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Ulliana Marques Sampaio

# Physical modification of starches and young bamboo culm flour using conventional and emerging technologies: extrusion cooking and non-thermal plasma

Modificação física de amidos e farinha de colmo jovem de bambu através de tecnologias convencional e emergente: extrusão termoplástica e plasma não-térmico

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Tese apresentada à Faculdade de Engenharia de Alimentos como parte dos requisitos exigidos para a obtenção do título de Doutora em Tecnologia de Alimentos

Thesis presented to the School of Food Engineering of the University of Campinas in partial fulfillment of the requirements for the degree of Doctor in Food Technology

Orientadora/Advisor: Prof[a]. Dr[a]. Maria Teresa Pedrosa Silva Clerici

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*"Eu tenho muitas coisas para conquistar e não posso ficar aí parado"*

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**RESUMO**

A demanda contínua por um sistema alimentar saudável e sustentável direciona a indústria alimentícia a usar matérias-primas locais, novas fontes de nutrientes e abordagens alternativas para tecnologias convencionais e emergentes a fim de desenvolver novos produtos e/ou modificar as propriedades tecnológicas das matérias-primas. O objetivo deste estudo foi avaliar e caracterizar as modificações nas misturas de farinha de arroz e farinha do colmo jovem de bambu e nas fontes de amido (milho, mandioca e batata) após os tratamentos de extrusão termoplástica (I) e plasma não-térmico (II), respectivamente. Para o método convencional (I), a farinha do colmo jovem de bambu (FCJB) foi obtida de um colmo de 4 meses de idade e foi avaliada quanto à sua composição centesimal, atividade prebiótica e características tecnológicas, como capacidade de inchamento, pH, acidez titulável, cor, propriedades de pasta e outras. A extrusão termoplástica foi realizada em uma extrusora dupla rosca com diferentes proporções de Farinha de arroz: FCJB (100:0, 95:5, 90:10 e 85:15). As amostras foram avaliadas quanto às propriedades de pasta, cor, índices de absorção e solubilidade de água, entre outros. As análises foram realizadas em triplicata e os resultados foram avaliados por análise de variância (ANOVA) em um nível de significância de 5%, usando o teste Scott-Knott para comparação entre as médias. A análise de componentes principais foi aplicada para comparar as amostras em relação aos tratamentos realizados e às características tecnológicas adquiridas após o processo. A FCJB apresentou um teor expressivo de fibras (>80%) e xilana e celobiose em sua composição química, que provavelmente foram responsáveis por promover o crescimento bifidogênico antes e depois da extrusão. A redução da expansão, o aumento da dureza e da densidade dos extrusados foram observados com o aumento da concentração de FCJB. Além disso, foi observado um aumento no torque e na energia mecânica específica, o que levou a uma alta conversão de amido em comparação com o controle. De modo geral, a FCJB mostrou potencial para ser uma nova fonte sustentável rica em fibras em produtos extrudados com atividade prebiótica. Para o método emergente (II), as diferentes fontes de amido (milho, mandioca e batata) foram tratadas usando um sistema contínuo de plasma de argônio não-térmico (PNT) em duplicata. A microestrutura, o teor de umidade, o pH, o amido danificado, a distribuição do tamanho das partículas, a força do gel e as propriedades de pasta foram determinados antes e depois do tratamento. O teste t ($p<0,05$) foi aplicado para comparar as médias. Em geral, o PNT, por meio do tratamento superficial indireto, reduziu o teor de umidade das amostras, aumentou o teor de amido danificado, a força do gel e criou fissuras na superfície dos grânulos de amido. De acordo com a fonte de amido, a ligação cruzada ou a despolimerização foram as principais reações que ocorreram durante o tratamento. Portanto, o

sistema PNT contínuo demonstrou ser uma tecnologia verde promissora para modificar o amido e para ser ampliada em escala industrial.
**Palavras-chave:** polissacarídeos, amido, farinha de arroz, fibras dietéticas, bambu, propriedades tecnológicas, prebióticos

**ABSTRACT**

A continuous demand for a healthy and sustainable food system drives the food industry to use local raw materials, new sources of nutrients, and to use alternative approaches for conventional and emergent technologies to develop new products and/or modify the technological properties of raw materials. The aim of this study was to evaluate and characterize the modifications in rice-young bamboo culm flour blends and starch sources (corn, cassava, and potato) after extrusion cooking (I) and non-thermal plasma (II) treatments, respectively. For the conventional method (I), the young bamboo culm flour (YBCF) was obtained from a 4-month-old culm and it was evaluated for its proximate composition, prebiotic activity, and technological characteristics, such as swelling, pH, titratable acidity, color, pasting properties, and others. The extrusion cooking was conducted in a twin screw extruder with different Rice flour: YBCF ratios (100:0, 95:5, 90:10, and 85:15). Samples were assessed regarding pasting properties, color, water absorption and solubility indexes, among others. The analyses were performed in triplicate and the results were evaluated by variance analysis (ANOVA) at a 5% significance level, using the Scott-Knott test for comparison between means. The Principal Component Analysis was applied to compare the samples regarding the treatments performed and the technological characteristics acquired after the process. YBCF presented expressive fiber content (>80%) and xylan and cellobiose in its chemical composition, which were probably responsible for promoting bifidogenic growth before and after extrusion cooking. Reduction of expansion, increase in hardness and density of extrudates were observed with the increase of YBCF concentration. Also, increase in torque and specific mechanical energy were observed, which led to a high starch conversion compared to the control. Overall, YBCF showed potential to be a new sustainable fiber-rich source in extruded products with prebiotic activity. For the emergent method (II), the different starch sources (corn, cassava, and potato) were treated using a continuous non-thermal argon plasma system (NTP) in duplicate. Microstructure, moisture content, pH, damaged starch, particle size distribution, gel strength, and pasting properties were determined before and after treatment. T-test ($p<0.05$) was applied to compare the means. Overall, NTP through the indirect superficial treatment, reduced the moisture content of the samples, increased damaged starch and gel strength, and created fissures on the granule surface. According to the starch source, cross-linking or depolymerization were the main reactions that occurred during treatment. Hence, the continuous NTP system showed to be a promising green technology to modify starch and to be scaled-up.

**Keywords:** polysaccharides, starch, rice flour, dietary fibers, bamboo, technological properties, prebiotic

**SUMÁRIO**

# CHAPTER I

## INTRODUCTION

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This chapter presents a general introduction to the subject covered in the thesis, the research objectives and a brief explanation of the organization of the document

# 1. INTRODUCTION

Food polysaccharides comprises starch and non-starch materials, which present distinguish properties (e.g., water solubility, viscosity, gelling behavior, digestibility) according to their molecule structure and degree of polymerization (ELIASSON and GUDMUNDSSON, 2006). Starch is found in cereals (50-80%), legumes (25-50%), and tubers (60-95%), providing the main food energy source for the population. It is used in a wide range of food products as an ingredient or as a food additive. Independent of the quantity and application, the main uses of native starch follow granule disruption to achieve the desired characteristics in products (BULEON and COLONNA, 2007).

Non-starch materials, such as fibers, constitute the plant cell walls, corresponding in this way to one of the most abundant biopolymers in the world (SERNA-SALVADIR and AYALA SOTO, 2020). In general, dietary fibers are used to control the technological properties and/or to improve the nutritional aspect of products. Over the years, many fiber-rich products have been launched into the market intending to prevent overweight, obesity, metabolic syndrome, and other diseases. Although it is possible to find more fiber containing products in the market, fiber intake by the world population is well below the recommendation of 25 g/day (FDA, 2023; ESFA, 2010) in most of the countries (SALMAS, DEVRIES and PLANK, 2017). Fibers are usually added in low quantities in a formulation due to their filler effect and sensory challenges. However, researchers are continuously developing methods to increase the fiber content in the products without compromising their characteristics.

