zdunek, 2013) as well as the stretching of C-O and C-C bonds in pectins (Szymanska-Chargot & Zdunek, 2013; Ying et al., 2017). Additionally, reductions in the 1144 and 1233 cm^{-1} bands were noted, associated with O-C-O bond stretching in xyloglucans, cellulose, and pectins, as well as C-O-C glycosidic linkages between

uronic acids (D. Chen et al., 2022; Szymanska-Chargot & Zdunek, 2013; Ying et al., 2017). These could suggest structural modifications in cell wall-associated polysaccharides, particularly pectins.

An increase was detected in the 1600–1625 cm^{-1} region, associated with the stretching vibration of pectins carboxyl groups (Canteri et al., 2019; Szymanska-Chargot & Zdunek, 2013), which could be correlated with their degree of methylation (DM) (Canteri et al., 2019). Additionally, some signals (1660 cm^{-1}) have been linked to the presence of lignin-like complexes in the cell wall (D. Chen et al., 2022). At 1740 cm^{-1}, an increase was observed, corresponding to the C=O stretching vibration of alkyl esters, which could be associated with pectin modifications, as reported by other authors (Canteri et al., 2019; Szymanska-Chargot & Zdunek, 2013; Z. Wang et al., 2024; Ying et al., 2017). Moreover, an increase in the 2857 and 2925 cm^{-1} bands was observed, associated with C-H bond vibrations in aliphatic chains, which may be related to changes in cellulose crystallinity (Kubovský et al., 2020), lipid compounds (Cremer & Kaletunç, 2003), or phenolic compounds (Espina et al., 2022). The increase in the 3662–3030 cm^{-1} region, associated with O-H bond stretching vibrations, may be related to changes in the exposure of these groups in phenolic compounds (Samyor et al., 2018; Z. Wang et al., 2024), as well as in polysaccharides such as cellulose, lignin, and hemicelluloses, which could arise due to modification reactions (Kubovský et al., 2020).

Fig. 2b presents the FTIR spectra of NEM, E-SBPTs, and Ts, highlighting structural modifications in starch after extrusion. Characteristic starch signals were predominant in the non-extruded mixture, while changes were observed in 1045, 1015, and 995 cm^{-1} in E-SBPTs. These peaks have been associated with C–OH bending and C–H_2-related modes and are known to be sensitive to molecular changes in starch structure (Ek et al., 2021). The 1045 cm^{-1} peak is linked to the crystalline form of starch, decreasing during gelatinization and increasing during retrogradation (Sevenou et al., 2002). More precisely, this band consists of two overlapping signals at 1040 and 1053 cm^{-1}. During retrogradation, 1040 cm^{-1} intensifies within hours due to the formation of double helices, while 1053 cm^{-1} increases over longer periods as starch molecules aggregate and crystallize (Capron et al., 2007). Given that no changes were observed at 1053 cm^{-1}, the slight increase in 1040 cm^{-1} suggested partial starch retrogradation in E-SBPTs. The 1015 cm^{-1} peak is associated with amorphous starch, increasing when the ordered structure is lost (from native to gelatinized starch) and decreasing during reordering (from gelatinized to retrograded starch). In non-extruded samples (NEM and Ts), this signal was not clearly visible; however, in E-SBPTs, it appeared alongside the 995 cm^{-1} peak. The 995 cm^{-1} peak is related to the crystalline structure of native starch and is sensitive to water content, as it is associated with hydrogen bonding in double helices (Ek et al., 2021).

In E-SBPTs, the intensity of this signal decreased compared to NEM and Ts, along with changes observed at 1015 cm^{-1} suggesting structural modifications in the ordered starch arrangement. However, in the presence of other raw materials, this behavior does not necessarily reflect starch crystallinity as it would in pure starch matrices, as reported for starch-cellulose extrudates (Ek et al., 2021). E-SBPTs also showed an increase in the 1500-1180 cm^{-1} region, suggesting modifications and interactions between cell wall polysaccharides, starch, and phenolic compounds (D. Chen et al., 2022; Ying et al., 2017). Additionally, an increase in the 1740 cm^{-1} band was observed compared to NEM and Ts, indicating a greater presence of carbonyl groups, which may be related to pectin demethylation, as seen in E-SBP, as well as phenolic compound modifications (Sivam et al., 2013). The ~3300 cm^{-1} band was slightly more intense in NEM, which was consistent with its higher moisture content, an effect also reflected in a slightly higher absorption at ~1645 cm^{-1}. Similar to what was observed in E-SBP, the 2925 cm^{-1} band increased after extrusion, which could be associated with changes in cellulose, starch, or phenolic compounds, a trend previously reported in extruded powder of lotus root nodes (H. Chen et al., 2018).

For the composite matrices (E-PPTs, E-CWTs, and E-PPCWTs), the main changes were observed at ~3300, ~2990-2800, 1740, 1500-1180, 1015, and 995 cm^{-1} (Fig. 2c). At 3300 cm^{-1}, associated with -OH stretching vibrations, all extruded samples showed increased absorption. E-PPTs exhibited the highest signal, followed by E-Ts, while E-PPCWTs and E-CWTs had similar intensities. This suggested that while extrusion itself induced conformational changes, the lower intensity in E-PPCWTs and E-CWTs compared to E-Ts may indicate fiber-starch interactions, as previously reported (Lazzari, 2011; Struck, 2018). Conversely, the increase in E-PPTs compared to E-Ts suggested an effect attributable to the presence of phenolic compounds.

In the ~2990-2800 cm^{-1} region, corresponding to C-H chain vibrations, extruded samples exhibited increased absorption, with a more pronounced effect in composite matrices suggesting structural modifications induced by both the extrusion process and the presence of fiber and phenolic compounds. The 1740 cm^{-1} band observed in E-CWTs and E-PPCWTs, also present in E-SBPTs, may be associated with cell wall polysaccharides, particularly pectins as this signal was absent in E-PPTs. In the 1500-1180 cm^{-1} region, E-PPCWTs showed lower intensity than the other extruded matrices, suggesting that fiber may influence the type and extent of phenolic compound interactions within the matrix.

Finally, signals at 1015 and 995 cm^{-1} may reflect starch structural modifications after extrusion, particularly in the reorganization of glycosidic linkages. E-PPTs and E-PPCWTs exhibited higher intensity than E-Ts and E-CWTs, suggesting that phenolic compounds may have influenced starch chain interactions, possibly limiting recrystallization or promoting the formation of less ordered structures. Conversely, the similarity between E-Ts and E-CWTs suggested that, in the absence of phenolic compound extracts, structural changes in starch were less pronounced, indicating that phenolic compounds may play a key role in the molecular reorganization of starch.

3.3 Quantification of phenolic compounds

Proanthocyanidins (PACs) were the predominant phenolic compounds in strawberry by-products, followed by anthocyanins, flavonols, phenolic acids, and monomeric flavan-3-ols (Table 2). Although PACs were the most abundant, in agreement with previous findings (Kosmala et al., 2014; Sójka et al., 2013), the relative proportions of the other phenolic groups differed from those reported in the literature (Jaroslawska, Juskiewicz, Wroblewska, Jurgonski, Boguslaw, et al., 2011; Kosmala et al., 2014; Pukalskienė et al., 2021; Tumbas Šaponjac et al., 2015). These discrepancies may be attributed to differences in strawberry cultivars, extraction methods, and industrial processing conditions (Kosmala et al., 2014; Rincon et al., 2025; Sójka et al., 2013).

The total content of flavan-3-ols (proanthocyanidins and monomers) was approximately 9801 µg/g dry matter, with PACs accounting for 97.5% of the total. The mean degree of polymerization (mDP) of PACs was 6.2. Terminal units were primarily composed of (+)-catechin (15.5%), while extension units included mainly (−)-epicatechin (57.7%), (+)-catechin (22.9%), and, to a lesser extent, epiafzelechin (3.3%) (Table 3). These results could suggest a specificity of (+)-catechin as unit of initiation (Rincon et al., 2025). Sójka et al. (2013) report comparable values for strawberry press cake, with mDP ranging from 5.8 to 7.1, although they indicate higher PACs contents, between 10,890 and 21,403 µg/g d.m.

Among anthocyanins, pelargonidin-3-*O*-glucoside was the most abundant compound in native strawberry by-products (SBP), constituing 85% of the total. It was followed by cyanidin-3-*O*-glucoside, pelargonidin-3-*O*-rutinoside, pelargonidin-3-*O*-acetylglucoside, cyanidin-3-*O*-(6"-malonylglucoside), pelargonidin-3-*O*-succinylarabinose, and pelargonidin-3-*O*-(6"-malonylglucoside). Pukalskienė et al. (2021) report a comparable pelargonidin-3-*O*-glucoside content

(789 µg/g d.m.) in strawberry pomace, while lower values are described by Hotchkiss et al. (2024) and Tumbas Šaponjac et al. (2015), with 562 µg/g d.m. and 290 µg/g d.m., respectively. Other studies show both higher (Jaroslawska, Juskiewicz, Wroblewska, Jurgonski, Krol, et al., 2011; Pukalskienė et al., 2021) and lower (Hotchkiss et al., 2024) cyanidin-3-*O*-glucoside content compared to the present findings. Cyanidin-3-*O*-glucoside is recognized as one of the most abundant anthocyanins in strawberries after pelargonidin-3-*O*-glucoside (Gasperotti et al., 2015; S. Y. Wang et al., 2002). However, its content in by-products varies depending on cultivar and processing conditions. For example, Tumbas Šaponjac et al. (2015) did not detect this compound on strawberry pomace, which may be attributed to its transfer into the juice during pressing or to its degradation during processing (Sójka et al., 2013). Additionally, pelargonidin-3-*O*-rutinoside (199 µg/g d.m.) and pelargonidin-3-*O*-malonylglucoside (201 µg/g d.m.) are also reported by Pukalskienė et al. (2021) in strawberry pomace.

Within the flavonol, quercetin-3-*O*-glucuronide was the predominant compound, followed by quercetin (aglycone), kaempferol-3-*O*-glucuronide, and kaempferol-3-*O*-malonylglucoside. These compounds have also been identified in previous studies on strawberry pomace, although often at higher concentrations. For example, Kosmala et al. 2014) report 2900 µg/g d.m. of quercetin-3-*O*-glucuronide and 2000 µg/g d.m. of quercetin. Jaroslawska, Juskiewicz, Wroblewska, Jurgonski, Krol, et al. (2011) describe quercetin levels of approximately 500 µg/g d.m., while (Tumbas Šaponjac et al., 2015) observe contents ranging from 10 to 60.63 µg/g d.m., depending on the cultivar. Kaempferol in this study was only detected in glycosylated forms, whereas other authors have reported it as aglycone (Jaroslawska, Juskiewicz, Wroblewska, Jurgonski, Krol, et al., 2011; Tumbas Šaponjac et al., 2015). As for phenolic acids, *p*-coumaroylhexoside acid was identified in the present work. Structurally related compounds have been reported in the literature, including a *p*-coumaric acid derivative at 700 µg/g d.m. (Kosmala et al., 2014) and *p*-coumaric acid at approximately 140 µg/g d.m. (Jaroslawska, Juskiewicz, Wroblewska, Jurgonski, Krol, et al., 2011). (Tumbas Šaponjac et al., 2015) describe a broader concentration range (0–340 µg/g d.m.), depending on the strawberry variety and processing conditions.

In the non-extruded matrix (NEM), the relative content among phenolic classes was maintained: PACs remained the predominant group, followed by anthocyanins, flavonols, phenolic acids, and monomeric flavan-3-ols (Table 2). Compounds that were initially present in very low concentrations in SBP were not detected in NEM, which may have fallen below the detection threshold due to dilution. Additionally, some phenolic compounds exhibited lower concentrations than expected when considering the 16.4% inclusion level of SBP. The greatest discrepancy was observed for PACs, followed by anthocyanins and phenolic acids. This deviation could be attributed to the inherent heterogeneous distribution of plant-derived ingredients within the matrix, as well as the presence of starch, which may reduce the extractability of phenolic compounds even when mixed at room temperature. In fact, it has been reported that interactions between starch and phenolic compounds, particularly polymeric ones such as PACs, may occur under ambient conditions, potentially limiting their recovery (Barros et al., 2012).

3.3.1 Effect of extrusion process on phenolic compounds

The extrusion process caused a significant reduction in most individual phenolic compounds, as well as in PACs, across the evaluated matrices (Table 2). In E-SBP, the most affected phenolic compounds were anthocyanins, with reductions exceeding 90 to 100% compared to SBP. A reduction of approximately 67% in PACs content was observed, along with a decrease in mDP, indicating a reduction in the number of monomeric units constituting the PACs (Table 3). Additionally, an increase in (+)-catechin and (-)-epicatechin as terminal units, as well as (+)-catechin as an extension unit, was observed, while (-)-epicatechin as an extension unit and epiafzelechin content were reduced. Among

flavan-3-ol monomers, (+)-catechin was reduced by 66%, whereas (-)-epicatechin increased more than threefold after the extrusion process. This behavior could be attributed to PACs degradation leading to the release of (-)-epicatechin (Le Bourvellec et al., 2013), its liberation from the by-product matrix due to enhanced extractability in the softened matrix, as previously suggested (Cao et al., 2021) or to the epimerization of (+)-catechin (Ananingsih et al., 2013). Quercetin-3-*O*-glucuronide and kaempferol-3-*O*-malonylglucoside were the least affected, showing reductions of approximately 26% and 30%, respectively, while quercetin and kaempferol-3-*O*-glucuronide exhibited reductions of slightly more than 60%. Derivatives of coumaric acid (*p*-coumaroylhexoside acid) showed reductions of around 46%.

In the extruded snack (E-SBPTs), reductions exceeding 95% were observed in most monomeric phenolic compounds, except for kaempferol-3-*O*-glucuronide, whose content doubled (Table 2). Similarly, Igual et al. (2021) report an increase in total phenolic compound content after the extrusion process, as well as in specific phenolic compounds such as ferulic acid, di-caffeic acid, quercetin-glucosyl-glucosyl-rhamnoside, isorhamnetin-glucoside, and isorhamnetin-acetyl-glucoside in corn-based snacks enriched with *Rosa canina*, while compounds such as syringic acid and *p*-coumaric acid were negatively affected. Morales et al. (2015) reported an increase in total phenolic compound content and phenolic compound groups such as hydroxybenzoic and hydroxycinnamic acids in lentil flour extrudates, although flavonols and antioxidant capacity were negatively impacted. This increase in some phenolic compounds may be related to enhanced extractability due to structural modifications of the strawberry by-products. It has been reported that the extrusion process can have two main effects: degradation and/or transformation of thermolabile phenolic compounds, and disintegration of the cell wall matrix, improving extractability (Ortiz-Cruz et al., 2020; Ruiz-Armenta et al., 2019; T. Wang et al., 2014b). PACs exhibited an 83% reduction, accompanied by a substantial decrease in their mDP (from 11 to 3) (Table 3). Similar observations are reported in studies on extrudates containing grape seeds, grape pomace (Khanal, Howard, & Prior, 2009) and blueberry pomace (Khanal, Howard, Brownmiller, et al., 2009) where an increase in the content of monomers, dimers, and trimers suggests a reduction in mDP because of the extrusion process. The decrease in mDP is commonly reported as an effect of thermal treatment due to proanthocyanidins degradation. Aditionally, increases in (+)-catechin and (-)-epicatechin as terminal units, as well as epiafzelechin as an extension unit, were observed, while (-)-epicatechin as extension unit and both (+)-catechin and (-)-epicatechin monomers decreased (Table 3). Compared to E-SBP (extruded by-products without starch), the presence of starch in E-SBPTs could limit the release or recovery of (-)-epicatechin, possibly due to interactions within the starchy matrix (Barros et al., 2012). The increased proportion of epiafzelechin may be related to its lower hydroxylation degree, which could reduce its affinity for starch and enhance its stability or extractability under these conditions (Le Bourvellec & Renard, 2012; Rincon et al., 2025). This increase in individual phenolic compounds has also been reported for extruded beans, where compounds such as quercetin, kaempferol, chlorogenic acid, and p-coumaric acid showed increased levels after extrusion (Korus et al., 2007).