The diversity of food products continues to expand as a consequence of high expectations of quality and consumers' awareness regarding food processing since the raw materials sources, sustainable processes, and healthy benefits.

Among the cereals, rice (*Oryza sativa* L.) is one of the most important food crops cultivated in the world and is mainly consumed in the form of processed and polished grains (FAO, 2021). The latter processing generates by-products such as broken rice, which has been transformed into flour for different applications (e.g., gluten-free products, soups, porridges and breakfast cereals), as its mild flavor characteristics, white colour, high digestibility, and hypoallergenicity, makes it a raw material of easy application (SOMPONG et al., 2011). Providing different destinations to by-products is one of the main ways to build a full cycle food system.

As a raw material, bamboo has an important economic, social, and environmental relevance (BRONDANI et al., 2017), with wide application in various sectors, including the food area. Bamboo is a grass belonging to the family *Poaceae*, subfamily *Bambusoideae*, which grows naturally in tropical, subtropical, and temperate regions, except in Europe, presenting more than 1250 species (LIESE and KOHL, 2015). In Brazil, there are approximately 18 million hectares of cultivated area located in the southwestern Amazon, corresponding to the largest native reserve in the world (DANTAS et al., 2005). The flour obtained from the young bamboo culm was recently characterized by the research team of Prof. Maria Teresa Clerici (State University of Campinas) in a pioneering study in Brazil as a new sustainable fiber-rich ingredient. The flour was successfully applied in pasta and cookies with a good sensory acceptance.

Young bamboo culm may be an alternative for the insertion of a new food ingredient in different formulations with industrial-scale production capacity (FELISBERTO, BERALDO, and CLERICI, 2015). The average composition of young bamboo culm consists of 4.66% moisture, 1.87% protein, 0.48% fat, 1.89% ash, 91.1% total carbohydrates, 66.6% total insoluble dietary fiber and 16.2% total starch, on a dry basis (FELISBERTO et al., 2017).

Starch and dietary fibers can be modified as isolated ingredients or even in a complex formulation. As stated before, starch granules need to be disrupted first to be used, while, even though, the insoluble dietary fibers have a nutritional importance, it is recommended to convert in soluble dietary fibers to improve the bioavailability and maximize their benefits (SERNA-SALVADIR and AYALA SOTO, 2020). Several methods can be applied to modify polysaccharides' structures, either by chemical, enzymatic, microbiological, or physical treatments. Thermal and nonthermal physical technologies have been used as an alternative to chemical processes and for having eco-friendly approach. These technologies have different principles and applications, and therefore, their use should be determined according to their advantages and aims. Thermal treatments can be useful, for example, to convert raw materials into different states of matter, providing specific textures to the finished product, while nonthermal treatments can be used, for example, to improve quality of the product by preserving nutrients (TAPPI, TYLEWICZ, and ROSA, 2020). Among the existing physical methods are extrusion cooking and non-thermal plasma.

Extrusion cooking is an established technology of great versatility, used for more than 50 years, which is considered a bioreactor that operates at high temperatures and in a short time. This process has become widespread because of its continuous operation, high productivity, low cost, and no waste generation. Extrusion cooking also aids in modifying the structure of compounds, improves solubility, starch and protein digestibility, increases soluble fiber content, and generates different types of ready-to-eat products, such as breakfast cereals and snacks (ALAM et al., 2016).

Plasma is defined as the fourth state of matter, formed by an ionized gas containing reactive species such as electrons, ions, free radicals, and a higher number of non-ionized neutral molecules. The use of plasma aiming to modify food properties is relatively new but promising, as it is an efficient, green procedure that uses low temperatures and does not produce waste (MISRA, SCHLUTER, and CULLEN, 2016).

These data were the starting points for the present project, which instigated the study of conventional and emerging technologies to modify starch and fiber-rich raw materials.

# 2. OBJETIVE

## 2.1 GENERAL OBJECTIVE

To evaluate and characterize the modifications of extrusion cooking on young bamboo culm flour (*Dendrocalamus latiflorus*) and rice flour, and of non-thermal plasma on commercial isolated starches, in order to leverage the knowledge about the technological and functional aspects of these raw materials in an already well-established industrial process and another emerging process.

## 2.2 SPECIFIC OBJECTIVES

- To present a literature review on the objects of study that involve the project, such as starch, physical processes of starch modification, and application of bamboo fiber
- To produce the flour from the young bamboo culm (*Dendorcalamus latiflorus*) and to characterize it regarding its physicochemical, technological, and prebiotic properties
- Evaluate the use of the young bamboo culm as an ingredient in rice flour-based extrudates and determine its technological, structural, and prebiotic properties after processing
- Apply non-thermal plasma in corn, cassava, and potato starches and evaluate the morphological and technological modifications resulting from the continuous process

# 3. ORGANIZATION

This document was organized into chapters, where presents articles published and/or submitted to journals. The arrangement of the chapters does not necessarily correspond to the chronological order in which the research was carried out but corresponds to the logic used to define the project.

**Chapter I - Introduction** presents an overview and justifications for the investment in this research. **Chapter II - Basic Principles: Composition and Properties**, **Chapter III - Identification and analysis of starch**, **Chapter IV - Physical modifications of starch**, and **Chapter V - Bamboo fiber application for food and feed** correspond to the theoretical foundation part, with details on starch, physical modification processes, and the use of bamboo fiber. Here, the intention was to perform a literature review to understand and define the raw materials and techniques to be used in the project, selecting the extrusion cooking and the non-thermal plasma.

Understanding the importance of complex carbohydrates, starch and fiber, in our diet and continuing the research initiated in an unprecedented way at Unicamp by Prof. Maria Teresa, Prof. Beraldo and Prof. Mária Felisberto, **Chapter VI – Technological and prebiotic aspects of young bamboo culm flour (*Dendrocalamus latiflorus*) combined with rice flour to produce healthy extruded products** refers to the young bamboo culm flour processing, its respective chemical, technological, and prebiotic activity characterization, as well as its use in the extrusion cooking process in rice-based products. Part of this article was developed with the help of students Gabriela Ribeiro (Profis) and Gabriela de Oliveira (SAE).

**Chapter VII - Continuous microwave non-thermal plasma system to modify starch properties** corresponds to the data obtained in partnership with the Institute of Food Technology (ITAL) that resulted in a patent. Commercial corn, cassava, and potato starches were modified using this technique and evaluated for their technological properties.

**Chapter VIII - General Discussion** presents a summary of the most important data obtained during the execution of the project. And finally, **Chapter IX - General Conclusion** indicates the potential use of young bamboo culm flour as a prebiotic ingredient in extruded products and the potential of non-thermal plasma technology as a method to be leveraged to industrial scale.

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# CHAPTER II

## BASIC PRINCIPLES: COMPOSITION AND PROPERTIES

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This chapter presents a review of the main aspects of starches

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# Basic principles: Composition and Properties

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## 1.1 Introduction

Until recently, starch was considered as the main energy source for human food; however, its nutritional role has expanded, once it also represents a source of dietary fiber. The evolution in the genetic, chemical and technological fields has led to the production of starches with changes in digestibility and resistant to the enzymes of the digestive system (Fuentes-Zaragoza et al., 2010; Jane, 2004; Goni et al., 1997; Eerlingen and Delcour, 1995).

Today, there is an increasing demand for healthier products, without changing the sensory properties of the processed products, and a variety of modified starches is available to meet these criteria.