In the matrix containing both cell wall material and phenolic compound extract (E-PPCWTs), several monomeric phenolic compounds, including anthocyanins, phenolic acids, and flavonols, were either absent or present in lower concentrations except by kaempferol-3-*O*-glucuronide (Table 2), which, as also observed in the extruded snack (E-SBPTs), showed a twofold higher content compared to the non-extruded mixture (NEM). Among monomeric flavan-3-ols, the content of (-)-epicatechin was higher, whereas (+)-catechin remained relatively unchanged in comparison with NEM. Despite the overall lower PACs content, mDP was higher (Table 3). This was accompanied by a predominance of (-)-epicatechin as a terminal unit and epiafzelechin as an extension unit, whereas the proportion of (+)-catechin as a terminal unit was notably lower. These findings may reflect a selective retention of highly polymerized proanthocyanidins by the cell wall components, affording protection against processing-related degradation (X. Liu, Le Bourvellec, et al., 2021; Lončarić et al., 2018). In this

formulation, PACs oligomers were added in extract form, making them more available for rearrangement and potential interactions with matrix constituents such as cell wall polysaccharides and starch (D. Amoako & Awika, 2016). In contrast, in E-SBPTs, phenolic compounds are naturally embedded within the by-product matrix, which may limit their degradation during the extrusion process. Additionally, although the phenolic compounds were derived from the same strawberry by-products, the composition and arrangement in the native material and the extract were inherently different, influencing their behavior under extrusion conditions.

In the matrix containing only the phenolic compound extract (E-PPTs), none of the phenolic compounds identified in the native matrices were quantified, except for proanthocyanidins, which were still present in very low amounts (Table 2). Considering the results observed in E-PPCWTs, these findings suggest that, beyond the degradation and loss typically associated with extrusion, the process itself may have promoted additional breakdown or hindered the recovery of phenolic compounds in the absence of a polysaccharide-rich matrix. Phenolics are known to exhibit selective binding affinities with various cell wall polysaccharides (Siemińska-Kuczer et al., 2022). These interactions occur primarily through non-covalent forces such as hydrogen bonding, hydrophobic interactions, and ionic bonds (Giuberti et al., 2020; X. Liu et al., 2020), and are strongly influenced by the molecular weight and polarity of the phenolic compounds, as well as the structural characteristics of the polysaccharides (D. Amoako & Awika, 2016). In this context, in the E-PPCWTs matrix, the interaction between strawberry by-product phenolic compounds and cell wall components could also avoid the total loss of phenolic compounds observed when phenolic compound extract was used alone (E-PPTs).

3.4 Cell wall composition

Galacturonic acid was the predominant component of the strawberry cell wall (172 mg/g cell wall), followed by cellulosic glucose (125 mg/g cell wall) and lignin (92 mg/g cell wall). In addition to these, the most abundant neutral sugars were xylose (48 mg/g cell wall), galactose (31 mg/g cell wall), and arabinose (average of 28 mg/g cell wall). Mannose, rhamnose and fucose were detected in lower amounts (Table 4). Arabinose and galactose are typically found in the side chains of pectins, while xylose, along with non-cellulosic glucose and fucose, is associated with hemicelluloses. These findings are consistent with previous reports (Cybulska et al., 2022; Hotchkiss et al., 2024; Rincon et al., 2025). The degree of methylation of pectins was 47%. Values ranging from 46% to 83% have been reported for different strawberry cultivars (Cybulska et al., 2022; Rincon et al., 2025).

In NEM matrix, galacturonic acid (154 mg/g cell wall) was the main component of the cell wall, followed by lignin (189 mg/g cell wall) and cellulose (119 mg/g cell wall), in contrast to the SBP cell wall, where cellulose predominated over lignin. Although both matrices were not subjected to extrusion, a higher degree of methylation (+61%) was observed in the NEM cell wall, because of the lower galacturonic acid content, which could be related to the de-starching treatment applied to this matrix. This treatment could promote β-elimination reactions, which are favored under neutral to slightly acidic pH. As the temperature increases, the rate of β-elimination increases faster than that of demethylation, leading to depolymerization of methylated homogalacturonan chains without significant methanol release (X. Liu, Renard, et al., 2021; Voragen et al., 2009). Similarly, the higher lignin content observed in NEM could result from the relative concentration of lignin due to the removal of other more extractable components during the starch remotion process.

Table 2. Phenolic compounds composition (ug/g d. m.) of the non-extruded and extruded analyzed matrices

Matrix	PACs	p-C-HexA	Q	Q-3-GlcA	K-3-GlcA	K-3-MalGlc	C-3-Glc	C-3-(6''-MalGlc)	Pg-3-Glc	Pg-3-Rut	Pg-3-(6''-MalGlc)	Pg-3-AcGlc	Pg-3-SucAra	(+)-Cat	(-)-Epi
SBP	9554.8 ± 407.2[a]	560,9 ± 18,3[a]	63.4 ± 2.0[a]	627.8 ± 27.1[a]	3.6 ± 0.3[a]	1.8 ± 0.2[a]	73.6 ± 3.6[a]	2.6 ± 0.2[a]	752.8 ± 18.8[a]	42.7 ± 2.2[a]	0.7 ± 0.0[a]	8.1 ± 0.3[a]	2.3 ± 0.1[a]	222.0 ± 2.2[a]	24.4 ± 0.6[a]
E-SBP	3141.4 ± 50.0[b]	302,5 ± 8,6[b]	22.6 ± 1.8[b]	464.2 ± 29.5[b]	1.4 ± 0.1[b]	1.3 ± 0.1[b]	3.1 ± 0.2[b]	ND	45.3 ± 1.6[b]	3.0 ± 0.1[b]	ND	ND	ND	75.5 ± 4.4[b]	91.9 ± 3.3[b]
NEM	596.5 ± 28.0[c]	53,3 ± 2,8[c]	9.5 ± 0.5[c]	94.8 ± 6.8[c]	0.4 ± 0.0[c]	0.3 ± 0.0[c]	5.8 ± 0.5[c]	ND	55.3 ± 2.3[c]	3.0 ± 0.1[c]	ND	ND	ND	22.9 ± 1.4[c]	1.9 ± 0.1[c]
E-SBPTs	100.70 ± 8.9[d]	1.0 ± 0.1[d]	0.1 ± 0.0[d]	2.7 ± 0.2[d]	0.8 ± 0.0[d]	ND	ND	ND	0.1 ± 0.0[d]	ND	ND	ND	ND	1.2 ± 0.1[d]	1.6 ± 0.1[d]
E-PPCWTs	63.6 ± 5.6[d]	1.3 ± 0.1[d]	ND	2.2 ± 0.1[d]	0.9 ± 0.1[d]	ND	ND	ND	ND	ND	ND	ND	ND	3.8 ± 0.1[e]	5.8 ± 0.2[e]
E-PPTs	2.0 ± 0.1[e]	0.03 ± 0.00[d]	ND	0.03 ± 0.00[d]	0.04 ± 0.00[e]	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND

SBP: strawberry by-products; **E-SBP**: extruded strawberry by-products; **NEM**: non-extruded mixture of strawberry by-products and tapioca starch; **E-SBPTs**: extruded snack formulated with strawberry by-products and tapioca starch; **E-PPCWTs**: extruded composite matrix of phenolic compounds extract, cell wall material (from SBP), and tapioca starch; **E-PPTs**: extruded composite matrix of phenolic compounds extract (from SBP) and tapioca starch; **E-CWTs**: extruded composite matrix of cell wall material (from SBP) and tapioca starch; **PACs:** Proanthocyanidins; **C-HexA**: Coumaroylhexoside acid; **p-C-HexA**: *p*-coumaroylhexoside acid; **Q**: Quercetin; **Q-3-GlcA**: Quercetin-3-O-glucuronide; **K-3-GlcA**: Kaempferol-3-*O*-glucuronide; **K-3-MalGlc**: Kaempferol-3-*O*-malonylglucoside; **C-3-Glc**: Cyanidin-3-*O*-glucoside; **C-3-(6''-MalGlc)**: Cyanidin 3-*O*-(6"-malonylglucoside); **Pg-3-Glc**: Pelargonidin-3-O-glucoside; **Pg-3-Rut**: Pelargonidin-3-*O*-rutinoside; **Pg-3-(6''-MalGlc)**: Pelargonidin-3-*O*-(6"-malonylglucoside); **Pg-3-AcGlc**: Pelargonidin-3-*O*-acetylglucoside; **Pg-3-SucAra**: Pelargonidin-3-*O*-succinylarabinose; **(+)-Cat**: (+)-Catechin; **(-)-Epi**: (-)-Epitechin
ND: Not detected
Different letters in the same column indicate significant differences ($p < 0.05$)

Table 3. Mean degree of polymerization (mDP) and proportion of constitutive units (%) of proanthocyanidins after furanolysis in the non-extruded and extruded analyzed matrices

Matrix	mDP	% (+)-Cat T	% (-)-Epi T	% (+)-Cat E	% (-)-Epi E	% Epiafz E
SBP	6.2 ± 0.2[a]	15.5 ± 0.4[a]	0.6 ± 0.1[a]	22.9 ± 0.6[a]	57.7 ± 0.6[a]	3.3 ± 0.0[a]
E-SBP	3.8 ± 0.2[b]	17.1 ± 0.7[b]	9.1 ± 0.5[b]	25.8 ± 0.8[b]	45.3 ± 1.1[b]	2.7 ± 0.2[b]
NEM	11.2 ± 0.7[c]	8.6 ± 0.6[c]	0.4 ± 0.0[c]	29.0 ± 2.2[c]	58.5 ± 3.0[c]	3.5 ± 0.3[c]
E-SBPTs	3.4 ± 0.2[d]	11.0 ± 0.8[d]	18.3 ± 0.9[d]	28.5 ± 0.5[c]	34.7 ± 1.2[d]	7.5 ± 0.5[d]
E-PPCWTs	15.1 ± 0.5[e]	1.1 ± 0.1[e]	5.5 ± 0.2[f]	30.8 ± 0.7[c]	58.3 ± 0.6[c]	4.3 ± 0.2[c]
E-PPTs	3.5 ± 0.3[d]	0	28.4 ± 2.3[e]	51.4 ± 1.6[d]	20.3 ± 1.3[e]	0

SBP: strawberry by-products; **E-SBP**: extruded strawberry by-products; **NEM**: non-extruded mixture of strawberry by-products and tapioca starch; **E-SBPTs**: extruded snack formulated with strawberry by-products and tapioca starch; **E-PPCWTs**: extruded composite matrix of phenolic compounds extract, cell wall material (from SBP), and tapioca starch; **E-PPTs**: extruded composite matrix of phenolic compounds extract (from SBP) and tapioca starch; **E-CWTs**: extruded composite matrix of cell wall material (from SBP) and tapioca starch; **mDP:** Mean Degree of Polymerization; **% (+)-Cat T:** Percentage of (+)-catechin as terminal unit; **% (-)-Epi T:** Percentage of (-)-epicatechin as terminal unit; **% (+)-Cat E:** Percentage of catechin as extension unit; **% (-)-Epi E:** Percentage of (-)-epicatechin as extension unit; **% Epiafz E:** Percentage of epiafzelechin as extension unit
Different letters in the same column indicate significant differences ($p < 0.05$)

Table 4. Neutral sugars, galacturonic acid, lignin and methanol contents (mg/g cell wall) of the different strawberry cell walls depending on the matrix structure

Matrix	Rha	Fuc	Ara	Xyl	Man	Gal	NCGlc	CGlc	Gal A	MeOH	DM	Lig
SBP	5.7 ± 0.5^{a}	2.9 ± 0.2^{a}	28.2 ± 2.9^{a}	48.9 ± 5.3^{ab}	9.9 ± 1.1^{a}	31.5 ± 3.4^{a}	11.6 ± 1.1^{a}	124.8 ± 6.8^{abc}	172.2 ± 10.6^{a}	14.7 ± 1.1^{ac}	47.1 ± 2.6^{a}	91.9 ± 5.2^{a}
E-SBP	4.6 ± 0.4^{bd}	1.6 ± 0.1^{b}	21.7 ± 1.0^{b}	51.2 ± 4.7^{ab}	8.2 ± 0.9^{a}	26.9 ± 1.6^{ab}	71.1 ± 2.2^{b}	115.2 ± 12.5^{a}	158.0 ± 9.7^{abc}	17.4 ± 0.8^{ab}	60.5 ± 3.4^{b}	144.9 ± 7.8^{b}
NEM	3.6 ± 0.3^{c}	1.1 ± 0.1^{c}	17.4 ± 1.2^{cd}	54.7 ± 5.8^{ab}	10.6 ± 0.7^{a}	21.0 ± 2.3^{b}	9.4 ± 0.6^{a}	119.4 ± 6.8^{ab}	154.4 ± 7.3^{bc}	13.8 ± 0.8^{c}	61.2 ± 0.8^{b}	188.6 ± 7.8^{c}
E-SBPTs	3.8 ± 0.2^{cb}	1.4 ± 0.1^{bc}	15.4 ± 1.4^{e}	42.5 ± 3.7^{a}	17.2 ± 1.0^{b}	27.5 ± 2.1^{a}	12.3 ± 0.1^{a}	125.2 ± 8.5^{abc}	147.2 ± 7.4^{bc}	19.7 ± 1.7^{b}	73.4 ± 5.1^{c}	78.9 ± 1.1^{a}
E-CWTs	4.8 ± 0.3^{ad}	1.5 ± 0.1^{b}	18.6 ± 1.1^{bd}	50.2 ± 4.6^{ab}	15.3 ± 1.2^{b}	30.0 ± 1.1^{a}	11.1 ± 1.0^{a}	140.1 ± 7.9^{bc}	165.2 ± 7.2^{ab}	18.0 ± 0.3^{b}	62.4 ± 5.5^{b}	205.9 ± 5.6^{c}
E-PPCWTs	4.7 ± 0.1^{bd}	1.4 ± 0.1^{bc}	16.3 ± 0.6^{cd}	57.6 ± 4.9^{b}	15.0 ± 0.6^{b}	25.7 ± 1.5^{ab}	10.9 ± 0.8^{a}	143.8 ± 8.1^{c}	136.6 ± 10.0^{c}	13.0 ± 0.7^{c}	59.4 ± 3.2^{b}	131.1 ± 12.6^{b}

SBP: strawberry by-products; **E-SBP**: extruded strawberry by-products; **NEM**: non-extruded mixture of strawberry by-products and tapioca starch; **E-SBPTs**: extruded snack formulated with strawberry by-products and tapioca starch; **E-PPCWTs**: extruded composite matrix of phenolic compounds extract, cell wall material (from SBP), and tapioca starch; **E-PPTs**: extruded composite matrix of phenolic compounds extract (from SBP) and tapioca starch; **E-CWTs**: extruded composite matrix of cell wall material (from SBP) and tapioca starch; **Rha**: rhamnose; **Fuc**: fucose; **Ara**: arabinose; **Xyl**: xylose; **Man**: mannose; **Gal**: galactose; **Glc**: glucose; **Gal A**: galacturonic acid; **CGlc**: glucose from cellulose; **NCGlc**: glucose from non-cellulose; **MeOH**: methanol; **DM**: degree of methylation; **Lig**: lignin.
Different letters in the same column indicate significant differences ($p < 0,05$) by Tukey test.

3.4.1 Effect of extrusion process on cell wall composition

A significant decrease in rhamnose (-19%), fucose (-43%), and arabinose (-23%) was observed in extruded strawberry by-products (E-SBP) compared to SBP, while non-cellulosic glucose (NCGlc) and lignin contents, and degree of methylation increased by 6-, 1.6- and 1.3-fold, respectively (Table 4). These results suggest partial degradation of pectic rhamnogalacturonan I regions (Jiang et al., 2021; Waldron & Faulds, 2007). In contrast, the levels of xylose, mannose, galactose, and cellulosic glucose remained stable, indicating that hemicelluloses and cellulose were less affected by the extrusion process. Additionally, the increase of degree of methylation may reflect a preferential retention of highly methylated pectins during extrusion, as less methylated pectins are more susceptible depolymerization and solubilization (X. Liu, Renard, et al., 2021). The higher lignin content may be related to its resistance to thermal degradation and its structural role in the cell wall matrix (Bindon et al., 2010; Dridi & Bordenave, 2021; C. M. G. C. Renard et al., 2017; H. Zhang et al., 2014)

In E-SBPTs, only arabinose decreased (-11%) compared to the non-extruded mixture (NEM) (Table 4), suggesting degradation or solubilization of arabinan side chains (Schmid, Trabert, et al., 2020; Waldron & Faulds, 2007). In contrast, rhamnose, fucose, xylose, and cellulosic glucose levels remained stable, indicating that pectins and hemicelluloses were less affected by extrusion. Meanwhile, mannose, galactose, and non-cellulosic glucose increased (1.3- to 1.6-fold), suggesting greater resistance of these components to thermo-mechanical stress. Lignin content decreased after extrusion, in contrast to what was observed in E-SBP (extruded strawberry by-products without starch). A concurrent increase in the degree of methylation was also observed, which may reflect a selective loss of less methylated pectin domains, while more methylated regions were preferentially retained, as previously discussed. Fiber modifications induced by the thermomechanical process of extrusion, such as solubilization and/or targeted degradation of specific polysaccharide components, have been reported in extruded plant-derived materials such as apple, chokeberry, and citrus pomace (Redgwell et al., 2011; Schmid et al., 2021; Schmid, Steck, et al., 2020; Schmid, Trabert, et al., 2020; Sobota et al., 2010). In addition, hydrothermal conditions during extrusion may promote interactions between starch and pectic polysaccharides (Zhai et al., 2021; Y. Zhang et al., 2021), thereby influencing the composition of the recovered cell wall. Starch gelatinization and associated molecular rearrangements may alter the distribution of pectins in the insoluble fraction and/or enhance associations with hemicelluloses, potentially modifying their susceptibility to degradation (Robin et al., 2012; Wolf, 2010).