Starch can be produced by conventional or family farming and is a source of subsistence for many farmers. The major starch sources are cereals, such as corn, and tubers and roots, such as potatoes and cassava. Starches are used for different purposes in food, including the production of glucose and/or fructose syrups, or as thickening agents and fat substitutes, among others. In other areas, starch has been increasingly used in several industries, such as paper, textiles, pharmaceuticals, cosmetics, packaging, steel, among others, as stated by Whistler and Paschall (1965), Wurzburg (1989), Eliasson (2004), BeMiller and Whistler (2009), and Bertolin (2010).

This great improvement may completely change the concept that starchy products are among the main causes of obesity, type II diabetes, and other chronic non-communicable diseases (Escott-Stump and Mahan, 2013). In the future, starch may be consumed as a source for slow release of glucose, which is essential for the brain, retina, labyrinth, and nervous system. With optimized starch use, there will be no excess glucose in the blood vessels, thus, reducing fatty acids conversion, which, when in abundance, is harmful to the human body.

With the great variety of modified starches and new modification techniques, in the future, an engineering aimed solely at obtaining a specific starch for each human needs may be a reality. Today, the modification processes have been performed according to the purpose, for example the resistant type IV starches, which are modified chemically to resist the enzymes of the digestive system and innumerable processing conditions, including high temperatures, high pressure, acidic pH, among other aggressive conditions, especially for those foods subjected to sterilization.

This chapter will address the basic principles to familiarize the reader and facilitate the understanding of the following chapters.

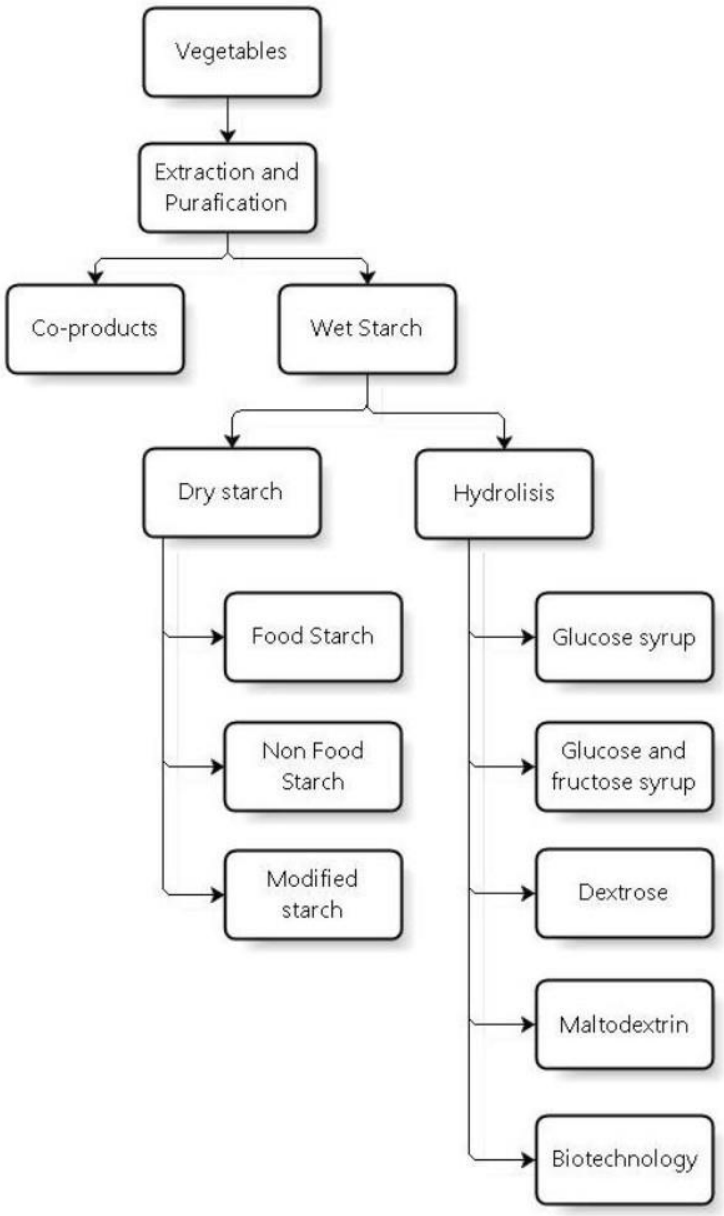


**Figure 1.1** Production of starch and its various applications

## 1.2 Obtaining starch

Starch and similar molecules can be found in plants, bacteria, and algae. In animals, glycogen, formed by glucose molecules, is present in muscle and liver in small amounts for maintaining the essential activities in the body. In bacteria and algae, studies have found new sources of obtaining carbohydrates for human consumption (Eliasson, 2004; BeMiller and Whistler, 2009). Economically, the starch of plant origin is the one with major importance for the food industry.

In plants, starch is stored inside plant cells in the form of energy, found in both chloroplasts (chlorophyll-containing plastid) as a transitory starch, being synthesized during photosynthesis and consumed at night by the plant, and amyloplasts, in which the synthesis and degradation occurs at separate times, with accumulation of starch at high concentrations in the reproductive structures (grains), vegetative structures (tubers, stems), fruits (banana, wolf fruit, and others) and roots (cassava, taro), to be used in other phases (germination, budding, fruit ripening and others) (Eliasson, 2004; BeMiller and Whistler, 2009).

For the extraction and industrial processes, the starch stored in the amyloplasts has the greatest economic viability.

Starch extraction methods are well established for cereal and roots commercial, but should be adapted to the new sources of starch, such as leaves, fruits, rhizomes, stems, legumes, nuts, plant shoots among others (Eliasson,2004). Although these new sources have technological properties different from those already commercialized, many are still in the research phase, without studies of economic viability for industrial use.

The selection of the extraction method depends on many factors, including origin, location in the plant, presence of nutrients, perishability after harvest, etc, which can vary from physical processes to the use of chemical reagents and enzymes. The principle of separation is based on the fact that starch is insoluble in cold water and has a proximate density of 1.5 $g \times mL^{-1}$, which ensures its decantation when mixed with water (Eliasson, 2004). Therefore, processing plants for starch extraction and purification use great volumes of water and usually generate co-products and waste that require biological treatments. Further details will be discussed in Chapter 4.

In some cases, starch is the main extraction product, as in the case of corn, cassava, and potatoes, but it is also considered a co-product, for example in the extraction of vital wheat gluten, which generates great amounts of starch rather than gluten, but with lower added value.

The major goal of the extraction process is obtaining starch with a high degree of purity, while other nutrients (minerals, proteins, and lipids) should be at concentrations below $1.5g \times 100g^{-1}$. When starch meets the purity requirements, it is ready to be used in the native state or modified for use in food processing industries (Tester et al., 2004).

When starch is impure, the modifications won't be as effective, due to the fact that other nutrients, such as proteins and lipids, are more reactive to chemical agents and physical processes than the starch itself, and impair the activity of amylolytic enzymes and reagents. Thus, in such cases, the modification technique requires an in-depth study to determine whether the reaction occurred with starch and/or another nutrient and whether the reaction led to changes in the protein cross-linking, the formation of toxic compounds, formation of free radicals, Maillard reaction, among others.

After extraction and purification, starch can be visually characterized as a fine white powder, insoluble in cold water, alcohol, ether, and other solvents, and soluble in dimethylsulfoxide (DMSO). One way of identifying the presence of starch in food products is by the use of indicator dye, glycerin or Lugol's iodine, which will color in the blue-red range (Brasil, 2010).

The identification of the source of extracted starch can be made by optical microscopy, since the starch granule presents distinct characteristics, such as birefringence under polarized light and presence of Maltese cross, among others (Brasil, 2010).

When viewed under an optical microscope, starch appears in the form of granules of varying sizes, shapes, and stratifications. The starch granule consists of hilum (point of origin of the ring structure) and lamellae or striae (light and dark areas). The starch characteristics including the spherical, ovoid, polyhedral, periformic, ellipsoid forms and size can be detected in the microscopic analysis, and a central point known as hilum is observed in polarized light, which can be punctuated, starry, linear, etc, followed by branching, forming the Maltese cross (Jane, 2004; Jane, 2006). The form, granule size, and the type of hilum or Maltese cross are good parameters to identify the most common starches from cereals, roots, and tubers, as can be seen in Table 1.1. Another important factor is that starch extracted can present different types of distribution (unimodal, bimodal, or trimodal). Table 1.1 shows an example of the bimodal distribution of the granule size in wheat, which may have different technological properties. When separated and analyzed alone, B-starches (15-20%) have 2-15 μm diameter, while A-starch granules (80-85%) have 20-35 μm, as reported by Jane et al., 1997.

**Table 1.1** Characteristics of some starches*

| Starch | Origin | Size (µm) | Morphology | Distribution |
|---|---|---|---|---|
| Maize | Cereal | 5-30 | Spherical/polyhedral with porous surface | Unimodal |
| Wheat | Cereal | 20-35 (A)<br>2-10 (B) | Lenticular (A-type)<br>Spherical (B-type) | Bimodal |
| Rice | Cereal | 3-8 | Polyhedrall | Unimodal |
| Potato | Tuber | 5-100 | Lenticular | Unimodal |
| Cassava | Root | 4-35 | Spherical/lenticular | Unimodal |
| Barley | Cereal | 2.3 (A)<br>7.5 (B)<br>20 (C) | Lenticular/Spherical | Trimodal |
| Waxy barley | Cereal | 10.2-13.6 (A)<br>2.1-3.1 (B) | Lenticular/Spherical | Bimodal |
| Durum | Cereal | 13.9 (A)<br>4.06 (B) | Lenticular/Spherical | Bimodal |

*Adapted from Tester and Karkalas (2002), Tester et al. (2004), BeMiller and Whistler (2009), Dendy and Dobraszczyk (2001)

The formation of starch granules starts at the hilum and can be of three distinct forms within the plastid, as follows:

- Simple: granule formed within the plastid, for example, potatoes, wheat, rye etc.
- Composite: more than one granule inside the plastid, for example, cassava, hazelnut, sweet potato, sago.
- Semi-composite: two or more simple granules, joined by deposition of layers (Zobel and Stephen, 1996)

### 1.3 Chemical composition of starch

As a fine powder, the cereal starch contains about 10-12% moisture, while the tuber starch has 14-15%. Maintaining this moisture value and using packaging to protect against variations in relative humidity are important factors for a longer storage of starch (years). The other nutrients should be in concentrations below 1.5-2.0% in starch, as can be seen in Table 1.2. They occur in starches as follows:

**Table 1.2** Chemical composition of some starches, in percentage*

| Starch | Carbohydrate | Lipid | Protein | Ash | Phosphor |
|---|---|---|---|---|---|
| Corn | | | | | |
| Regular | 98.9 | 0.6 | 0.35 | 0.1 | 0.015 |
| Waxy | 99.5 | 0.2 | 0.25 | 0.07 | 0.007 |
| High Amylose | 99.4 -98.7 | 0.4-1.1 | - | 0.2 | 0.070 |
| Wheat | 98.6 | 0.8 | 0.4 | 0.2 | 0.060 |
| Rice | 98.3 | 0.8 | 0.4 | 0.5 | 0.010 |
| Potato | 99.5 | 0.05 | 0.06 | 0.4 | 0.080 |
| Cassava | 99.6 | 0.1 | 0.1 | 0.2 | 0.010 |

*Adapted from BeMiller and Whistler (2009), Tester and Karkalas (2004), Eliasson and Gudmundsson (2006)

- Lipids in cereal starch granules may be internal, such as lysophospholipids and free fatty acids (FFA) and externally as triglycerides, phospholipids, and FFAs, which may be from the amyloplast membrane; in other starches as oat starch, the lipid may be complexed to amylose.
- Proteins (max 0.6%) may also be on the surface or internally as membrane proteins and enzymes responsible for the synthesis and degradation of starch
- Mineral salts are mainly represented by phosphorus, which may be phospholipids, monoester, and inorganic phosphate. Native starches generally contain small phosphorus levels (0.1%). In the case of rootstocks, phosphorus is covalently bound to starch (Hogde et al., 1948), whereas in cereal starch, this mineral occurs mainly as phospholipids (Lim et al., 1994). As an example, the amount of phosphate groups in potato starch range from 1 phosphate group per 200 to 400 glucose units, while the other starches have lower values (Swinkels, 1985).

Pure starch is formed by glucose monomers linked by glycosidic bonds, being a homopolysaccharide. The way the glucose molecules are joined can be:

- by α-1-4 glycosidic bonds, with few α-1-6 branching points, forming the amylose molecule, which is essentially linear, with a molecular mass between $10^5$ and $10^6$

Daltons. Amylose has great ability to form complexes with lipids due to its helical structure, and it may be free or complexed with lipids.

- by α-1-4 glycosidic bonds, with a large number of α-1-6 bonds, forming a branched structure, so-called amylopectin, with a molecular weight from $10^7$ to $10^9$ Daltons. Amylopectin unit chains are relatively short when compared to amylose molecules with a broad distribution profile. They are typically 18–25 units long on average.
- by glycosidic bonds forming a small amount of intermediate material, from 5 to 7%, where the glucose molecules are in short or long branches, exhibiting different properties from amylose and amylopectin (Eliasson, 2004; BeMiller and Whistler, 2009; Bello-Perez et al, 2010).

Amylose and amylopectin are radially arranged in the starch granule with their terminal reducing groups oriented towards the center or hilum, in which the deposition of these polymers takes place (Swinkels, 1985). These molecules are linked by hydrogen bonding, which maintains the integrity of the granule and establishes the physical strength and solubility, presenting differences in solubility and paste formation (gel), as can be seen in Table 1.3.

**Table 1.3** Amylose and amylopectin characteristics*

| | **Amylose** | **Amylopectin** |
|---|---|---|
| Mass Molecular | $10^5$-$10^6$ | $10^7$-$10^8$ |
| Degree of Polymerization | 1500-6000 | $3x10^5$- $3x10^6$ |
| Dilute solutions | Unstable | Stable |
| Solubility | Variable | Soluble |
| Complexion formation | Favorable | Unfavorable |
| Gel | Stiff, irreversible | Soft, reversible |
| Films | Coherent | - |
| Iodine color | Blue | Red-Purple |
| Iodine afinitty | 19-20% | 1% |
| Difraction | Crystalline | Amorphous |
| Digestibility (β-amylase) | 100% | 60% |

*Adapted from Ciacco and Cruz (1982) and Zobel (1988)

When the starch granule is analyzed by X-ray diffraction, amorphous regions and semi-crystalline regions are observed, which are characteristic of amylose and amylopectin, respectively. The elucidation of how these molecules are organized in the granule allowed to advance in the studies on starch modifications, due to the difference in the reactivity of individual starch granules (Donald et al., 2001).

The amylose and amylopectin contents vary according to the origin of the granule (Table 1.4). With the genetic advances in the area, new modified starches can be obtained with the introduction of waxy genes, wx (recessive) and Wx (dominant) and ae genes (high amylose) into the plant. In this way, many plants present conventional starches and also waxy starches (above 85% amylopectin) and high amylose content (above 40% amylose). While the waxy starch is brittle, the high-amylose starch resists the activity of amylases and is difficult to gelatinize (Tester et al., 2004; Wurburg, 1989; BeMiller and Whistler, 2009).