The E-PPCWTs composite matrix exhibited a composition similar to the non-extruded mixture (NEM) in most components, except for lower lignin content (-31%) and higher levels of rhamnose (+32%), mannose (+25%), and cellulosic glucose (+16%) (Table 4). The E-CWTs composite matrix displayed a broader compositional profile, with higher contents of rhamnose (+36%), fucose (+33%), mannose (+45%), galactose (+43%), cellulosic glucose (+17%), galacturonic acid (+7%), and methanol (+30%). In both matrices, arabinose, xylose, non-cellulosic glucose, and the degree of methylation remained relatively stable, suggesting that certain polysaccharide domains, such as arabinan, xyloglucan, and highly methylated pectins, were less affected by the extrusion process.

The differences between these matrices could be linked to the combined presence of starch and phenolic compounds in E-PPCWTs, which may have influenced the organization of cell wall components during extrusion. Overall, the composition of this matrix presents a profile where multiple modifications could occur simultaneously, including solubilization and/or degradation of cell wall components (Schmid et al., 2022b; Schmid, Trabert, et al., 2020), influenced by various interactions such as phenolic compounds (including lignin)–polysaccharides (Kang et al., 2019; C. M. G. C. Renard et al., 2017), phenolic compounds–starch (D. Amoako & Awika, 2016; F. Zhu, 2015),

and starch–polysaccharides (S. Li et al., 2023; Zhai et al., 2021), These interactions may affect the structural dynamics of the matrix and influence the distribution and association of cell wall components. Moreover, starch gelatinization might have altered the accessibility or stabilization of specific polysaccharides (S. Li et al., 2023; Qiu et al., 2024) or these polysaccharides could interfere with starch reassociation during cooling (Ek et al., 2021). Therefore, it is probable that multiple concurrent factors, including matrix composition, process dynamics, and interactions among various components, collectively influence the recovery and composition of the cell wall fraction after extrusion.

Conclusions

FTIR analysis showed structural changes in fiber, starch, and phenolic compounds after extrusion while SEM revealed that the presence of fiber and phenolic compounds influenced the cohesion and porosity of the extrudates. Extrusion process caused a significant reduction in the content of most individual phenolic compounds, particularly anthocyanins and flavonoids, with losses exceeding 95% in some cases. However, compounds, such as kaempferol-3-*O*-glucuronide and (-)-epicatechin, increased after extrusion, which may reflect enhanced extractability due to matrix modification, or in the case of (-)-epicatechin, partial release from proanthocyanidins. Differences in phenolic compounds stability were identified depending on the composition of the extruded matrix. Specifically, the formulation containing both cell wall material and phenolic compound extract (E-PPCWTs) showed greater retention of phenolic compounds compared to the formulation containing only phenolic compound extract (E-PPTs), suggesting that the presence of cell wall components may have contributed to their stability during extrusion, potentially through their interactions.

The analysis of proanthocyanidins (PACs), their subunit composition, and mean degree of polymerization (mDP) indicated that extrusion promoted the fragmentation of phenolic polymers in most treatments, with PACs reductions of up to 83% in some matrices. However, in E-PPCWTs, an increase in mDP was observed, suggesting that under specific conditions, the process may have promoted the degradation of low molecular weight proanthocyanidins. Extrusion selectively modified the cell wall composition of strawberry by-products and composite matrices. Pectic rhamnogalacturonans were the most affected, while hemicelluloses and cellulose remained stable. Matrix composition influenced lignin behavior and the recovery of neutral sugars. Starch and phenolic compounds influenced polysaccharide reorganization, highlighting the complex interplay of components during extrusion.

The extrusion of matrices formulated with strawberry by-products and its fractions not only modified the composition and structure of cell wall and phenolic compounds but also promoted distinct interactions between these components depending on the matrix used. The results suggested that fiber may modulate phenolic compounds stability during extrusion, while phenolic compounds could influence polysaccharide reorganization and the structural properties of the extruded matrix. These findings provide valuable insights for future research on the stability and functionality of fiber- and phenolic compound-rich ingredients in extruded products, contributing to the development of formulations with improved structural stability and enhanced both functional and nutritional properties. Moreover, the valorization of strawberry by-products through extrusion may support the creation of innovative functional snacks with potential health-promoting attributes and added market value, in line with current trends toward sustainable and health-oriented food products.

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Chapter 5. Extrusion of model systems with tapioca starch and strawberry by-products: starch structural changes, *in vitro* digestibility, glycemic index, and phenolic compound bioaccessibility

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Abstract

The valorization of strawberry by-products as functional ingredients in thermally processed foods has gained increasing attention due to their high dietary fiber and phenolic compound contents. Extrusion cooking is a versatile technique widely used in the development of expanded products, but its effects on starch structure, digestibility, and the stability and release of phenolic compounds remain insufficiently understood, particularly in complex food matrices. This study evaluated the impact of extrusion on starch structural changes, *in vitro* digestibility, glycemic index, and phenolic compound bioaccessibility in model systems formulated with tapioca starch and strawberry by-products. Native and extruded matrices were analyzed, including combinations with isolated cell wall material and phenolic compound extract. X-ray diffraction and HPSEC-MALLS analyses revealed a marked starch crystallinity and molecular weight reduction following extrusion, especially in phenolic compound-enriched matrices. Starch digestibility increased across all extruded samples, with higher rapidly digestible starch (RDS) and glycemic index values. However, matrices containing cell wall and/or phenolic compounds exhibited an increase in slowly digestible starch (SDS) and showed a reduced glycemic index, highlighting the role of matrix composition. In terms of phenolic compound bioaccessibility, extrusion enhanced phenolic released in the extruded snack but bioaccessibility was reduced in matrices combining cell wall and phenolic compounds, where most compounds remained in the indigestible fraction. These findings emphasize the effect of both formulation and processing conditions in modulating the nutritional and functional behavior of extruded products enriched with fruit by-products.

Keywords: heat treatment, nutritional functionality, digestible starch fractions, molar mass distribution, interaction

1 Introduction

Extrusion cooking is a versatile technology widely employed in the development of ready-to-eat foods such as breakfast cereals and snacks. This process combines high temperature, mechanical shear, and short residence time, involving structural, physicochemical, and nutritional modifications of food ingredients (Alam et al., 2016; Medina-Rendon et al., 2023). Due to its versatility, extrusion has become a strategic method for incorporating underutilized plant-based materials into mainstream food products, contributing not only to product innovation but also to waste valorization and functional food development.

One of the major applications of extrusion in functional food design is the incorporation of fruit and vegetable by-products. These materials are rich in dietary fiber, phenolic compounds, and other bioactive compounds, making them attractive ingredients for nutritional improvement and health claim development (Grasso, 2020; Offiah et al., 2019; Patil & Kaur, 2018). Strawberry by-products (SBP), derived from juice processing, are especially promising due to their high content of proanthocyanidins, anthocyanins, and phenolic acids (Pukalskienė et al., 2021). However, the harsh conditions of extrusion, particularly high shear and thermal input, can lead to the degradation, transformation, or release of these compounds (Hirth et al., 2015; Neder-Suárez et al., 2021).

The digestibility of starch and its glycemic response are also notably affected by extrusion. Depending on processing conditions and ingredient composition, extrusion can increase rapidly digestible starch (RDS) and reduce resistant starch (RS), thereby altering the glycemic index (GI) of the final product (Altan et al., 2009; Lv et al., 2022; Oladiran & Emmambux, 2018). Moreover, dietary fiber and phenolic compound, especially when they are together, may interact with starch, modifying its gelatinization behavior, molecular degradation, and enzyme accessibility (D. Amoako & Awika,

2016; Mohamed, 2023). phenolic compounds may also bind to dietary fiber, altering their bioaccessibility in the gastrointestinal tract (Jakobek, 2015; Saura-Calixto et al., 2007), and can exert prebiotic effects through their microbial metabolization in the colon (Núñez-Gómez et al., 2023).

Although previous studies have assessed changes in phenolic compounds and starch digestibility during extrusion (Altan et al., 2009; Hirth et al., 2015; Oladiran & Emmambux, 2018; Schmid et al., 2020), few have explored how different structural fractions from a single plant material, such as the cell wall and phenolic compounds of strawberry by-products (SBP), modulate the physicochemical and nutritional responses of extruded matrices. Additionally, the interplay between extrusion conditions, ingredient interactions, and compound stability remains poorly understood.

In this context, the present study aimed to investigate the impact of extrusion process on the macromolecular properties of starch, its *in vitro* digestibility, glycemic index, and phenolic compound bioaccessibility in tapioca starch-based matrices enriched with SBP and their isolated fractions. By deconstructing SBP into its structural components, cell wall material and phenolic extract, and reintegrating them into controlled formulations, this research provides new insights into the complex modification that occur during extrusion and how they influence the nutritional functionality of the final product.

2 Materials and methods

2.1 Raw materials

Strawberry (*Fragaria × ananassa*) by-products (SBP) used in this research were obtained from pulp extraction process and provided by Monkeyfruit S.A. (Popayán, Colombia). The processed fruits of the Sabrina variety. were sourced from local cultivars in the municipality of Sotará, Cauca, The SBP were frozen at −80 °C for 12 h, then dried in a vacuum freeze dryer (YR05188, Kalstein, Paris, France) for 72 h. The dried SBP were grinded (MF 10 basic Molino IKA, Germany) rapidly and the powder was vacuum sealed and stored at −20 °C until use. Tapioca (*Manihot esculenta*) starch was supplied by Ingredion Colombia S.A. (Cali, Colombia).

2.2 Standards and chemicals

All reagents were of analytical grade, HPLC solvents were of chromatographic purity, and water was purified through deionization using a Milli-Q system (Millipore, Bedford, MA, USA). Folin-Ciocalteu reagent and sodium carbonate were obtained from Panreac (Barcelona, Spain). Methanol, acetone, ethanol, hydrochloric acid, sulfuric acid, sodium hydroxide, formic acid, and acetic acid were purchased from Merck (Darmstadt, Germany). Dimethyl sulfoxide (DMSO) was supplied by Sigma-Aldrich (St. Louis, MO, USA). Pepsin (from porcine gastric mucosa), pancreatin (from porcine pancreas), and bile salts were obtained from Sigma-Aldrich (St. Louis, MO, USA) and used for *in vitro* digestion. Enzymes for starch digestibility assays, including pancreatic α-amylase (PAA), amyloglucosidase (AMG), and glucose oxidase/peroxidase (GOPOD) reagent, were provided in the Digestible and Resistant Starch Assay Kit (K-DSTRS) from Megazyme International (Bray, Ireland).

2.3 Preparation of cell wall material and phenolic compound extract

Cell wall (CW) was prepared from strawberry by-products following the method described by Le Bourvellec et al. (2011). Briefly, 100 g of strawberry by-product powder was washed with 70% ethanol and extracted at room temperature until the absence of sugars was confirmed by a negative reaction in the phenol-sulfuric acid test. The samples were then sequentially washed twice with a 60% acetone-water solution, once with an 80% acetone-water solution, and finally with pure acetone until

the supernatant became colorless. The residue was dried at 40 °C for 24 h and subsequently weighed, yielding 54.7% on a dry basis. This yield was later taken into account for the formulation of the matrices containing cell wall material.

The washing liquid was recovered, concentrated using a rotary evaporator, and subsequently purified. For purification, the extracts were dissolved in water acidified with 0.01 M HCl at a 1:10 ratio (Hejniak et al., 2019) and passed through a Sep-Pak C18 cartridge. The cartridge was pre-conditioned with 8 mL of methanol, followed by 4 mL of pH 7.0 water and 5 mL of 0.01 M HCl. The sample was then washed with 10 mL of pH 7.0 water and eluted with 12 mL of methanol to obtain the phenolic compound extract (Sarria Villa et al., 2021). The resulting methanolic extract was freeze-dried and subsequently reconstituted in water for use in the extrusion formulations. The concentration of total phenolic compounds in the reconstituted extract was determined using the Folin–Ciocalteu method, yielding 2.64 mg GA/mL. This concentration was later used to determine the volume of extract to be incorporated into the phenolic-enriched matrices.

2.4 Preparation of matrices for extrusion

Different matrices were prepared and used to evaluate the impact of the extrusion process, an extruded snack formulation on both starch, fiber and phenolic compounds (Table 1). Composite matrices consisting of various combinations of tapioca starch, cell wall material, and phenolic compound extract were prerared. The extruded snack (E-SBPTs) was formulated by mixing tapioca starch with 16.5% (d.m.) matter) strawberry by-products and processed at a moisture content of 18.6%, a condition previously identified through optimization studies to improve both processing performance and product quality. In addition, a non-extruded matrix with the same formulation (NEM) was included as a control of the extrusion process.

Table 1. Description of the samples analyzed in the study

Sample Code	Sample description
Ts	Native tapioca starch (control)
SBP	Strawberry by-products (pomace), non-extruded
NEM	Non-extruded mixture of strawberry by-products and tapioca starch
E-Ts	Extruded tapioca starch (control)
E-SBPTs	Extruded snack, composite of strawberry by-products and tapioca starch
E-PPCWTs	Extruded composite matrix: phenolic extract + cell wall (from SBP) + tapioca starch
E-CWTs	Extruded composite matrix: cell wall (from SBP) + tapioca starch
E-PPTs	Extruded composite matrix: phenolic extract (from SBP) + tapioca starch

Composite matrices were formulated using tapioca starch. One matrix consisted of tapioca starch with cell wall material extracted from strawberry by-products (E-CWTs), another combined tapioca starch with a purified extract of phenolic compounds obtained from strawberry by-products (E-PPTs), and a third included both cell wall material and phenolic extract along with tapioca starch (E-PPCWTs). The amount of each component was calculated to replicate the contribution of cell wall and phenolic compounds present in a formulation containing 16.5% (d.m.) strawberry by-products, as used in the E-SBPTs snack. Specifically, the amount of cell wall material was adjusted based on its extraction yield (54.7%), while the volume of phenolic extract was determined according to its total phenolic concentration (2.6 mg GA/mL), ensuring equivalent polyphenol input across formulations.

Furthermore, strawberry by-products were extruded alone at 18.6% moisture content (E-SBP), enabling the evaluation of structural and chemical changes induced by processing. In parallel a matrix containing only extruded tapioca starch (E-Ts) was prepared as a control to determine the influence of the other components on the evolution of starch during the extrusion process. Due to the fine texture of these materials, the composite matrices were processed at 30% moisture content, as lower moisture

levels hindered their proper passage through the extruder. Each formulation was thoroughly mixed and adjusted to the target moisture content, followed by blending for ten minutes at medium speed using a KP26M1XER commercial mixer (KitchenAid, MI, USA). The prepared mixtures were then packed in polyethylene bags and stored for 12–14 hours to ensure moisture equilibration and homogeneity before extrusion.

2.5 Extrusion process

Extrusion cooking was carried out using a laboratory-scale co-rotating twin-screw extruder (HAAKE™ Rheomex OS, ThermoFisher Scientific, Germany) with an 11 mm screw diameter, a length-to-diameter ratio of 40:1, and a 3 mm die opening. The final barrel zones and the die were set at temperatures of 140/160/180/192.02 °C. Screw speed and feed rate were set at 140 rpm and 20 g/min, respectively. The extrudates were dried at 60 °C for 30 minutes, packed in polyethylene bags, and stored in airtight containers until further physical analysis. For chemical analysis, the extruded samples were ground using a laboratory grinder (A 11 basic IKA mill, Germany) and passed through a mesh-60 sieve.