**Table 1.4** Amylose and amylopectin content of starches*

| Starch | Amylose (%) | Amylopectin (%) | Examples |
|---|---|---|---|
| Normal | 20-35<br>18-25 | 70-80<br>75-82 | Cereals<br>Tubers |
| Waxy | <8% | 92-100 | Waxy corn, waxy potato, waxy rice |
| High amylose | ≥50 | ≤50 | High amylose corn (resistant starch) |

*BeMiller and Whistler (2009)

## 1.4 Properties

Raw starch is insoluble in water, has no thickening properties and tends to settle while in solution. To exhibit the properties of the thickening agent, as a stabilizer, moisture retainer, gel former, binder, it must undergo modification processes prior to use or next to food processing (Eliasson, 2004). Table 1.5 shows some starch applications.

To select a starch for addition in food formulations, its nutritional function as an energy source or dietary fiber and/or aesthetic function (texture, flavor retention, appearance) should be taken into account. In this way, knowing the process of food production and the intrinsic factors is very important for selection of starch, for example, type of processing, type of product, conservation method, pH, moisture content, shelf life, ingredients that can interact with starch, among other factors.

When starch plays a nutritional function, it may be in the following forms (Singh et al., 2010; Fuentes-Zaragoza et al., 2010):

- Inaccessible to digestion, when raw, acting as resistant starch (RS) or when modified by a physical, chemical, or genetic process, leading to the formation of starch with high amylose content, and corresponds to undigested starch within 120 min of digestion, which is the total starch minus the amount of glucose released; classified into 5 types, as demonstrated in the Chapter 8.
- Slowly digestible starch (SDS), when retrograded or in the presence of fibers; corresponds a high amount of glucose released between 20 and 120 min of *in vitro* digestion; and
- Easily digested, being a source of energy when cooked, suffering amylase attack; corresponds a high amount of glucose released in the first 20 minutes of digestion.

**Table 1.5** Application of starch in foods and package for foods

| Product | Starch function | Some articles |
|---|---|---|
| Mayonnaise,<br>Salad dressing | Emulsify/thickener<br>Stability, fat replacer | Alimi et al. ( 2013)<br>Bortnowska et al. (2014) |
| White sauces<br>white sauce | Freezing and thermal stability<br>Gluten free | Sanz et al. (2016)<br>Bortnowska et al. (2016) |
| Dairy products | Stabilizers | Tasneem e al. (2014) |
| Ice cream | Fat and sugar replacer<br>Freeze-thaw stability | Šubarić et al. (2010)<br>Liu et al. (2015) |
| Candy of black raspberries | Controlled release of phytochemicals | Gu et al. (2015) |
| Bakery products | Fat replacer in muffins and cookies<br>Fat replacer in cake and cookies | Lee and Puligundla (2016)<br>Serinyel and Öztürk (2016) |
| Bread | Fat replacer | Balic et al. (2017) |
| Glúten free bakery products | Gluten substitute | Mancebo et al. (2016)<br>Martinez and Gomez (2017) |
| Yogurts, Cream cheese | Thickener | Klemaszewski (2011) |
| Package | Biocomposite, sustentable package | Roy et al. (2011)<br>Rodriguez-Gonzalez et al. (2003) |
| Films | Biodegradable, Edible | Durango et al. (2006)<br>Mali et al. (2004) |

The greater nutritional function of starch is as a source of energy since commercial resistant starches are still costly.

The replacement of the usual fibers, such as celluloses and hemicelluloses, by resistant starch in food is due to RS does not affect color, odor, and flavor of the food product, besides contributing to the texture and resisting the thermal processing for a prolonged time, depending on the type of starch used (Fuentes-Zaragoza et al., 2010).

During gelatinization, raw starch undergoes changes, allowing the hydrolysis by amylolytic enzymes and providing pasting properties, with thickening function in the food product. The production of pre-gelatinized starches, with good solubility, thickening properties, and cold paste formation has been one of the main starch modifications, being widely used in instant powders as thinning agents (Powell, 1965).

### 1.4.1 Gelatinization and retrogradation

In the conventional gelatinization method, the starch granules are slowly heated under little stirring in the presence of water, which promotes imbibition, swelling and release of amylose and amylopectin polymers over a prolonged period (Leach, 1965), such as in the preparation of cooked rice, porridge, and cereal and tuber soups. Due to the cost and time to prepare starch products, various methods have been successfully used, such as extrusion, roller drying, drum drying, microwave, dryer ovens, thermoplastic extruders, high pressure processing and many others, which promote rapid starch gelatinization followed or not by drying, thus obtaining pre-gelatinized starches that can be used as cold thickeners.

The starch gelatinization is a physical process, in which the granule swells and breaks when heated in water, releasing amylose and amylopectin, forming a thick solution, due to the numerous hydrogen bonds with water. The starch gelatinization process depends on starch origin and physical and chemical characteristics, type of damaged starch, amylose/amylopectin ratio, whether native or modified, composition of food to be added, presence of sugars, salts, fat, fibers, and the processing conditions, including the rate of temperature rise, agitation of the medium, and process type (Leach, 1965). Table 1.6 shows some examples of starch paste from cereals and tubers, with some important differences such as paste clarity and texture.

After the starch gelatinization, with paste formation and the amylose and amylopectin molecules forming hydrogen bonds with the water and cooling of the paste, retrogradation and syneresis take place, that is, there is a new organization of the molecules, with an increase in viscosity of the medium and water release (Leach, 1965). This process occurs during cooling of cereal-based porridges, such as oats and corn, with the formation of surface layers, which is less noticeable in cassava and potato-based porridges, due to their slow retrogradation rate and syneresis.

**Table 1.6** Pasting properties of native starch *

| Starch | Hot Viscosity | Texture | Clarity | Agitation Resistance | Retrogadation Speed During Cooling |
|---|---|---|---|---|---|
| Corn | Medium | Short | Opaque | Medium | High |
| Waxy Corn | Medium High | Long | Clear | Low | Medium to Low |
| Wheat | Medium to Low | Short | Opaque | Medium | Medium High |
| Sorghum | Medium | Short | Opaque | Medium | High |
| Rice | Medium to Low | Short | Opaque | Medium | High |
| Potato | Very High | Long | Clear | Medium to Low | Medium |
| Cassava | High | Long | Clear | Low | Low |
| Sweet Potato | High | Long | Clear | Low | Medium |

*Adapted from Swinkels (1985), Whistler and BeMiller (1997)

Starches with higher amylose content retrograde in the first few hours of cooling, while waxy starches show little increase in viscosity during cooling and freezing, thus they are preferred for products that will be subjected to freezing (Swinkels, 1985).

Many modification processes aim at altering the gelatinization and retrogradation, since these properties are unique to starch, thus impairing its application in the native form as a substitute for sugar, fat, and protein in foods.

### 1.4.2 Ability to undergo changes

According to Light (1990), the modified starch can be used in food for 3 main reasons:

- Provide functional attributes in food applications that normally native starches cannot provide. Ex. pudding mix, salad dressing;
- Be one of the most available nutrients for modifications; and

- Provide economic advantages in many applications using more expensive additives, such as gums.

The first starch modification is the enzymatic modification that occurs naturally in plants, during synthesis and degradation processes. In grains, the germination process increases the amylase activity, leading to the starch hydrolysis. For human consumption, the gelatinized starch undergoes hydrolysis by the salivary and pancreatic amylases of the digestive system, releasing smaller molecules, which will be easily hydrolyzed (Escott-Stump and Mahan, 2013).

The enzymatic modification of starch has become commercially important after the production of enzymes at industrial scale, such as germinated cereals (malting process) or the production of amylases by bacteria and fungi, meeting the high demand for starch products derivatives.

The advantages of using enzymes are the specificity of the reaction, which occurs in mild conditions of temperature, pH, and agitation, in addition to the low generation of effluents. The enzymes concentration is much lower than that of starch, and the reaction may be interrupted with small variations in the reaction medium. The enzyme-modified starch is considered GRAS (Generally recognized as safe). Glucose/fructose syrups and alcoholic drinks such as whiskey and beer are examples of products that undergo enzymatic activity using starch as the starting substrate (Dendy and Dobraszczyk, 2001).

The physical modifications can be smooth and come from the extraction process, leading to the formation of damaged starch, or drastic as gelatinization, the formation of dextrins by pyroconversion, among others (Wurzburg, 1989). All these processes are considered GRAS.

Chemical modification is directly associated with the side hydroxyl groups of the amylose and amylopectin molecules, which are capable of going thought hydrolysis, oxidation, etherification, esterification, and cross-linking (Wurzburg, 1989). The resulting modified starch exhibits different properties when compared to the native starch, and the degree of modification may vary according to the chemical reagent, process time, and temperature. In this case, starch should be modified within the specific legislation and in the limit amount for use in food products. For example, the phosphate in the form of phosphorus in starch molecules is not more than 0.5% for potato or wheat, and cannot be more than 0.4% for other phosphate starches (Hamilton and Pascall, 1967).

Many combined methods have been used to reduce the chemical reagents and the generation of effluents. Many modified starch processes are protected by patents, once the large industries have always searched for modified starches for new applications or to improve the technological quality of the existing ones.

### 1.5 Conclusion

As a key ingredient for human nutrition, starch presents great industrial importance, since it is a product of easy obtaining and characterization. However, small modifications in the granule can lead to great changes in the technological properties, thus studies in this area are in constant expansion.

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# CHAPTER III

## IDENTIFICATION AND ANALYSIS OF STARCH

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This chapter presents a review of the physical, technological and instrumental analyses used to identified starch properties

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# Identification and analysis of starch

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## 2.1 Introduction

A number of methods with different purposes can be used to characterize starch in foods, including the identification, quantification, assessment of purity degree, type of physical, chemical, and enzymatic modifications, and interactions between starch and other compounds. In addition, the nutritional characteristics of starch, its stability to the processing and storage conditions, and the interactions with the environment can also be assessed. In this context, specific evaluation methods have been developed to provide information to guide the scientific development in the field of starch granules.

The selection of analysis methods should fit with overall aims, including time of analysis, operating costs, ease of execution, waste generation, besides having to meet the common requirements of a laboratory analysis, such as precision, accuracy, repeatability, detection limit, and specificity. As an example, the presence of raw starch in foods can be investigated by the simple use of Lugol's solution (iodine solution and potassium iodide), or by optical microscopy, scanning electron microscopy, and atomic force microscopy methods, whose choice will depend on the type of response desired.

Starch is composed of the major three elements, i.e. C, H, and O; thus its quantification is quite complex, since these elements are found in almost all organic compounds. For this reason, in many food composition tables, the carbohydrates level, which includes sugars, dietary fibers, and starch is calculated by difference by the sum of percentages of water, protein, minerals, and lipids subtracted from 100.

This chapter will cover the main analysis methods of starch granules in a didactic way, from the simplest to the most sophisticated techniques.

## 2.2 Identification of the starch grain

Starch granules can have specific requirements for food and non-food uses. The product specifications include the purity degree, origin, and whether it is native or modified starch, which can be assessed by determining the moisture levels, ash, pH, acidity, fiber, color characteristics, presence of sulfur dioxide, particle size distribution, paste viscosity, phosphorus content, microbiological specifications, among others. All methodologies can be easily found in books intended for food analysis, such as AOAC and AACCI.

Many countries have their own legislation, aiming to establish the conformity of starch with regard to commercialization. The analysis method and the interpretation of the results should be well established for those working with starch grains, since many application and modification processes require high purity starch, without starch-based blends and microbial contamination.

Considerations regarding the interpretation of results will be given below, once the starch identification and characterization is the first step for the scientific development in the field of starch granules.

Raw starch, when purified and in the dry powder form, should be white, odorless with a characteristic flavor, and insoluble in water below its gelatinization temperature. The Lugol solution can be used to quickly identify the raw or cooked starch, pure or not, which will color red (glucose, low molecular weight dextrins) to blue (raw or gelatinized starch).

Starch is also insoluble in alcohol, ether, and other solvents, and soluble in dimethylsulfoxide (DMSO), and has a density of approximately 1.5 $g/cm^3$ (Brasil, 2010), so the extraction and purification process are favored. According to Singh et al. (2007), the starch solubility in DMSO can vary according to the type of starch, particle size, amylose content, and the modification processes.

The starch color can be measured by the CIELab system, which is represented by the color coordinates L* representing luminosity (0 = black; 100 = white), a* (+ a = redness; -a = greenness), and b* (+b = yellowness; -b = blueness), while the parameters Hue (hab) and Chroma (C*) can be obtained from the a* and b* values. The parameter Hue is measured as an angle over a circle divided into 100 parts, while C* is a measure of opacity (Giese, 2000; Minolta, 1994). It is also possible to use comparative color systems, such as the Maerz and Paul's color dictionary (1950), or by reflectance using a reflectance meter that measures the intensity of whiteness (Mishra and Rai, 2006).

Commercially, starch granules are white and opaque, but when extracted under laboratory conditions and without the use of oxidizing reagents or enzymatic inhibitors, they can exhibit from yellow to green tones due to the presence of pigments or enzymatic browning, such as the starch from wolf fruit, chestnut, and bamboo culm, as reported by Clerici et al. (2011), Schmiele et al. (2015), and Felisberto et al. (2017), respectively, as slightly colored starches (gray).

The identification of the starch origin is of paramount importance and can be a simple task for the raw starch. Although optical microscopy (OM) allows the characterization of granule shape and origin, the origin cannot be identified in gelatinized, very broken, or micronized starch. The Maltese cross of the raw starch can be seen under polarized light and presents differences according to the type of starch, birefringence, and shape characteristics, which can be spherical, oval, polygonal, disc, elongate, and others. The positive birefringence indicates a radial orientation of the macromolecules, which is normal for the growth rings and the surface of the granule, confirming that the extraction process did not affect the granule structure, once according to BeMiller and Whistler (2009), the loss of birefringence on heating is indicative of disordering processes.

The intrinsic viscosity is essentially a measure of the internal friction or resistance to the displacement of polymer molecules in solution. If properly used, it provides an excellent criterion of relative molecular size (Islam et al., 2001). Starch grains from different plant sources have different intrinsic viscosity, such as those found by Rocha et al. (2008) for two Peruvian carrot varieties, from 2.39 to 2.18, and by Franco et al. (1988) for manioc starch and maize starch, with values of 2.30 and 1.83, respectively.

Measurements of pH and acidity vary according to the type of starch and the degree of impurities, including the presence of minerals, free fatty acids, proteins, phenolic compounds, sulfite or others, which can affect the pH due to the anion release in the starch solution water. In addition, during the extraction process, the long-term dissolution can lead to fermentative and enzymatic processes with the formation of acidic compounds. To avoid this process, sulfur-containing compounds such as bisulfites can be used; however, for some starches, no residual sulfur levels are acceptable at the end of the process. Mishra and Rai (2006) determined the pH values of maize, potato, and tapioca starches, which were 6.2, 7.15, and 4.8, and correlated them with the phosphorus content, since potato starch had the highest phosphorus content (41 mg%), followed by corn (23 mg%) and tapioca (7.54 mg%).

Demiate and Kotovicz (2011) analyzed the pH and acidity of cassava starch, and found that the native starch presented a pH value of 4.99 and acidity corresponding to 0.75 mL of NaOH 1 N.100 $g^{-1}$, while the sour cassava starch presented low pH (2.92 to 4.03) and high acidity values (3.10 to 8.63 mL of NaOH 1 N.100 $g^{-1}$). These starch granules presented different expansion properties when dissolved in water and cooked, with a greater expansion of the sour cassava starch, which is widely used for gluten-free cookies in Brazil.