2.6 X-ray diffraction (XRD)

X-ray diffraction (XRD) analysis was performed using a Malvern-PANalytical Empyrean 2012 diffractometer (Malvern Panalytical Ltd., Malvern, UK), equipped with a Pixel 3D detector and a Cu Kα radiation source (λ = 1.541874 Å) operating at 45 kV and 40 mA. The goniometer was set to an Omega/2θ configuration, scanning from 2° to 40° (2θ). The sample stage was configured in Reflection-Transmission Spinner mode with 4-second rotations. The scan was conducted with a step size of 0.02° and a time per step of 52 seconds. Spectral data were processed using Origin software (version 2018, OriginLab Corporation, Northampton, MA, USA).

The relative crystallinity of the samples was calculated according to the method described by (Ek et al. (2021), using the Equation (1).

$$Crystallinity(\%) = \frac{Crystalline\ area}{Crystalline\ area + amorphous\ area}\ x\ 100 \quad (1)$$

2.7 Molar mass analysis of starch

Starch was pretreated as described by Rolland-Sabaté et al. (2011), using dimethyl sulfoxide (DMSO) to aid in solubilization. Samples were solubilized at a concentration of 0.5 g/L using microwave-assisted heating under pressure and then diluted to 0.25 g/L before analysis. Prior to injection, solutions were filtered through a 5 µm membrane. High-performance size exclusion chromatography (HPSEC) coupled with multi-angle laser light scattering (MALLS), quasi-elastic light scattering (QELS), viscometry, diode array detection (DAD), and fluorimetry (HPSEC-MALLS-QELS-VS-DAD-Fluo) was conducted. A 50 µL aliquot of each prepared starch solution was injected into the system. The HPSEC-MALLS setup consisted of a Prominence Ultra-Fast Liquid Chromatography system (Shimadzu, Kyoto, Japan), equipped with an LC-20AD pump, DGU-20A5 degasser, SIL-20ACHT autosampler, CTO-20AC column oven, SPD-M20A diode array detector (DAD), and RID-10A refractive index detector (RID), all from Shimadzu (Tokyo, Japan). The system was further equipped with a DAWN HELEOS 8+ multi-angle laser light scattering detector (Wyatt Technology Corp., Santa Barbara, CA, USA), featuring a K5 flow cell and a GaAs laser (λ = 660 nm), a WyattQELS® module positioned at 110°, and a ViscostarIII viscometer. Chromatographic separation was carried out at 30 °C using a Shodex KW 802.5 column (300 × 7.8 mm) with a corresponding guard column (Shodex, Tokyo, Japan). The mobile phase was Millipore water containing 0.2 g/L

sodium azide, filtered through a 0.1 µm Omnipore™ membrane (Millipore, Milford, MA, USA) and degassed prior to use. The flow rate was maintained at 0.6 mL/min.

Weight-average (Mw), number-average (Mn), and peak (Mp) molar mass were calculated using ASTRA® software (version 7.1.4, Wyatt Technology Corp.). Chromatograms and molar mass distribution plots were generated using Microsoft Excel.

2.8 In vitro starch digestibility and estimated glycemic index (GI)

In vitro starch digestibility was determined using the *Digestible and Resistant Starch Assay Kit* following the manufacturer's instructions based on AOAC Method 2017.16/2022.01. Approximately 50 mg of sample (dry basis) was incubated at 37 °C in a shaking water bath with a mixture of pancreatic α-amylase and amyloglucosidase (PAA/AMG) in maleate buffer (pH 6.0) under constant stirring for 4 hours. Aliquots of 1.0 mL were taken at 20, 120, and 240 min to determine rapidly digestible starch (RDS) and slowly digestible starch (SDS) and total digestible starch (TDS), respectively. Each aliquot was immediately transferred into 20 mL of 50 mM acetic acid to stop the enzymatic reaction. These solutions were mixed, and 0.1 mL of each was incubated with 0.1 mL of amyloglucosidase (100 U/mL) to hydrolyze remaining maltose to glucose. Glucose content was then determined spectrophotometrically using the glucose oxidase/peroxidase reagent (GOPOD) at 510 nm (Varioskan™ LUX, ThermoFisher Scientific, Germany). SDS was calculated as the difference between the glucose content at 120 min and 20 min.
To quantify resistant starch (RS), a 4.0 mL aliquot was removed after 240 min, mixed with an equal volume of 50 % v/v ethanol, and centrifuged. The pellet was washed with aqueous ethanol to remove free glucose, then solubilized in sodium hydroxide to dissolve the resistant starch fraction. After neutralization, the sample was incubated with AMG to hydrolyze starch into glucose, which was again measured using GOPOD reagent.

The contents of RDS, SDS and RS were obtained with the following equations (2), (3) and (4).

$$RDS(\%) = 100\ x\ (G20 - G0)\ x\ 0.9/TS \quad (2)$$

$$SDS(\%) = 100\ x\ (G120 - G20)\ x\ 0.9/TS \quad (3)$$

$$RS(\%) = 100 - RDS - SDS \quad (4)$$

where G0, G20, G120 is the glucose released within 0, 20, 120 min, respectively. TS represents the total starch content.

For the estimation of the glycemic index (GI), an additional aliquot was taken at 60 minutes. The hydrolysis curve was constructed by plotting the amount of reducing sugars released at different time intervals (0–240 min). The hydrolysis index (HI) for each sample was calculated by dividing the area under the hydrolysis curve (AUC) of the sample by the AUC of a reference food (white bread). The estimated GI was then calculated using Equation (5) proposed by Goñi et al. (1997). All analyses were performed in triplicate.

$$GI = 39.71 + 0.549\ x\ HI \quad (5)$$

2.9 Bioaccesibility of phenolic compounds

The *in vitro* gastrointestinal digestion procedure included both gastric and intestinal phases and was conducted following the method described by Nieto Calvache et al. (2016) and Saura-Calixto et al.,

(2007) with minor modifications. Briefly, 1.2 g of sample were mixed with 30 mL of a pepsin-HCl solution (0.3 g/100 mL pepsin in 0.04 N HCl) and incubated at 37 °C for 2 hours with orbital shaking at 120 rpm to simulate gastric digestion. After this stage, the pH was adjusted to 7.5–8.0 using 2 N NaOH, and 40 mL of an intestinal solution (0.05 M KH_2PO_4) containing 0.6 g/100 mL bile salts and 0.3 g/100 mL pancreatin was added. The mixture was incubated for another 2 hours at 37 °C under the same shaking conditions to simulate intestinal digestion. After digestion, the samples were centrifuged (3000 g, 15 min) and the supernatants were collected. The remaining insoluble indigestible fraction was washed twice with 5 mL of distilled water, and the resulting supernatants were pooled. The combined supernatants were transferred into dialysis tubes (molecular weight cut-off: 12,000–14,000 Da) and dialyzed against distilled water for 48 h at 37 °C (water flow: 7 L/h) to remove bile salts and other low molecular weight compounds. The dialyzed solution, corresponding to the soluble indigestible fraction, was frozen, whereas the insoluble indigestible fraction was freeze-dried and stored at -18 °C for further characterization.

The phenolic compound content of both the soluble and insoluble indigestible fractions was evaluated using the spectrophotometric techniques described below. In the insoluble fraction, both free (FPP) and bound (BPP) phenolic compounds were quantified. The amount of phenolic compounds potentially accessible in the small intestine was calculated as the difference between the total phenolic compound content (free and bound) in the original sample and the phenolic compounds associated with both the soluble and insoluble indigestible fractions. Additionally, the antioxidant capacity of the free phenolic compound extracts from the matrices was also evaluated before and after digestion (soluble and insoluble fractions).

2.9.1 Free and bound phenolic compounds content

Phenolic compounds are categorized into free phenolic compounds (FPP), which are extracted with aqueous-organic solvents, and bound phenolic compounds (BPP) usually attached to the cell wall which need an acid hydrolysis to be extracted because they are bound to macromolecules like proteins, dietary fiber or polysaccharides from the cell wall.

The FPP extraction and analysis was based on the method described by Pico et al. (2020) with any modifications. 2 g (±0.0500 g) of powder sample was weighed in a centrifuge tube and 8 mL of 80 % methanol in 0.1% formic acid were added for the first extraction during 15 min using a wrist shaker. The sample was centrifuged for 5 min at 3500 rpm and 20 °C and 40 µL of 2 % EDTA were added to the supernatant for the stabilization of flavan-3-ols. The sample solution was kept on ice. 8 mL of 70 % acetone in 0.1 % formic acid were added to the pellet for a second extraction for 15 min using the wrist shaker. After centrifugation, the supernatant was combined with the methanolic extract from the first extraction and made up to 20 mL with deionized water. The extract was kept at −80 °C for the colorimetric determination by Folin-Ciocalteu reaction.

The pellet was also kept at −80 °C for further sequential determination of BPP. The extraction of this fraction was based on the method described by Pico et al. (2019). 0.8 g (± 0.0050 g) of sample from the FPP pellet were weighed in a glass tube and 10 mL of methanol/sulfuric acid (90/10) were added. The hydrolysis was carried out for 22 h at 85 °C, with magnetic stirring. The sample was then centrifuged at 3500 rpm for 20 min and the supernatant was made up to 25 mL with deionized water. The extract was kept at −80 °C for the colorimetric determination by Folin-Ciocalteu reaction.

For the Folin-Ciocalteu reaction, 260 µL of Milli-Q water, 26 µL of 7.5 % (v/v) Na_2CO_3, 20 µL of extract and 20 µL of Folin–Ciocalteu reagent were mixed in a 96-well plate. The samples were incubated for 1 h at room temperature in the dark and the spectrophotometric determination was performed at 765 nm in a multimode microplate reader Varioskan™ LUX (ThermoFisher Scientific,

Germany). For the blank, 20 µL of deionized water were used. The total content of FPP and BPP were expressed as mg of gallic acid equivalents (GA)/100 g dry matter, based on a GA calibration curve. All analyses were carried out in quadruplicate and the handle of the reagents was performed in conditions as dark and cold as possible.

2.9.2 Antioxidant activity

Two antioxidant assays were employed to evaluate the antioxidant activity of the free polyphenol extracts from the samples before and after digestion. The 1,1-diphenyl-2-picrylhydrazyl (DPPH) radical scavenging activity was determined following the method described by Luo et al. (2022) with slight modifications. Briefly, 15 µL of the sample solution was mixed with 300 µL of a 30 mg/L DPPH solution and incubated in the dark at room temperature for 30 minutes. Absorbance was then measured at 515 nm. Results were expressed as µmol Trolox equivalents per gram of dry weight (µmol TE/g d.w.) of the original sample, using 90% ethanol as the blank.

The 2,2′-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) (ABTS) radical scavenging activity was assessed based on the procedure of Luo et al. (2022) with minor modifications. A 7.4 mM ABTS stock solution was mixed with an equal volume of 2.4 mM potassium persulfate and allowed to react in the dark at room temperature for 12–16 hours to generate the radical solution. This solution was then diluted with deionized water to an absorbance of 0.70 ± 0.02 at 734 nm. Subsequently, 10 µL of sample solution was added to 290 µL of the ABTS solution, incubated in the dark for 30 minutes at room temperature, and the absorbance was measured at 734 nm. Deionized water was used as the blank, and results were expressed as µmol TE/g d.w. of the original sample.

2.10 Statistical analysis

Statistical analyses were performed with at least three replicates. Results were expressed as mean value ± standard deviation and statistically evaluated for differences between means using a Tukey's test, at a 95% confidence interval using IBM® SPSS® Statistics (Version 27, IBM Corp., Armonk, NY, USA).

3 Results and discussion

3.1 X-Ray diffraction (XRD)

The X-ray diffraction (XRD) analysis allowed the evaluation of structural modifications within the different matrices before and after extrusion, providing insights into changes in starch crystallinity and the influence of the different components of the food matrix.

Tapioca starch exhibited a characteristic A-type diffraction pattern (Fig. 1a), with well-defined peaks at 2θ of 15°, 17.1°, 17.8°, and 23°, consistent with previous studies on this raw material (Atichokudomchai et al., 2001; Aviara et al., 2010). The crystallinity value (Table 2) was higher than those reported in other studies with values of 39% (Abedi & Pourmohammadi, 2020), 42% (Dome et al., 2020) and 40% (Atichokudomchai et al., 2001). Meanwhile, SBP exhibited a characteristic semicrystalline pattern (Fig. 1a), featuring a broad amorphous hump near 18° and a distinct crystalline peak of ~22° at 2θ (Osman et al., 2020; Segal et al., 1959; S. Wang et al., 2019), where cellulose is reported as the main contributing component (S. Wang et al., 2019). The crystallinity level observed in SBP (Table 2) was similar to that reported for blackberry pomace (37%) (Osman et al., 2020), but higher than those found in pomace from cranberry, blueberry, Concord grape, and apple (ranging between 18% and 34%) (S. Wang et al., 2019). The non-extruded mixture (NEM) retained the characteristic features of native starch (Ts), although its crystallinity decreased due to the presence

of strawberry by-products (Table 2, Fig. 1a). In contrast, E-SBPTs showed a drastic reduction in crystallinity (Table 2, Fig. 1a) due to the significant degradation and macromolecular modification of starch caused by shear forces and extrusion temperature (Neder-Suárez et al., 2024). Similar findings have been reported for extrudates formulated with blue corn flour, spinach powder, and black beans (Neder-Suárez et al., 2024), as well as for extruded green banana flour (Pico et al., 2019) and corn grits blended with pineapple stem starch in extruded snacks enriched with oyster mushroom powder (Tangsrianugul et al., 2023).

Extrusion significantly reduced crystallinity across all thermally treated samples, as expected due to starch gelatinization and the disruption of its crystalline structure during processing. Extruded tapioca starch (E-Ts) retained slightly higher crystallinity, suggesting that in the absence of other components such as fiber or phenolic compounds, starch underwent partial post-extrusion reorganization, maintaining some crystalline domains.

Table 2. X-ray diffraction crystallinity (%) of tapioca starch (Ts), strawberry by-products (SBP), and both non-extruded and extruded matrices, and macromolecular characteristics of starch-containing samples

Matrix	% Crystallinity	Mn (kDa)	Mp (kDa)	Mw (kDa)
SBP[A]	41.4 ± 0.2[b]	-	-	-
Ts	51.7 ± 0.1[a]	100560 ± 2886[a]	260850 ± 6949[a]	147805 ± 3249[a]
NEM	49.3 ± 0.2[c]	89142 ± 5176[b]	227731 ± 13788[b]	132726 ± 8410[b]
E-Ts	5.8 ± 0.0[e]	10644 ± 673[d]	11834 ± 593[d]	13771 ± 828[d]
E-SBPTs	4.8 ± 0.1[d]	3618 ± 551[e]	4048 ± 551[e]	4514 ± 660[e]
E-CWTs	3.3 ± 0.2[f]	11374 ± 100[d]	12274 ± 162[d]	15410 ± 148[d]
E-PPTs	4.3 ± 0.1[g]	8980 ± 52[e]	9117 ± 7[e]	13549 ± 24[e]
E-PPCWTs	5.2 ± 0.1[d]	7894 ± 2[e]	8292 ± 39[e]	10660 ± 27[e]

SBP: strawberry by-products; **Ts**: Native tapioca starch; **NEM**: non-extruded mixture of strawberry by-products and tapioca starch; **E-Ts**: Extruded tapioca starch; **E-SBPTs**: extruded snack formulated with strawberry by-products and tapioca starch; **E-PPCWTs**: extruded composite matrix of phenolic compounds extract, cell wall material (from SBP), and tapioca starch; **E-PPTs**: extruded composite matrix of phenolic compounds extract (from SBP) and tapioca starch; **E-CWTs**: extruded composite matrix of cell wall material (from SBP) and tapioca starch; **Mw**: Weight-average molar mass, **Mn**: number-average molar mass; **Mp**: peak molar mass.
[A]Strawberry by-products crystallinity was calculated respect to cellulose according to Segal et al. (1959) method.
Different letters in the same column indicate significant differences ($p < 0.05$).

The composite matrices incorporating strawberry by-product fractions exhibited distinct crystallinity responses (Table 2). Crystallinity increased in the following order: E-CWTs < E-PPTs < E-CWPPTs. This suggested cell wall material incorporation may have further interfered with starch reorganization, similar to findings on corn starch behavior with different levels of cellulose incorporation (Ek et al., 2021). In comparison, phenolic compounds may have a lesser impact on the loss of crystalline structure than fiber. Additionally, a minor peak appeared at 2θ of 20.23° in E-PPTs, also present in E-PPCWTs, along with a slight signal at 12.80° (Fig. 1b), which has been associated with V-type starch patterns linked to amylose-lipid complex formation and starch retrogradation (Lv et al., 2022; Ma et al., 2021; Neder-Suárez et al., 2024; van der Sman et al., 2018; Zeng et al., 2022). However, these patterns may also result from interactions between phenolic compounds and starch, as previously reported (D. B. Amoako & Awika, 2016, 2019; Guo et al., 2019; Huo et al., 2025; Y. Li et al., 2023; R. Wang et al., 2022; Wu et al., 2024), which is worth considering since this signal was absent in E-CWTs (Fig. 3b). Notably, these signals were also observed in E-SBPTs and at similar angles in E-Ts, supporting the possibility of multiple concurrent processes since extrusion disrupts starch molecular chains, reducing molecular weight and increasing the presence of smaller amylose and amylopectin fractions, which may enhance their reorganization and interactions with other components (Lv et al., 2022; Zeng et al., 2022). The E-PPCWTs may exerted combined effect of both components, where fiber and phenolic compounds interacted with the starch matrix, partially limiting its post-extrusion reorganization but not causing as pronounced a disruption as when cell wall material alone was incorporated.

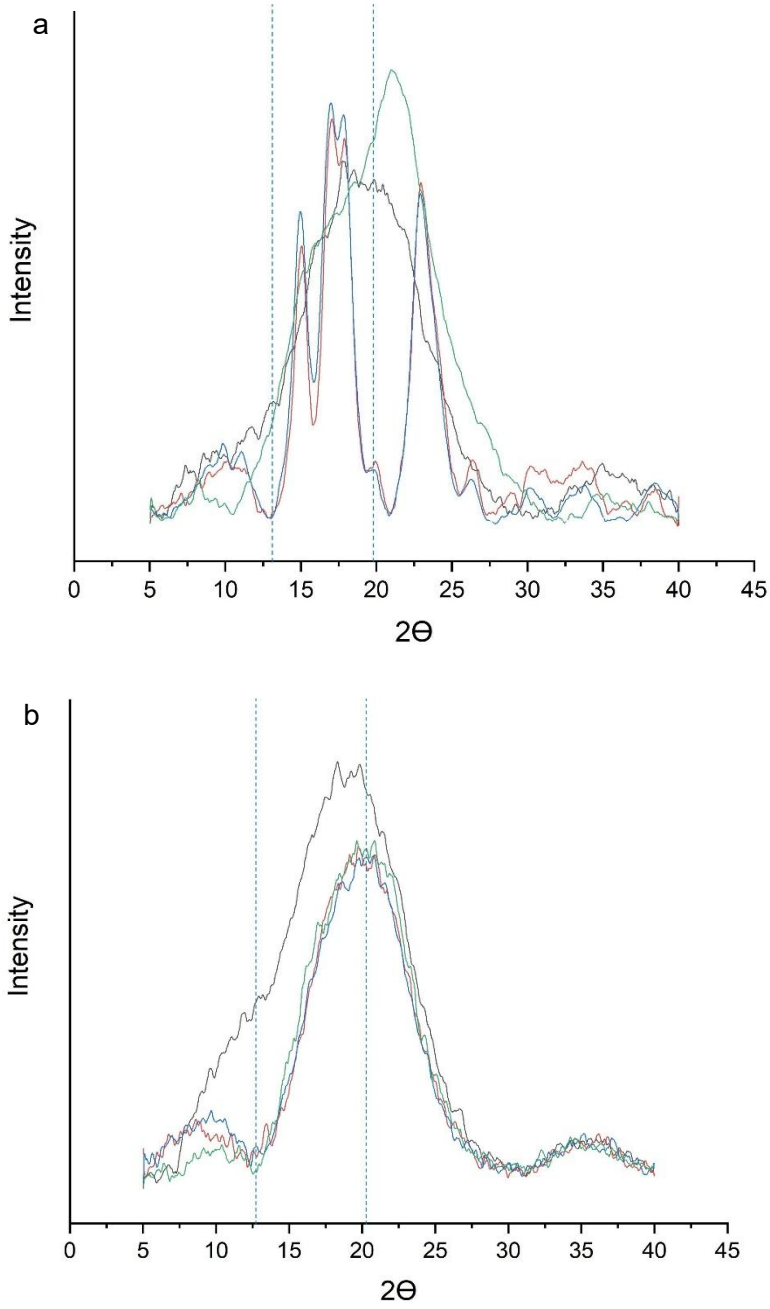

Fig. 1. X-ray diffractograms of (a) – tapioca starch (Ts), – strawberry by-products (SBP), – non-extruded mixture (NEM) and – extruded snack (E-SBPTs), (b) – extruded tapioca starch (E-Ts) and composite matrices containing Ts and cell wall (– E-CWTs) or phenolic compounds extract (– E-PPTs) or both (– E-PPCWTs)

3.2 Molar mass distribution of the starch

The starch present in the non-extruded mixture (NEM) (Fig. 2a) exhibited a typical bimodal profile, similar to that of tapioca starch (Ts) (Fig. 2b), with a main peak at an elution volume of approximately

5.4 mL, corresponding to amylopectin, and a shoulder at around 5.6 mL, associated with amylose (Sagnelli et al., 2016; Yang et al., 2020).

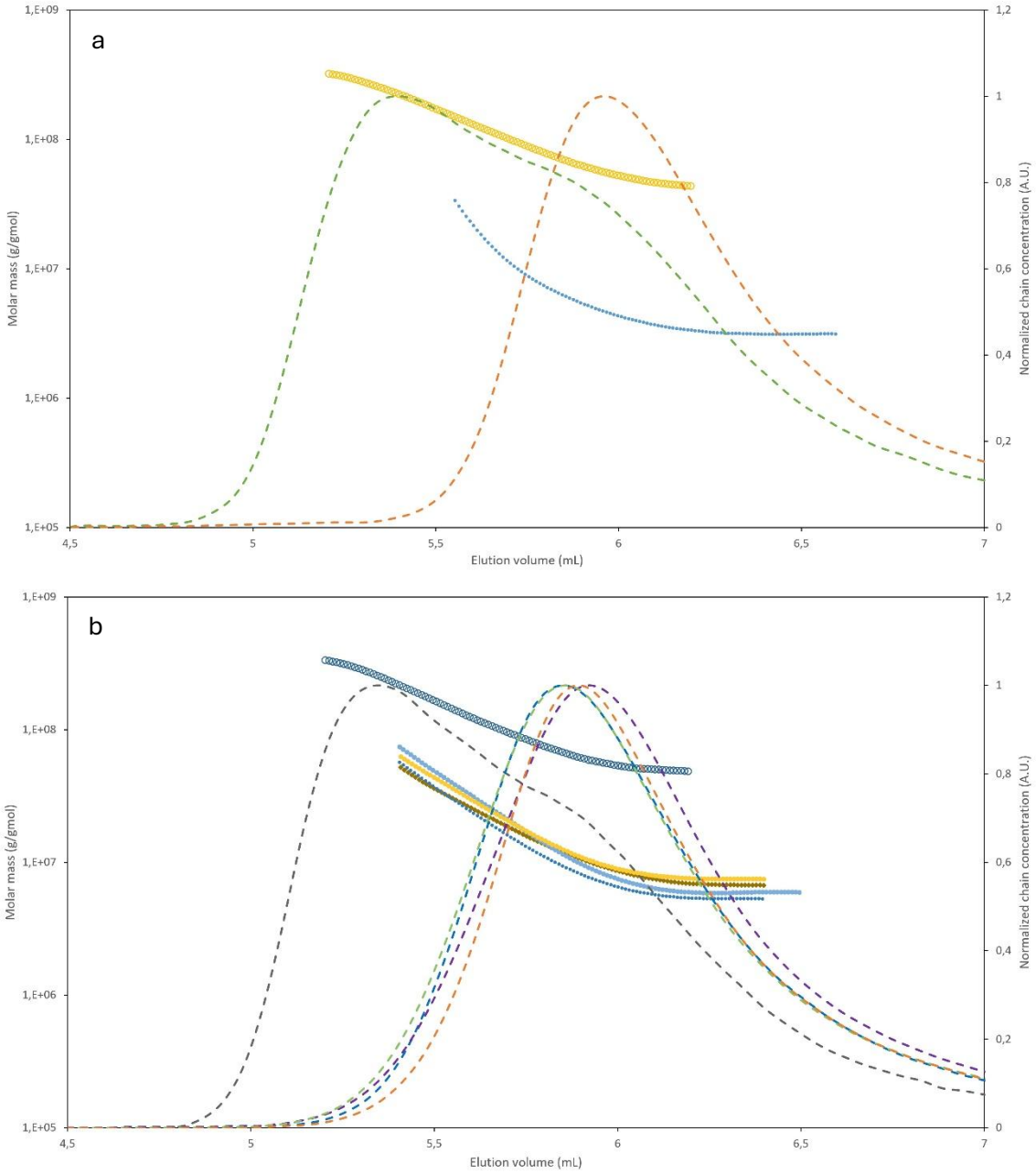

Fig. 2. HPSEC-MALLS chromatograms (RID signal, dashed lines) and molar mass distribution (dotted lines) as a function of elution volume for (a) ○ --- non-extruded mixture (NEM) and • --- extruded snack (E-SBPTs); and (b) ○ --- tapioca starch (Ts), ▪ --- extruded tapioca starch (E-Ts), and composite matrices containing Ts and cell wall (• --- E-CWTs) or phenolic compounds extract (• --- E-PPTs) or both (• --- E-PPCWTs)

As shown in Table 2, Ts exhibited a weight-average molar mass (Mw) of 147.8 kDa, while NEM presented a lower Mw value of 132.7 kDa. This difference could be attributed to mechanical effects during mixture preparation, which may have caused partial disaggregation of the starch chain conformation either through direct structural modification or via interactions between starch and other components from the strawberry by-products (Hernández-Bautista et al., 2025).

Following extrusion, a marked degradation of starch was observed. In E-SBPTs and E-Ts, Mn, Mp, and Mw values were drastically reduced compared to non-extruded samples, indicating intense fragmentation of polymer chains under the thermal and mechanical shear conditions of the extrusion process. High temperature, pressure, and shear forces generated during extrusion can readily disrupt hydrogen bonds among starch molecules, especially in amylopectin (Huang et al., 2022), leading to their modification. Regarding the composite matrices (Fig. 2b), E-CWTs showed molar mass values similar to E-Ts. However, in the presence of phenolic compounds (E-PPTs and E-PPCWTs), molar mass decreased significantly. This may be associated with enhanced degradation of both amylopectin and amylose fractions. It has been reported that starch degradation during extrusion is size-dependent (Liu et al., 2010) and that glycosidic bonds near amylopectin branching points are more susceptible to cleavage, greatly reducing the overall size of these molecules (Yang et al., 2020). M. Li et al. (2014) further suggest that the rigid crystalline regions of amylopectin are more vulnerable to shear degradation, whereas amylose, with its smaller, flexible, and amorphous structure, is less affected. Consequently, the proportion of molecules within the molecular weight range of amylose increases after extrusion due to the extensive breakdown of amylopectin into smaller fragments (Huang et al., 2022). Based on this, a lower crystallinity would be expected in these samples, as observed for extruded corn starch (M. Li et al., 2014), considering that amylose is less crystalline than amylopectin in its native form. However, amylose–ligand associations, such as with phenolic compounds, could lead to the formation of highly crystalline structures (D. B. Amoako & Awika, 2019). For HPSEC-MALLS analysis, starch was first isolated through a series of purification steps, which may result in the partial or complete dissociation of starch–polyphenol complexes. This may have exposed starch chains that, having been stabilized by such interactions, had not undergone retrogradation or natural structural reorganization. Once released, these chains, potentially shorter, degraded, or more linear, were recorded as lower molecular weight fractions during analysis, which could explain the reduced Mw values observed in E-PPTs and E-PPCWTs.

3.3 In vitro starch digestibility and estimated glycemic index (GI)

In vitro starch digestibility was evaluated using three fractions: rapidly digestible starch (RDS), slowly digestible starch (SDS), and resistant starch (RS), along with the total digestible starch (TS) fraction (Table 3).

Extrusion significantly increased RDS in all samples compared to the non-extruded mixture (NEM). This effect was expected as the most common outcome of food extrusion is the degradation of starch granules, which improves their digestibility. High shear stress during extrusion destroys the integrity of starch granules, increasing the contact area between starch and amylase during hydrolysis (Qiu et al., 2024). Additionally, starch gelatinization induced by thermal and mechanical treatment during extrusion enhances the accessibility of digestive enzymes (Dehghan-Shoar et al., 2011). Native starch granules are inherently more resistant to digestion than gelatinized starch due to their crystalline and granular structures (Huang et al., 2022). Similar results have been observed in extrudates based on tapioca starch with grape pomace (Oladiran & Emmambux, 2018), barley flour with grape and tomato by-products (Altan et al., 2009), rice-based extrudates with orange peel, grape seeds, and tomato pomace (Yaĝci & Göĝûş, 2009) and corn-based extrudates containing tomato skin, seeds, and paste (Dehghan-Shoar et al., 2011).

Regarding SDS, E-Ts exhibited the lowest proportion, whereas composite matrices containing both phenolic compounds and cell wall (E-CWTs and E-PPCWTs) showed higher SDS values, suggesting that these compounds may have limited starch digestion by forming compact structures or due to interaction with the starch granule (Mohamed, 2023; Robin et al., 2012) or to inhibition of amylase as phenolic compounds, and mainly proanthocyanidins, interact with proteins leading to their

inhibition (Le Bourvellec & Renard, 2012). The SDS level in E-SBPTs, although higher than in E-Ts, was significantly lower than in the composite matrices. This difference could be partially attributed to the moisture content of the mixtures, as a high water content during extrusion has been reported to increase the proportion of slowly digestible starch by promoting the formation of double helices and enhancing the degree of crystallinity, which influences starch swelling, gelatinization, and subsequent amylose retrogradation into short-ordered structures that affect digestibility (Lv et al., 2022; Sumargo et al., 2016; Zhang et al., 2022).

Table 3. *In vitro* starch digestibility values (g/100 g total starch) and estimated glycemic index of the non-extruded and extruded analyzed matrices

Matrix	RDS	SDS	RS	GI
NEM	1.7 ± 0.2^{a}	13.3 ± 0.7^{a}	72.1 ± 2.5^{a}	46.9 ± 0.5^{a}
E-Ts	81.6 ± 1.7^{b}	3.3 ± 0.4^{c}	1.2 ± 0.1^{b}	82.1 ± 2.4^{c}
E-SBPTs	85.9 ± 3.4^{b}	6.8 ± 1.0^{b}	1.4 ± 0.1^{b}	77.6 ± 1.3^{b}
E-PPCWTs	78.4 ± 5.8^{b}	15.9 ± 1.7^{a}	1.9 ± 0.0^{d}	77.1 ± 0.5^{b}
E-PPTs	77.5 ± 3.3^{b}	27.8 ± 2.9^{d}	2.6 ± 0.1^{c}	79.3 ± 1.0^{bc}
E-CWTs	79.2 ± 3.6^{b}	21.8 ± 2.3^{e}	2.5 ± 0.3^{c}	76.2 ± 1.9^{b}

NEM: non-extruded mixture of strawberry by-products and tapioca starch; **E-Ts**: Extruded tapioca starch; **E-SBPTs**: extruded snack formulated with strawberry by-products and tapioca starch; **E-PPCWTs**: extruded composite matrix of phenolic compounds extract, cell wall material (from SBP), and tapioca starch; **E-PPTs**: extruded composite matrix of phenolic compounds extract (from SBP) and tapioca starch; **E-CWTs**: extruded composite matrix of cell wall material (from SBP) and tapioca starch; **RDS**: Rapidly digestible starch, **SDS**: Slowly digestible starch, **RS**: Resistant starch, **GI**: Estimated glycemic index

Different letters in the same column indicate significant differences ($p < 0.05$)

Among the composite matrices, E-PPTs exhibited the highest SDS content (27%), followed by E-CWTs (21.8%), which also showed the highest proportion of RS (2.6% and 2.5%, respectively) compared to E-PPCWTs. This suggests that the presence of phenolic compounds and fiber may have altered starch reorganization during extrusion, promoting the formation of structures partially resistant to enzymatic digestion. During extrusion, the increase in shear stress could lead to the fragmentation of starch molecules, reducing their molecular weight, shortening amylose chains, and debranching amylopectin. These changes in starch structure may enhance its interaction with non-starch polysaccharides, phenolic compounds, and other compounds (Mohamed, 2023). Another potential reason for the reduced starch digestibility in these products is competition for available water by fiber components, which limits starch gelatinization and decreases its susceptibility to digestion (Dehghan-Shoar et al., 2011), Additionally, phenolic compounds may exert an inhibitory effect on digestive enzymes (Oladiran & Emmambux, 2018).