The cyanogenic glycosides (linamarin and lotustralin) found in cassava and other plants must be present in starch below the limit of detection. The analyses can be performed by spectrophotometric (Fukushima et al., 2016) or chromatographic methods, as reported by Cho et al. (2013), who measured the total cyanide by ion exchange chromatography following acid hydrolysis and distillation of cyanide ion collected in sodium hydroxide. The major drawback of this analysis is the safety of the method due to the toxicity of the compound tested.

After the identification and evaluation of the degree of purity for use in chemical and physical modifications, the analysis of the damaged starch can also be performed, once the damages suffered during the extraction process can change the starch characteristics, such as solubility, water swelling, and pasting properties, in addition to increasing the susceptibility to fermentative process and enzymatic hydrolysis. Damaged starch can be determined by extraction procedures (Blue Value), dye-staining procedures, near-infrared procedures and enzyme digestion procedures, as described by AACCI (2010):

- Method 76-31.01 Determination of Damaged Starch - Spectrophotometric Method. The damaged starch granules are hydrated, followed by maltosaccharide hydrolysis and limit dextrins by fungal alpha-amylase. Amyloglucosidase is then used to convert dextrins to glucose, which is specifically determined spectrophotometrically after glucose oxidase/peroxidase treatment.
- Method 76-33.01 Damaged Starch - Amperometric Method by SDmatic. The method measures the kinetics of iodine absorption in a liquid suspension, using an amperometric probe. This analysis was developed for wheat flour since the damaged starch increases the water absorption of flour, which is important in the bread fermentation process.

Damaged starch is very important in wheat flour for use in bakery products and pasta. In addition, Horstmann et al. (2017) have shown that it is also important for the production of gluten-free bread, once it can affect the water absorption properties, solubility, dough fermentation rate, and color of the final product.

Phosphate groups may be naturally present in starch granules, as in potato starch (Mishra and Rai, 2006) or from phosphate bonds (Seker et al., 2003) or in the form of natural phospholipids, as in barleys (Song and Jane, 2000), or added (Ai et al., 2013), and can be evaluated by the molybdenum blue spectrophotometric method described by Smith and Caruso (1964), and 31P nuclear magnetic resonance (31P NMR ) (Sang et al., 2007).

To avoid determination errors and the use of toxic reagents for phosphorus analysis, Genkina and Kurkovskaya (2013) proposed a method based on the chemical gelation of starch in a starch-phosphatidylcholine blend, using DMSO followed by the quantification of phosphorus int the gel formed by $^{31}$P NMR using tributyl phosphate as a standard.

The microbiological characterization, presence of toxins, and degree of soiling depend on the specifications of each producer country, as it indicates the hygienic-sanitary safety of the starch manufacturing processes, for example, the starch granules for use in enteral, parenteral, infant, and special formulas, which must follow strict microbiological standards, including spores counts. Starch extracted from the artisanal manner and sun-dried may be at higher risk for contamination.

## 2.3 Physicochemical characterization

Although the main starch fractions (amylose and amylopectin) are composed of the same glycosidic monomer, glucose, they exhibit totally different characteristics and performance. The presence of both macromolecular components may interfere with the physicochemical analysis, thus it is necessary to separate these fractions. Other compounds, such as lipid fractions, also need to be removed.

### 2.3.1 Separation of amylose and amylopectin

Starch fractions can be separated using well-established methodologies. The interference of amylopectin on the amylose quantification, as well as the intermediate material, can be minimized through the fractionation of the polymers. Better fractionation and analysis conditions are obtained with the use of free fatty starch granules. The degreasing process is usually obtained by cold extraction using the Soxhlet system or by immersing the sample in petroleum ether or hexane. After that, the sample is solubilized in 90% dimethylsulfoxide (DMSO) solution at room temperature and constant stirring, or in a heating bath using 6-10 molar urea (to reduce the dielectric constant of the solvent), or using an alkaline solution of NaOH or KOH ≤ 2 molar in a heated medium (to promote starch solubilization at alkaline pH) (Jane, 2009). At high pH levels, greater attention is required with heating, as it may lead to the alkaline starch degradation, thus affecting the determinations.

The more common techniques for separation is the solubilization of amylose that is complexed with 1-butanol. After total starch gelatinization, the alcoholic reagent is added in the proportion of 20% (v / v) and allowed to stand at room temperature for 24 to 36 hours for to form the complex. The separation is performed by centrifugation and the sample can be subjected to a further amylose complexation step with 1-butanol to improve the process efficiency (Schoch, 1942).

#### 2.3.1.1 Amylose

The alpha-helix chain of amylose has the characteristic of complexing with iodine, as the inner part of the helical conformation is hydrophobic. The determination of the amylose content is traditionally accomplished by the formation of amylose-iodine complex, generating a blue color, due to an electron relay of the polyiodide ions (Jane, 2009). The main interference of this analysis is the ability of amylopectin long chains to form complexes with iodine, leading to a less intense blue staining, thus overestimating the amylose content. The ability of lipids (especially free and saturated fatty acids) to physically complex with the amylose alpha-helix may also occur, reducing the ability to complex with iodine and underestimating the results. For this reason, the amylose levels reported in the literature refer to the apparent amylose. As an example, the native maize starch exhibited an apparent amylose content of 26.38% while the absolute amylose was 19.94%, for the same sample.

The interference of the amylopectin long chains was observed in chestnut starch, with apparent amylose of 24.00% and absolute amylose of 20.48%. The chestnut starch presented 11.12% amylopectin chains with a degree of polymerization (DP) above 37 (whereas the highest chain detected presented DP of 76) and average chain length of amylopectin of 19.60, while maize starch presented only 7.45% amylopectin chains with a degree of polymerization > 37, with an average chain length of amylopectin of 18.75 (Sehn et al., 2012; Schmiele et al., 2015). It is known that a DP around 42 is a characteristic of B2-chain, with chain length belonging to two clusters.

The blue color formed in the amylose-iodine complex is also used for the determination of blue-value (absorbance of 1% starch solution containing 2 mg iodine and 20 mg potassium iodide at 680 nm). The greater the linear chain length and the higher the amylose concentration, the more intense is a blue color. The blue-value of starch granules is considered a qualitative test for amylose (Bertoft, 2004). Table 2.1 shows the blue-value of some starch granules.

**Table 2.1 –** Amylose content and corresponding blue value of different starches

| **Starch** | **Rice**[1] | **Waxy rice**[1] | **Yam**[2] | **Pueraria root**[3] | **Potato**[4] | **Sweet Potato**[3] | **Corn**[4] |
|---|---|---|---|---|---|---|---|
| Blue value | 0.0594 | 0.004 | 0.530 | 0.399 | 0.310 | 0.447 | 0.400 |
| Amylose content (%) | 25.50 | 0.99 | 26.56 | 30.30 | 16.70 | 34.40 | 21.30 |

[1]Lii, Shao, and Tseng (1995); [2]Ogunmolasuyi et al. (2017); [3]Liang et al. (2017); [4]Saibene et al. (2008).

The apparent amylose can also be measured by the iodine affinity, through potentiometric titration or with an automated amperiometer. The technique is based on the affinity of the α-helix with the iodine forming a blue complex. On average, 20 mg iodine complexes with 100 mg amylose, therefore the apparent amylose concentration is given by the multiplication of iodine affinity by 5 (Takeda et al., 1987), once approximately 20 g of iodine is bound per 100 g amylose (Song and Jane, 2000).