In E-PPCWTs, where both cell wall fiber and phenolic compounds were combined, SDS and RS values were the lowest among composite matrices (15.9% and 1.9%, respectively). This suggests that the combination of fiber and phenolic compounds reduced their individual effects on starch digestibility, possibly due to interactions between these components (D. B. Amoako & Awika, 2019; Jakobek, 2015; X. Liu et al., 2020; Renard et al., 2017), which may have limited the ability of phenolic compounds to stabilize the starch structure or the ability of fiber to modify its enzymatic accessibility. The entrapment of phenolic compounds within the fiber network in E-PPCWTs may explain the reduced effect compared to E-PPTs, where phenolic compounds were more available.

According to the starch digestibility results, the glycemic index (GI) of the extruded matrices was significantly higher than that of the non-extruded sample (NEM) (Table 3). In general, the extruded samples exhibited GI values ranging from 76.2 to 82.1. These values were slightly lower than those reported by (Oladiran & Emmambux, 2018) for tapioca starch extrudates with grape pomace inclusion. E-Ts had the highest GI value (82.1), which aligns with its high starch content and

digestibility level. The inclusion of strawberry by-products in the formulation (E-SBPTs) slightly reduced the GI, suggesting that the presence of fiber and phenolic compounds had a moderate effect on starch digestion, as also observed in the digestibility results.

Among the composite matrices, E-CWTs and E-PPCWTs exhibited the lowest GI values (76.2 and 77.1, respectively), whereas E-PPTs had the highest (79.3). Although the differences were not statistically significant, even when compared to E-SBPTs (processed at lower moisture content), the slight reduction observed in E-CWTs and E-PPCWTs may suggest that incorporating cell wall fiber may have contributed to modifying the glycemic response of the extrudate through the mechanisms described earlier.

3.4 Bioaccesibility of phenolic compounds

The amount of phenolic compounds potentially bioaccessible in the small intestine was then determined for each sample (Table 4).

The results showed that approximately 2.1 mg/g of phenolic compounds from NEM and E-SBPTs were able to pass through the dialysis membrane, whereas only 0.48 mg/g were dialyzed from E-PPCWTs. These values corresponded to 45%, 51%, and 19% of the total phenolic compounds in the original dry samples, respectively (Fig. 3) representing the phenolic compounds considered as potentially bioaccessible in the small intestine. Compared to NEM, E-SBPTs exhibited higher phenolic compound bioaccessibility, indicating that under the processing conditions applied, extrusion may promote the release of phenolic compounds into the small intestine. In contrast, E-PPCWTs retained most of its phenolic content in the indigestible fraction due possibly to their interactions with the cell wall (X. Liu et al., 2020). Several studies have reported that phenolic compounds retained in the indigestible fraction reach the colon, where they become fermentable substrates for the commensal microbiota which convert them into readily absorbable low-molecular weight metabolites, as native compound they also can exert antioxidant activity (Herrera-Cazares et al., 2017; Saura-Calixto et al., 2007).

Table 4. Potential bioaccessibility of total phenolic compounds in the non-extruded mixture (NEM), extruded snack (E-SBPTs), and extruded composite matrix containing cell wall material, polyphenol extract, and tapioca starch (E-PPCWTs)

Matrix	**FPP in the matrix (mg/g)**	**BPP in the matrix (mg/g)**	**Soluble indigestible fraction**	**Insoluble indigestible fraction**		**Bioaccesible phenolic compounds (mg/g)**
			PP (mg/g)	**FPP (mg/g)**	**BPP (mg/g)**	
NEM	1.1 ± 0.1^{a}	3.5 ± 0.4^{a}	0.9 ± 0.0^{a}	0.9 ± 0.0^{a}	0.8 ± 0.0^{a}	2.1 ± 0.4^{a}
E-SBPTs	0.7 ± 0.1^{b}	3.5 ± 0.2^{a}	1.2 ± 0.1^{b}	0.4 ± 0.0^{b}	0.5 ± 0.0^{b}	2.2 ± 0.1^{a}
E-PPCWTs	0.3 ± 0.0^{c}	2.2 ± 0.1^{b}	0.1 ± 0.0^{c}	0.6 ± 0.0^{c}	1.3 ± 0.0^{c}	0.5 ± 0.1^{b}

NEM: non-extruded mixture of strawberry by-products and tapioca starch; **E-SBPTs**: extruded snack formulated with strawberry by-products and tapioca starch; **E-PPCWTs**: extruded composite matrix of phenolic compounds extract, cell wall material (from SBP), and tapioca starch; **FPP**: Free phenolic compounds, **BPP**: Bound phenolic compounds, **PP**: Phenolic compounds

Different letters in the same column indicate significant differences ($p < 0,05$). Values are expressed on a dry weight basis

The impact of extrusion process on phenolic compound bioaccessibility was influenced by both processing parameters and the composition of the matrix, which together regulate the degree and nature of structural modifications. In this study, differences in the moisture content during processing

of E-SBPTs and E-PPCWTs suggest that specific mechanical energy (SME) may play an important role in phenolic compound modification. Due to the lower moisture content in E-SBPTs, the SME was higher compared to E-PPCWTs (3544 and 2291 KJ/Kg, respectively; data not shown). A similar trend was observed in extrudates containing tomato derivatives, where the bioaccessibility of bioactive compounds increased with rising SME values (Dehghan-Shoar et al., 2011). As noted by the authors, extrusion involves intense thermal and mechanical processing, which reduces the particle size of insoluble fiber and can solubilize non-starch polysaccharides from the cell wall, thus increasing their accessibility. These physicochemical modifications may also lead to diverse interactions among matrix components, as previously discussed in Chapters 3 and 4, which ultimately influence the amount of phenolic compounds available for absorption in the gastrointestinal tract (Jakobek, 2015; R. Wang et al., 2022).

Hossain & Jayadeep (2022) also reported an increase in total phenolic compound bioaccessibility, up to 40%, in extruded whole corn flour, a value higher than that observed in the present study. Evaluations by Schmid et al. (2022), on extrudates make with corn starch and chokeberry pomace showed variable responses among phenolic compound classes: while the bioaccessibility of phenolic acids and flavonols increased, that of anthocyanins decreased, particularly with rising SME levels. Similarly, Herrera-Cazares et al. (2021) found that in mango bagasse-based extrudates, phenolic acids exhibited higher bioaccessibility, whereas xanthones and flavonoids were less bioaccessible.

In addition, structural features of the extrudate, such as its degree of cohesion and porosity, may also influence phenolic compound bioaccessibility (Hernández-Bautista et al., 2025). As observed in the previous chapter, the matrix structure of E-PPCWTs showed higher compactness compared to E-SBPTs, potentially limiting the release and diffusion of phenolic compounds during digestion.

The antioxidant activity of the non-extruded and extruded samples was evaluated before and after *in vitro* digestion using ABTS and DPPH radical scavenging assays (Fig. 4). In the undigested samples, the non-extruded mixture (NEM) exhibited the highest antioxidant capacity, followed by the extruded snack (E-SBPTs) and the composite matrix containing cell wall and polyphenol extract (E-PPCWTs). This trend reflects the total phenolic compounds content of each matrix and aligns with the differences in phenolic composition previously observed and interactions between phenolic compound and cell wall material.

Following *in vitro* digestion, antioxidant activity remained detectable in the insoluble fraction, which was associated with phenolic compounds entrapped within the food matrix. These phenolic compounds bound to the matrix will therefore reach the colon, where they are released through fermentation by the commensal microbiota and exert local bioactive effects (Herrera-Cazares et al., 2021; Luo et al., 2022; Saura-Calixto et al., 2007).

Interestingly, the soluble fraction after digestion, excluding the dialyzable portion, also showed measurable antioxidant capacity in all treatments. This suggests that certain antioxidant compounds released during digestion, although unable to diffuse through the dialysis membrane due to their molecular size or interactions with the food matrix (Cuvas-Limon et al., 2022), remain solubilized and potentially bioactive within the gastrointestinal environment.

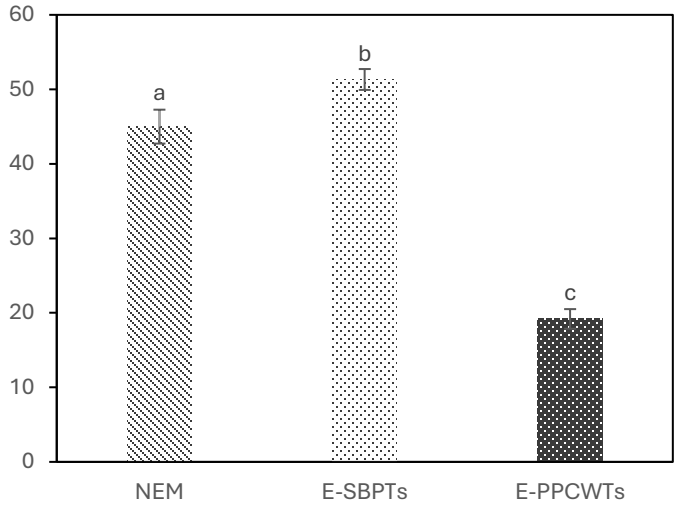

Fig. 3 Percentage of potential bioaccesibility of total phenolic compounds in the non-extruded mixture (NEM), extruded snack (E-SBPTs), and extruded composite matrix containing cell wall material, polyphenol extract, and tapioca starch (E-PPCWTs). Different letters between samples indicate significant differences ($p < 0,05$)

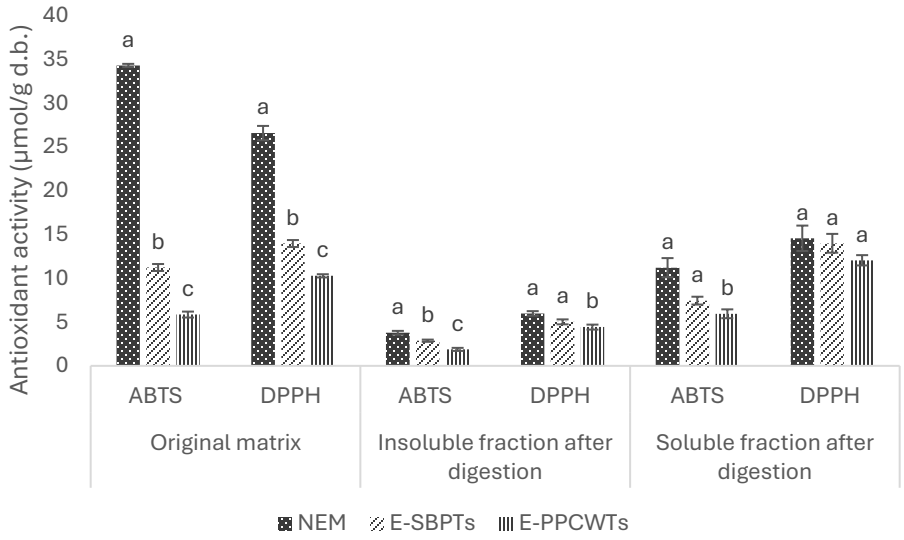

Fig 4. Antioxidant activity phenolic compounds in the non-extruded mixture (NEM), extruded snack (E-SBPTs), and extruded composite matrix containing cell wall material, polyphenol extract, and tapioca starch (E-PPCWTs) and their corresponding digestive fractions evaluated by ABTS and DPPH methods

Conclusions

This study demonstrated that the extrusion of tapioca starch-based matrices enriched with strawberry by-products and their structural fractions substantially modified starch digestibility, glycemic response, and phenolic compound bioaccessibility. Extrusion significantly increased rapidly digestible starch (RDS) and glycemic index (GI) across all samples, consistent with starch gelatinization and structural degradation under thermal and mechanical stress.

X-ray diffraction and HPSEC-MALLS analyses confirmed that extrusion induced substantial changes in starch crystallinity and molecular weight distribution. These modifications were influenced by the presence of fiber and phenolic compounds, which in some cases contributed to partial reorganization or the formation of starch–phenolic compound complexes. Such structural transformations are closely linked to starch digestibility outcomes, as reflected in the performance of the composite matrices.

Composite matrices containing cell wall fiber and phenolic compounds modulated starch digestibility by increasing slowly digestible starch (SDS) and resistant starch (RS) fractions compared to starch-only extrudates. However, when both fiber and phenolic compounds were combined in a single matrix, their individual effects were attenuated, suggesting possible interactions that influenced their functional responses. This finding emphasizes the importance of matrix composition in regulating nutritional outcomes.

The analysis of phenolic compound bioaccessibility and antioxidant activity revealed that extrusion enhances the release of phenolic compounds in some formulations, such as E-SBPTs, while in others, particularly the composite matrix E-PPCWTs containing both fiber and phenolic compounds, a substantial portion of these compounds remained associated with the indigestible fraction. This highlights the dual functionality of both fiber and phenolic compounds in modulating not only the structural organization of the matrix but also the release, transformation, and fate of bioactive compounds during digestion, but it also reflected the impact of extrusion conditions, which determined the extent of modification and interaction among matrix components. Moreover, the antioxidant activity results demonstrated that the nutritional potential of extruded matrices cannot be solely attributed to the dialyzable fraction. A significant part of the antioxidant capacity resided in the non-dialyzable fractions, both soluble and insoluble, emphasizing their potential bioactivity along the gastrointestinal tract, particularly in the colon. Together, these findings reinforce the need to consider the matrix composition and the distribution of phenolic compounds among digestible and non-digestible fractions when evaluating the health-promoting properties of fiber- and polyphenol-rich extruded foods.

Overall, these results contribute to a better understanding of how plant-based by-products and their fractions influence the nutritional quality of extruded products. The study offers valuable insights for the formulation of functional snacks with improved fiber and polyphenol retention, glycemic modulation, and enhanced structural integrity. Furthermore, the use of extrusion as an established, scalable technology offers promising opportunities for the industrial application of these findings in the development of commercial fiber- and phenolic compound-enriched snacks. Optimizing formulations and processing conditions could facilitate the production of market-ready products with enhanced nutritional and functional profiles, supporting the valorization of fruit by-products at an industrial scale.

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Chapter 6. General discussion

The valorization of agro-industrial by-products through their incorporation into food matrices has garnered increasing attention in the development of functional, sustainable, and high value-added products. Among these by-products, strawberry processing residues are a rich source of bioactive compounds such as phenolic compounds and dietary fiber, as demonstrated in Chapter 2. However, their integration into complex food formulations poses significant technological challenges. In this context, extrusion emerges as a promising technology that combines physical and chemical transformations under controlled conditions, enabling the modulation of ingredient structure and functionality.

This thesis addressed the implications of incorporating strawberry by-products (SBP) into a starch-based puffed extrudate from complementary perspectives, with the aim not only of optimizing techno-functional properties but also to enhance the understanding of structural modifications occurring during processing and their relationship with the hypoglycemic properties (starch digestibility and *in vitro* glycemic index) of snack containing both phenolic compounds and fiber.

As shown in Chapter 3, variations related to the extrusion process, such as SBP inclusion level, feed moisture, and die temperature, affected physical quality attributes and mainly phenolic compound content, compare to raw material content including proanthocyanidins and their structural characterization such as degree of polymerization and constitutive unit composition. Fig. 1 presents a principal component analysis (PCA) of all variables analyzed in each treatment. Figure 1a shows the biplot of the PCA applied to the studied variables, while Figure 1b displays the distribution of treatments projected onto the plane defined by the first two principal components (PC1 and PC2), which together explain 70.4% of the total variance (PC1: 46.4 %, PC2: 24.2 %).

In the upper right quadrant, most individual phenolic compounds identified by UHPLC-DAD (C-HexA: Coumaroylhexoside acid; p-C-HexA: p-coumaroylhexoside acid; Q: Quercetin; Q-3-GlcA: Quercetin-3-*O*-glucuronide; K-3-GlcA: Kaempferol-3-*O*-glucuronide; K-3-MalGlc: Kaempferol-3-*O*-malonylglucoside; Pg-3-Glc: Pelargonidin-3-*O*-glucoside; Cat: (+)-Catechin; EpiCat: (-)-Epicatechin, along with proanthocyanidins, the main phenolic compounds of strawberry and free polyphenol content (FPP) and color index (CI), clustered together. Their moderate correlation may indicate a common variation pattern associated with treatments 2 and 6 (Fig. 1b), which involved higher SBP content (25 %) and lower die temperature (180 °C). The correlation with color index suggests that the presence of these phenolic compounds may be moderately associated with increased snack coloration, due to their direct pigment contribution (i.e. anthocyanins) or their role in reducing oxidative degradation that affects color stability (Ačkar et al., 2018; Aussanasuwannakul et al., 2022; Falfán Cortés et al., 2014).

In the upper left quadrant, variables related to textural and physicochemical properties such as lightness (L^*), expansion index (EI), spatial frequency of ruptures related to crispness (NSr), and proanthocyanidins characteristics as their average degree of polymerization (mDP), bound phenolic compounds (BPP), and (-)-epicatechin as extension unit of proanthocyanidins, along with specific mechanical energy (SME), clustered together. These variables were associated with treatments involving low feed moisture and SBP inclusion (runs 1 and 3, Fig. 1b), or high inclusion levels at elevated temperature (run 4, Fig. 1b). Such conditions simultaneously promoted greater product expansion and thus higher crispness (NSr) and lightness (L^*), due to the formation of less compact air cells, a greater number of bubbles during extrusion cooking, and thinner cell walls, which affect light reflection (Chan et al., 2019). SME showed a strong

association with increased mDP, likely due to various chemical reactions involving terminal and extension units of (-)-epicatechin (Epicat T and E), which positively correlated with SME. In contrast, the total content of PACs and their (+)-catechin subunits both as terminal and extension (Cat T and E) projected in the opposite direction of SME and mDP and were associated with high-moisture treatments such as runs 7 and 8 (Fig. 1b). These relationships indicate that proanthocyanidin modification were strongly influenced by SME, which could modulate the proanthocyanidins composition (i.e. ratio between extension units), and reactions such as oxidation and hydrolysis (Ananingsih et al., 2013; De Taeye et al., 2014; La Mantia et al., 2023; Lončarić et al., 2018).

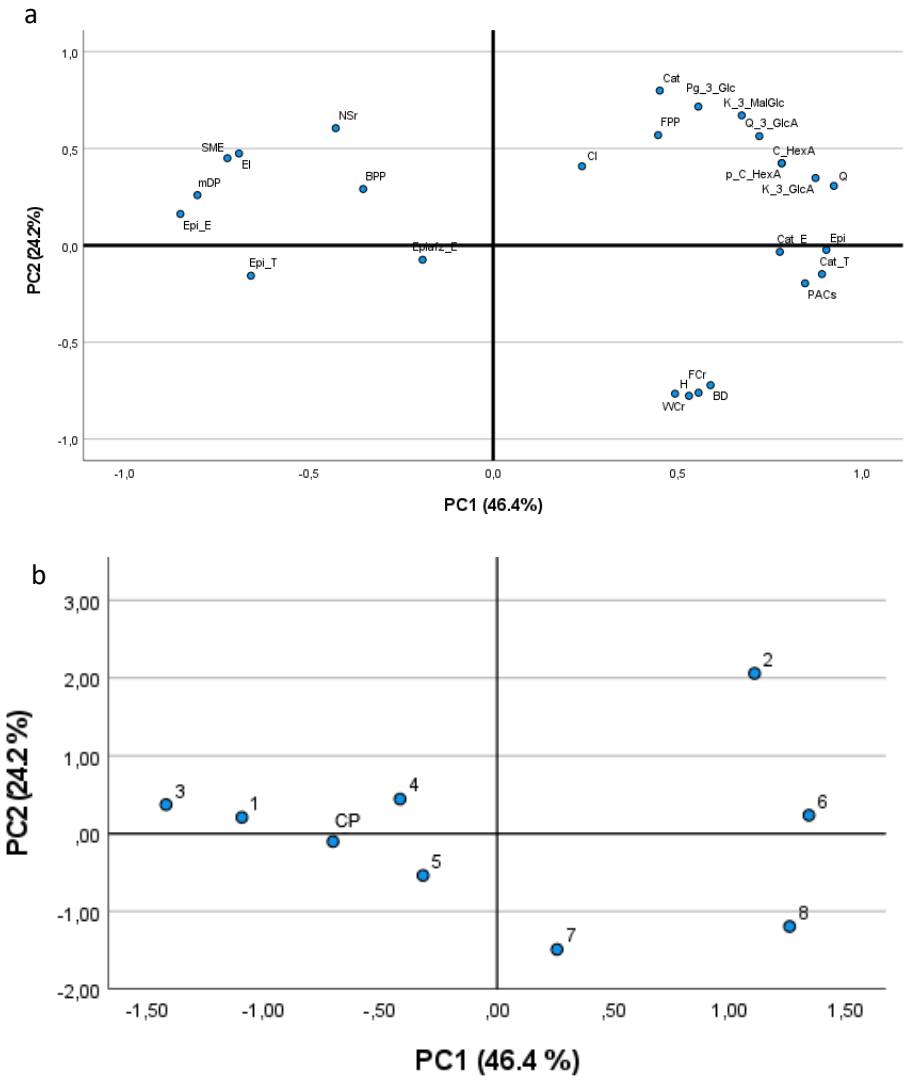

Fig. 1. PCA plot of (a) the variables analyzed and (b) the treatments applied in the optimization of the puffed snack.
EI: Expansion index, BD: Bulk density, H: Hardness, FCr: Crushing force, NSr: Spatial frequency of ruptures, WCr: Crispness work, CI: Color index, SME: Specific mechanical energy, FPP: Free phenolic compounds, BPP: Bound phenolic compounds, PACs: Proanthocyanidins; p-C-HexA: p-coumaroylhexoside acid; Q: Quercetin; Q-3-GlcA: Quercetin-3-*O*-glucuronide; K-3-GlcA: Kaempferol-3-*O*-glucuronide; K-3-MalGlc: Kaempferol-*O*-3-malonylglucoside; Pg-3-Glc: Pelargonidin-3-O-glucoside; (+)-Cat: (+)-Catechin; (-)-Epi: (-)-Epitechin, mDP: Mean Degree of Polymerization; % (+)-Cat T: Percentage of (+)-catechin as terminal unit; % (-)-Epi T: Percentage of (-)-epicatechin as terminal unit; % (+)-Cat E: Percentage of catechin as extension unit; % (-)-Epi E: Percentage of (-)-epicatechin as extension unit; % Epiafz E: Percentage of epiafzelechin as extension unit.
Treatments applied in the optimization of the puffed snack, expressed as strawberry by-products content (%)/die temperature (°C)/feed moisture (%), where 1: 15/180/20, 2. 25/180/20, 3: 15/200/20; 4: 25/200/20; 5: 15/180/24; 6: 25/180/24; 7: 15/200/24; 8: 25/200/24; CP: Central point 20/190/22

In the lower right quadrant, variables such as hardness (H), crispness force (FCr), crispness work (WCr), and bulk density (BD) clustered together. This alignment suggested that the corresponding runs, associated with high temperature and moisture (runs 7 and 8), yielded denser and harder products with reduced expansion, traits that may negatively affect sensory acceptance.

Based on the central composite design analysis, optimal extrusion conditions were defined to maximize EI and phenolic compound content (FPP and BPP) while minimizing BD and WCr: die temperature (DT) 192.01 °C, feed moisture (FM) 18.64 %, and SBP content 16.46 %. These conditions were experimentally validated, showing good model fitting. In addition to the analysis in Chapter 3, Table 1 compares the bulk density (BD) and textural properties of a commercial starch-based puffed snack (Kibo®, Colombia), formulated with rice, bean, oat, and sacha inchi, with those of the optimized snack developed in this study. This commercial product was selected as a relevant market reference, as it represents one of the few extruded snacks enriched with plant-based ingredients available locally. Despite differences in formulation, the optimized SBP snack exhibited characteristics aligned with this type of commercial product. According to t-test analysis ($p < 0.05$), both snacks showed similar BD and textural properties, with the optimized snack presenting a significantly higher NSr value, indicating greater crispness and lower crispness work (WCr).

Table 1. Comparison of the properties of the optimized puffed snack with strawberry by-products (SBP) and a commercial puffed snack

Response variable	Optimized puffed snack with SBP	Commercial puffed snack
BD (g/cm^3)	0.25 ± 0.02^a	0.21 ± 0.02^a
H (N)	25.03 ± 2.82^a	21.79 ± 2.25^a
FCr (N)	12.71 ± 1.26^a	12.60 ± 1.30^a
NSr (mm-1)	19.86 ± 1.14^a	12.64 ± 1.23^b
WCr (N.mm)	0.70 ± 0.09^a	0.97 ± 0.08^b

BD: Bulk density, **H**: Hardness, **FCr**: Average crushing force, **NSr**: Spatial frequency of ruptures, **WCr**: Crispness work.
Different letters in the same row indicate significant differences ($p < 0.05$) by *t*-test.

The changes induced by the extrusion process result from the interaction of multiple factors, and understanding their implications on the behavior of plant-based raw materials requires insight into how key constituents or potentially functional compounds are affected. In this regard, Chapters 3 and 4 aimed to deepen the analysis of matrix composition changes by using isolated SBP fractions (cell wall and polyphenol extract), in order to independently evaluate their impact on bioactive compound stability, cell wall composition, starch structure, digestibility, and glycemic index. This systematic approach revealed that extrusion of cassava starch-based matrices enriched with strawberry by-products and their structural fractions significantly altered the composition and organization of the main components, with direct effects on both nutritional and structural functionality.

From the phenolic perspective, a marked reduction in the content of most individual phenolic compounds was observed when SBPs were processed alone, particularly anthocyanins and flavonoids, with losses exceeding 95 % in some cases. However, compounds, such as kaempferol-3-*O*-glucuronide and (-)-epicatechin, increased after extrusion, suggesting that the process may have promoted their release or conversion from more complex structures, especially (-)-epicatechin which could be released after proanthocyanidins degradation (Ananingsih et al., 2013; Cao et al., 2021; Le Bourvellec et al., 2013). The stability of phenolic compounds was influenced by matrix composition; the formulation containing both phenolic compound extract and cell wall (E-PPCWTs) had a higher phenolic compound content compared to the formulation containing only phenolic compounds (E-PPTs), suggesting a protective effect of fiber among phenolic compounds during processing. The analysis of proanthocyanidins and their structural characteristics such as their constitutive unit

composition and degree of polymerization indicated that extrusion promoted their depolymerization and in relation modification of their composition in most treatments. However, an increase in mDP was observed in E-PPCWTs due to cell wall selectivity towards proanthocyanidins high degree of polymerization (X. Liu et al., 2020; Lončarić et al., 2018).

Regarding dietary fiber, extrusion primarily affected pectin structure such as rhamnogalacturonan I (RG-I), while hemicelluloses and cellulose remained relatively stable. An increase in lignin content was observed in extruded strawberry by-products (E-SBP) and compared to SBP, due to their resistance to process but also may be due to their interactions with proanthocyanidins (Bindon et al., 2010; Dridi & Bordenave, 2021; C. M. G. C. Renard et al., 2017; H. Zhang et al., 2014). In matrices containing cell wall material (E-CWTs and E-PPCWTs), the process altered the solubility of glucomannans and galactans and promoted pectin demethylation, demonstrating that polysaccharide rearrangement was dependent on matrix composition. These structural changes were corroborated by FTIR analysis, which showed alterations in characteristic bands of fiber, starch, and phenolic compounds. SEM images further revealed that the presence of fiber and phenolic compounds influenced extrudate cohesion and porosity, confirming that these components affect not only composition but also the physical architecture of the matrix.

To integrate and analyze the relationships among the studied variables, principal component analyses (PCA) were performed in three complementary approaches (three PCA groups) (Table 2). First, the relationship between crystallinity, molar mass distribution, starch digestibility, glycemic index, and polyphenolic composition was explored in NEM, E-SBPTs, E-PPTs, and E-PPCWTs (PCA 1) (Fig. 2a), as all these matrices contain phenolic compound analysis; however, E-PPTs lacks incorporated fiber, which limits its relevance for cell wall-related analyses. Second, PCA (PCA2) was conducted using cell wall composition variables along with the same physico-chemical properties in NEM, E-SBPTs, and E-CWTs (Fig. 2b), as phenolic composition was not assessed in E-CWTs. Finally, a combined PCA (PCA 3) incorporating all variables, including polyphenol bioaccessibility, was performed on NEM, E-SBPTs, and E-PPCWTs, the only matrices for which the full set of data was available.

Table 2. Variables assessed in each matrix and sample groupings used for principal component analysis (PCA)

Matrix	Phenolic composition	Cell wall composition	Crystallinity	Molar mass distribution	Starch digestibility	Glycemic index	Phenolic compound bioaccessibility	PCA group
NEM	✓	✓	✓	✓	✓	✓	✓	All PCA groups
E-SBPTs	✓	✓	✓	✓	✓	✓	✓	All PCA groups
E-PPCWTs	✓	✓	✓	✓	✓	✓	✓	All PCA groups
E-PPTs	✓	✗	✓	✓	✓	✓	✗	PCA 1 only (phenolics-related)
E-CWTs	✗	✓	✓	✓	✓	✓	✗	PCA 2 only (cell wall-related)

NEM: Non-extruded mixture, **E-SBPTs**: Extruded snack, **E-PPCWTs**: Matrix containing phenolic compounds extract, cell wall material and tapioca starch, **E-PPTs**: Matrix containing phenolic compounds extract and tapioca starch, **E-CWTs**: Matrix containing cell wall and tapioca starch

The first two principal components in PCA1 (PC1 and PC2) accounted for 90% of the total variance, with PC1 explaining 70.6% and PC2 19.4% (Fig. 2a). The PCA results revealed significant associations between phenolic compounds (both monomers and polymers) and the structural and

functional parameters of starch. A strong positive correlation was observed between most individual phenolic compounds (e.g., quercetin, kaempferol-3-*O*-glucuronide, (+)-catechin), proanthocyanidins and starch molar mass parameters (Mn, Mp, Mw), resistant starch (RS) content, and crystallinity percentage. These findings suggest that the presence of phenolic compounds may influence the post-extrusion structural rearrangement of starch, promoting the formation of partially ordered regions or V-type complexes, thereby contributing to the retention of crystallinity in some matrices. This behavior may be attributed to the ability of phenolic compounds to interact with starch, hindering enzymatic access and stabilizing specific polymer regions, thus favoring the formation of ordered structures or V-type complexes (D. B. Amoako & Awika, 2016, 2019; Guo et al., 2019; Huo et al., 2025; Y. Li et al., 2023; Wu et al., 2024), as confirmed by X-ray diffraction analyses. These interactions contribute to the retention of partially crystalline structures, which are associated with higher resistance to digestion and lower glucose availability.

Additionally, the negative correlations observed between phenolic compounds and rapidly digestible starch (RDS) fractions, as well as with the estimated glycemic index (GI), support the notion that phenolic compounds could modulate starch structure and functionality not only by inhibiting enzymatic activity but also by promoting a less digestible structural reorganization. Collectively, these findings reflect the impact of phenolic compounds on starch conformation, their degree of organization, molar mass, and digestive functionality, highlighting their role as structural and functional modulators in extruded products enriched with plant by-products.

The first two principal components in PCA2 (PC1 and PC2) accounted for 77.5% of the total variance, with PC1 explaining 44% and PC2 33.5% (Fig. 2b). PCA correlations revealed significant interactions between structural components of cell wall, starch characteristics, and digestibility. A positive correlation was observed between structural sugars such as rhamnose (Rha), and cellulosic glucose (CGlc) with rapidly digestible starch (RDS), slow digestible starch (SDS) and glycemic index (GI). This relationship may suggest that these sugars may be associated with increased starch availability for enzymatic action, possibly due to reduced cell wall integrity, which could enhance starch digestion rate. This observation aligns with Rovalino-Córdova et al. (2018) who demonstrated that mechanical and enzymatic treatment of bean cell walls increased starch digestibility, although total digestibility remained similar compared to intact cell walls. Conversely, these parameters were negatively correlated with starch crystallinity and pectin's degree of methylation. This pattern suggests that samples with higher starch crystallinity exhibited lower starch digestibility and glycemic response, consistent with reduced enzymatic accessibility (J. Zou et al., 2020), and more highly methylated pectins could contribute to stabilizing crystalline starch structures, probably through reduced interactions with water and starch (K. Liu et al., 2025). The inverse relationship with rhamnose may reflect that matrices richer in RG-I pectins (indicated by higher rhamnose) tend to have more disorganized structures and lower starch crystallinity (Yin et al., 2021).

Similarly, variables such as slow digestible starch (SDS), arabinose (Ara), xylose (Xyl), galacturonic acid (GalA), and lignin appeared closely clustered, indicating a positive association that may suggest these components, mainly related to homogalacturonans (HG) and RG I (Soomro et al., 2024; Waldron & Faulds, 2007) may contribute to reduced starch digestion rate. Additionally, reports have highlighted the role of lignin in reducing starch digestibility (J. Zhu et al., 2022; W. Zou et al., 2021). Mn, Mp, Mw, and RS were tightly clustered, indicating a strong positive correlation among these structural starch parameters. This could suggest that samples with larger macromolecular structures resulted in greater resistant starch content, as seen in the non-extruded mixture (NEM). Taken together, these findings highlight how fiber composition, including structural sugars and lignin, as well as pectin's degree of methylation, could significantly modulate the physicochemical and functional properties of starch, directly affecting its molecular structure and digestive behavior.

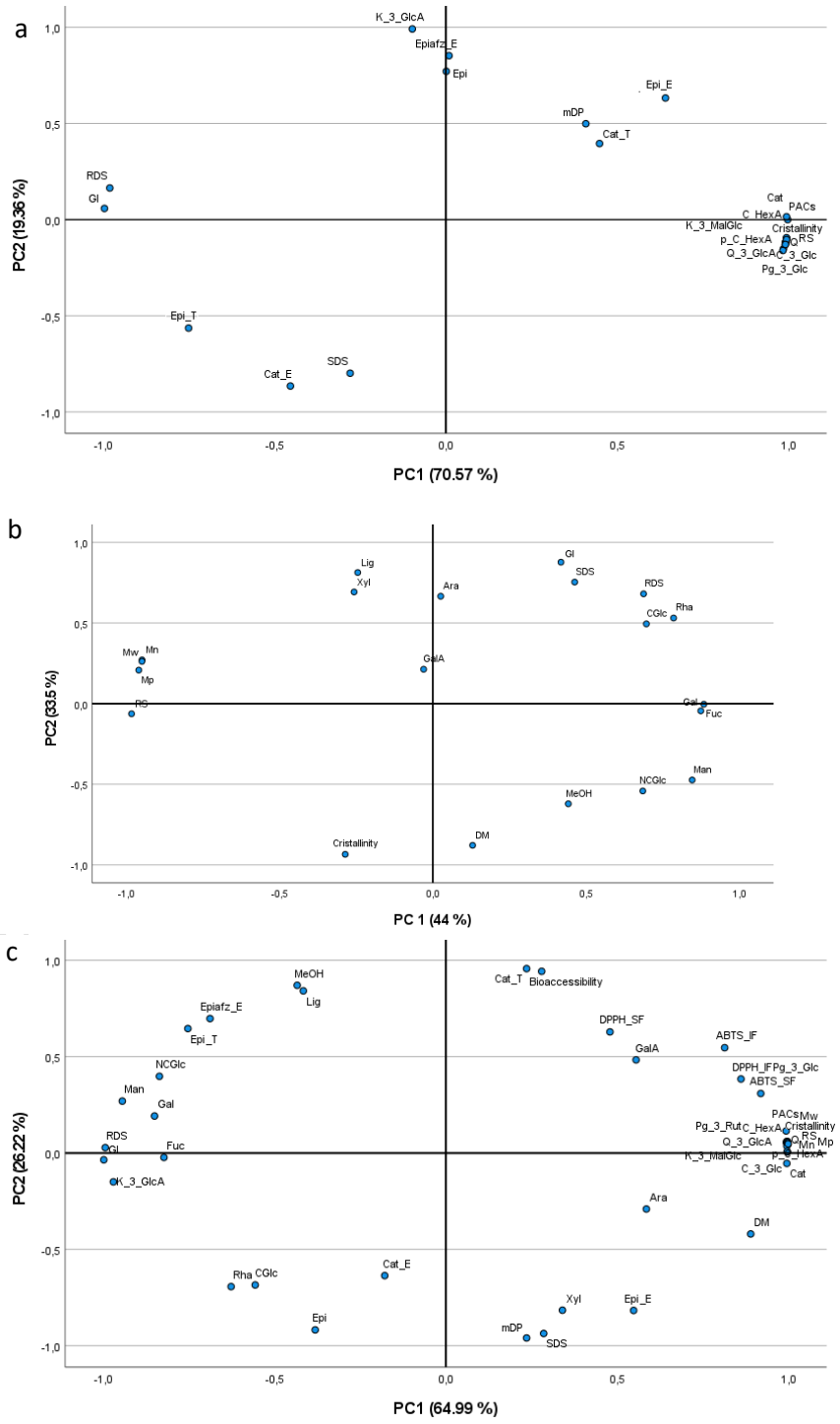

Fig. 2. PCA plot of comparison of (a) phenolic compound composition, starch molar mass distribution and digestibility and glycemic index in NEM, E-SBPTs, E-PPTs and E-PPCWTs, (b) cell wall composition, starch molar mass distribution and digestibility and glycemic index in NEM, E-SBPTs, E-CWTs and E-PPCWTs and (c) cell wall composition, polyphenolic composition, starch molar mass distribution and digestibility, glycemic index and bioaccesibility of phenolic compounds in NEM, E-SBPTs and E-PPCWTs.
PACs: Proanthocyanidins; p-C-HexA: p-coumaroylhexoside acid; Q: Quercetin; Q-3-GlcA: Quercetin-3-*O*-glucuronide; K-3-GlcA: Kaempferol-3-*O*-glucuronide; K-3-MalGlc: Kaempferol-*O*-3-malonylglucoside; Pg-3-Glc: Pelargonidin-3-O-glucoside; (+)-Cat: (+)-Catechin; (-)-Epi: (-)-Epitechin; mDP: Mean Degree of Polymerization; % (+)-Cat T: Percentage of (+)-catechin as terminal unit; % (-)-Epi T: Percentage of (-)-epicatechin as terminal unit; % (+)-Cat E: Percentage of catechin as extension unit; % (-)-Epi E: Percentage of (-)-epicatechin as extension unit; % Epiafz E: Percentage of epiafzelechin as extension unit Rha: rhamnose, Fuc: fucose, Ara: arabinose, Xyl:

xylose, Man: mannose, Gal: galactose, Glc: glucose, Gal A: galacturonic acid, CGlc: glucose from cellulose, NCGlc: glucose from non-cellulose, MeOH: methanol, DM: degree of methylation, Lig: lignin; Mw: Weight-average molar mass, Mn: number-average molar mass; Mp: peak molar mass; RDS: Rapidly digestible starch, SDS: Slowly digestible starch, RS: Resistant starch, GI: Estimated glycemic index; ABTS_IF: antioxidant activity (ABTS assay) of insoluble fraction after digestion; ABTS_SF: antioxidant activity (ABTS assay) of soluble fraction after digestion; DPPH_IF: ntioxidant activity (DPPH assay) of insoluble fraction after digestion; DPPH_SF: antioxidant activity (DPPH assay) of soluble fraction after digestion

The first two axes in PCA3 (PC1 and PC2) accounted for 91 % of the total variance, with PC1 and PC2 explaining 65 % and 26 % of the total variance, respectively (Fig. 2c). Extrusion promoted the release of phenolic compounds in formulations such as E-SBPTs, whereas in E-PPCWTs a significant proportion of these compounds remained associated with the indigestible matrix fraction due to interactions between cell walls and phenolic compounds mainly proanthocyanidins (Le Bourvellec & Renard, 2012; X. Liu et al., 2020). It is important to highlight that the two extruded matrices differed in their moisture content, which implies differences in specific mechanical energy input as well as in the cohesion and porosity of the extrudates, as observed for these samples. These factors may influence phenolic compounds bioaccessibility (Dehghan-Shoar et al., 2011; Hernández-Bautista et al., 2025). In the PCA plot, an opposite projection between bioaccessibility and SDS was observed, which may be attributed to the interactions formed during processing between starch and phenolic compounds, as well as with other macromolecules such as non-starch polysaccharides. These interactions could hinder the release of phenolic compounds from the food matrix, thereby reducing their bioaccessibility during digestion (Hernández-Bautista et al., 2025). The positive correlation observed between mDP and SDS suggests that proanthocyanidins with higher mDP may interact more effectively with starch molecules, forming complexes that alter starch structure. These interactions may increase the proportion of slowly digestible starch (SDS), as the resulting complexes are more resistant to enzymatic hydrolysis (Xu et al., 2021). On the other hand, the negative correlation between bioaccessibility and mDP indicates that more complex and higher-molecular-weight proanthocyanidins, due to their lower solubility, polymer structure and/or their association with fiber and starch (Bindon et al., 2010; Dridi & Bordenave, 2021; C. M. G. C. Renard et al., 2017; Xu et al., 2021; H. Zhang et al., 2014), may have limited ability to cross intestinal membranes, thereby reducing their bioaccessibility.

In general, as shown in Fig. 2c, the relationships observed in Fig. 2a, namely the positive correlation between starch crystallinity parameters, molar mass distribution, resistant starch (RS) content, and both monomer and polymer of phenolic compounds, were also maintained. Additionally, antioxidant activity clustered with these variables, suggesting a potential association between structural stability, phenolic compound composition, and antioxidant potential in the extruded matrices. Likewise, the relationships shown in Figure 2b between cell wall composition, RDS, and GI were also preserved. The integration of these variables provided a more comprehensive understanding of the interactions among matrix components and their impact on the nutritional quality of the extruded product. Overall, these findings offer valuable insights into how plant by-products and their structural fractions influence the nutritional and structural quality of extruded products. This knowledge is crucial for the development of functional snacks with improved structural stability, enhanced retention of bioactive compounds, and moderated glycemic response.

Altogether, the results of this research demonstrate that the composition and structure of plant by-products, as well as their mode of incorporation into extruded matrices, significantly modulated the structural and nutritional functionality of the final product. The combination of physicochemical, structural, and functional analyses enabled the identification of relationships among fiber and phenolic compound composition, starch structure, digestibility, and the bioaccessibility of bioactive compounds. Notably, the presence of phenolic compound and dietary fiber fractions affected starch reorganization during extrusion, altering its crystallinity, molar mass, and digestibility. Moreover, proanthocyanidins, meaning phenolic compound with a high complex structure tended to remain trapped within the indigestible matrix due to their specific interaction with cell wall, reducing their

bioaccessibility but potentially enhancing their functionality at the colonic level (Le Bourvellec et al., 2019). These findings underscore the importance of considering component interactions in the development of functional foods from agro-industrial by-products, with a focus on sustainability and functionality.

4 Conclusions

The findings of this research provide a comprehensive understanding of how the incorporation of strawberry by-products (SBP) and their structural fractions into starch-based extruded matrices influenced the nutritional and structural functionality of the final product. The application of extrusion as a processing technology enabled the integration of these by-products into functional snack formulations, while simultaneously modulating key physicochemical properties.

The optimization of extrusion parameters demonstrated the possibility of achieving desirable physical quality attributes, such as expansion and crispness, alongside improved retention of bioactive compounds, particularly phenolic compounds. Variations in processing conditions significantly affected the stability, extractability, and structural complexity of phenolic compounds, especially proanthocyanidins, whose degree of polymerization was strongly linked to specific mechanical energy input and matrix composition.

Furthermore, the use of isolated SBP fractions allowed for a differentiated analysis of their individual effects on starch structure, digestibility, and glycemic response. The presence of phenolic compounds was associated with higher starch crystallinity, larger molar mass polymers, and increased resistant starch content, suggesting the formation of starch-phenolic compound complexes that reduce enzymatic accessibility and slow glucose release. Conversely, the results related to cell wall composition suggest that its sugar profile and its degree of methylation of pectin may also play a significant role in modulating starch digestion dynamics, with specific pectic and hemicellulosic components contributing to a reduction in starch digestibility.

Notably, the inclusion of both fiber and phenolic compounds modified the structural organization of the matrix, as evidenced by increased cohesion and porosity. These structural changes were also associated with variations in phenolic compound bioaccessibility, as more complex proanthocyanidins tended to remain bound to the indigestible fraction, limiting their intestinal absorption but potentially enhancing their activity in the colon as cell wall may act as substrate for microbial bacteria allowing a better metabolization into small phenolic acid of proanthocyanidins.

Overall, this study highlighted the relevance of matrix composition and processing conditions in shaping the functional performance of extruded snacks enriched with agro-industrial by-products. The strategic incorporation of fiber and phenolic compounds not only improves the nutritional value of the final product but also contributes to the development of sustainable, health-oriented food systems and limited food waste. These insighted provide a foundation for the design of functional foods with targeted health benefits and enhanced use of food processing residues.

5 Recommendations and future perspectives

Based on the findings and interpretations presented in this study, the following recommendations and directions for future research are proposed to further advance the understanding of the physicochemical and nutritional properties of phenolic compound- and fiber-enriched extruded products formulated with strawberry by-products, and to support their technological development and potential industrial application. Particular emphasis should be placed on scaling up the process,

optimizing product formulation, and evaluating consumer acceptance to facilitate the transition from laboratory-scale findings to marketable functional snack products.

- Undertake pilot-scale extrusion studies to evaluate the scalability of the optimized formulations and processing conditions, and to assess their technological feasibility for commercial snack production. These studies should consider process robustness, energy efficiency, cost-effectiveness, and potential adjustments needed for industrial implementation.

- Integrate techno-economic analyses to assess the feasibility of industrial-scale production, considering raw material sourcing, energy consumption, production yield, and estimated market value. These evaluations will be key for guiding investment decisions and ensuring economic viability.

- Include sensory evaluation and consumer acceptance studies, involving both descriptive and hedonic analyses, to assess the palatability, texture, and overall acceptability of the extruded products. These evaluations should also explore the possible incorporation of minor ingredients or additives (e.g., natural flavors, sweeteners, texturizers) to optimize sensory attributes, particularly flavor, for market-ready products.

- Perform shelf-life and storage stability assessments, especially regarding the retention of phenolic compounds and the maintenance of textural and sensory attributes over time, to support the development of market-ready functional snacks.

- Conducte *in vivo* studies to validate the *in vitro* findings related to starch digestibility, glycemic index, and phenolic compounds bioaccessibility. Animal models or human trials could provide a more accurate assessment of the physiological impact and offer a better understanding of the bioavailability of bioactive compounds.

- Investigate the colonic phase of digestion, particularly to determine the fate of phenolic compounds and dietary fiber that resist upper gastrointestinal digestion. This would clarify their potential prebiotic and antioxidant effects in the large intestine.

- Perform comparative analyses using strawberry by-products under standardized moisture conditions, matching those of composite matrices, to better isolate the effect of formulation from that of processing variables such as specific mechanical energy.

- Assess the bioaccessibility of individual phenolic compounds, rather than total phenolic compounds, to identify which specific molecules are more readily released and potentially absorbed during digestion.

- Study interactions with other functional ingredients, such as plant proteins or lipids, which could influence the formation of phenolic compounds macronutrient complexes and impact both functionality and bioavailability.

- Explore the potential residual presence and traceability of pesticide compounds throughout the extrusion process, given the intensive use of pesticides in strawberry production. Evaluating how thermal processing affects pesticide degradation or persistence in the final product is essential to ensure food safety and consumer confidence in formulations based on fruit by-products.

These recommendations aim to guide future research toward a more comprehensive understanding of the physicochemical, nutritional, sensory, and functional properties of phenolic compound- and fiber-

enriched extruded products. This knowledge will be critical for the rational design of health-promoting, consumer-accepted snacks and for the sustainable valorization of agro-industrial by-products through scalable industrial processes.

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