The determination of the apparent amylose may be affected by the amylopectin long chains, resulting in a false positive, thus the determination of absolute amylose can be performed as an alternative. For that, starch is suspended in DMSO, followed by precipitation of amylopectin with lectin concanavalin A (glycoprotein capable of complexing with some carbohydrates). After centrifugation, the amylose is hydrolyzed to glucose by the amylolytic enzymes, and the monosaccharide is quantified using the glucose oxidase/ peroxidase reagent (GOPOD). Concanavalin A should be used with caution due to its toxicity.

The absolute amylose can also be determined by fractionation of the amylose and amylopectin and subsequent evaluation of the iodine affinity of these fractions and the parent starch. The final calculation is made by the following formula: $(I_{starch} - I_{amylopectin})/(I_{amylose} - I_{amylopectin}) \times 100$ (Song and Jane, 2000).

The most accurate techniques used for the amylose quantification are based on high-performance size-exclusion chromatography (HPSEC), coupled to simultaneous refractive index (RI) and low-angle laser light scattering (LALLS) detection, or gel-permeation chromatography (GPC). However, in all these procedures, the amylopectin fraction remaining in the sample may be an interference factor. Thus, an alternative is the use of debranched enzymes, which will promote the hydrolysis of α-1-6 glycosidic bonds, releasing amylopectin short chains, and consequently improve the separation of longer amylose fractions.

**2.3.1.2 Amylopectin**

The quantification of amylopectin can be performed after the amylose separation by enzymatic hydrolysis to release glucose, followed by GOPOD quantification. However, this information does not provide the amylopectin chain length, which is critical to the study of this fraction.

In this regard, the most suitable method for determining amylopectin refers to the quantification of the number of chains of this macromolecule. For that, after amylose separation, the sample is subjected to total gelatinization and hydrolysis of the glycosidic bonds using the enzyme isoamylase. Then, the linear chains are quantified by High-Performance Anion Exchange Chromatography with Pulse Amperometric Detection (HPAEC-PAD) (Wong and Jane, 1995; Moraes et al., 2013; Schmiele et al., 2015). Linear chains can also be identified by GPC or HPSEC, and classified as A (DP 6-12), B1 (DP 13-24), B2 (DP 25-36), B3 (DP ≥37), and C (DP ≥100) chains (Hanashiro et al., 1996).

Magnetic Nuclear Resonance (NMR) spectroscopy may be an alternative technique to study the amylopectin conformation, through the variations in the 1H- and 13C-NMR (Nilsson et al., 1996; Zhang and Xu, 2017). It is a non-destructive technique, thus, it does not generate effluents and does not require enzymatic treatment.

**2.3.1.3 Intermediate and phytoglycogen material**

In addition to amylose and amylopectin, the starch granules also contain intermediate materials called glucans. These materials are more easily found in starch granules with high amylose content, mainly from potato, barley, and corn, and some types of peas, including those with a wrinkled appearance, besides oat, normal corn, wheat, and rye. However, the intermediate materials are not yet fully known and vary according to the starch source. In addition, they are heterogeneous and have structures similar to amylose due to the iodine affinity (Bertoft, 2004) and amylopectin due to the high average internal and external chain length (Han et al., 2017). Thus, the intermediate materials can be defined as a polymeric material formed by glycosidic linkages between glucose monomers, with a branched structure similar to amylopectin and amylose chain length (Li et al., 2008).

These intermediate materials are probably originated from immature clusters formed by the enzyme granule-bound starch synthase (GBSS), which is responsible for the formation of amylose (Hanashiro et al., 2008), in synergy with the starch synthases (SS) and starch branching enzymes (SBE) and debranching enzymes (DBE) (Wang et al., 2017), resulting in the cleavage of amylose chain and the transfer of chains to an amylopectin molecule. However, this phenomenon has not yet been confirmed experimentally (Vilaplana et al., 2014).

The quantification of intermediate materials of cereal starch was performed through the dispersion/solubilization of starch in dimethyl sulphoxide and subsequent separation of amylose and amylopectin. This fractionation was obtained by the complexation phenomena between amylose and the intermediate materials with thymol (phenolic compound belonging to the terpene group), with precipitation and separation of amylopectin. Then, amylose was reprecipitated in butanol, with the intermediate material consisting of anomalous amylose and/or long chain amylopectin remaining soluble (Banks and Greenwood, 1967).

Similarly, the primary precipitation of amylose with 1-butanol was also carried out, followed by precipitation of the intermediate materials with iodine from the amylopectin fraction, showing that the higher the amylose content, the higher the intermediate material concentration (Adkins and Greenwood, 1969). To improve the separation of the components, Wang et al. (1993) used gel-permeation chromatography (GPC) on Sepharose CL-2B, and identified smaller branched components rather than amylopectin, at different concentrations depending on the starch source.

A blend containing 6% 1-propanol and 6% isoamyl alcohol can also be used to separate the amylose and amylopectin from the intermediate material. These methods confirm that although the intermediate materials have characteristics similar to amylopectin, they are capable of precipitating with 1-butanol, such as amylose. In addition, the regular branched structure of the intermediate materials may exhibit partial inhibition of enzymatic hydrolysis, thereby reducing the starch digestion rate (Bertoft et al., 2000), which is a tendency of the slowly digestible starch, rapidly digestible starch, and resistant starch (topics that will be covered in more detail in Chapter 8).

A water-soluble polysaccharide can also be found in the starch fraction, especially in the sugary–1 mutants of maize and rice endosperm. This component is called phytoglycogen and presents glucose as monomer and degree of polymerization of ~ 10, with internal branches with DP between 7.0 and 8.0 and extremely short external branches with DP ~ 3.0 (Jane, 2009).

## 2.4 Morphological and structural characterization

### 2.4.1 Polarized light microscopy

Optical microscopy allows the evaluation of starch characteristics in relation to the shape, size, and distribution of the granule from different plant sources (Van de Velde et al., 2002). When a polarized lens is used, the Maltese cross can be seen under the polarized light when starch has not undergone gelatinization. As soon as the starch undergoes heating in the presence of water, birefringence end point temperature (BEPT) loss is observed. This is a simple test with good sensitivity.

The loss of birefringence occurs over a wide temperature range (10 to 15 °C). However, there is a good relationship between BEPT and DSC analysis for starch granules from different plant sources (Biliaderis, 2009).

### 2.4.2 Scanning electron microscopy

Surface images are extremely important for studying the starch granules. The first techniques involve scanning probe microscopy (SPM), atomic force microscopy (AFM) and scanning tunneling microscopy (STM). The image resolution was improved with the advent of scanning electron microscopy (SEM). Starch is a non-conductive material, which is a limitation factor, thus the use of metals (gold or platinum) in coating the surface of biological material minimizes this limitation. The technique is performed within a vacuum chamber, which may partially modify the surface of the starch granules, which can be prevented by using moderate vacunm. This new method is called environmental scanning electron microscopy (ESEM) and is widely used for both the study of plant genetic development (Stabentheiner et al., 2010) and for high-moisture samples (Roman-Gutierrez et al., 2002).

The SEM technique presents the best image resolutions of dehydrated samples. When water removal is not possible or the matrix can be altered by drying, the temporary immobilization of the aqueous phase (freezing) is important to allow cryo-SEM imaging. In this technique, although the image resolution is lower, it is an alternative when studying the swelling power, water absorption, amylose leaching, retrogradation phenomena (Matignon and Tecante, 2017), and the stability of emulsions or nanoemulsions using modified starch as emulsifiers (Zhang et al., 2016; Chiu et al., 2017).

High-quality images can be obtained by low-voltage scanning electron microscopy (LVSEM), as the onset of low voltage electrical current promotes lower heating rates (Perez et al., 2009; Huang et al., 2017).

SEM also allows the study of the surface characteristics (smooth or porous), particle size, format (polyhedral, oval, flattened, hexagonal and whether the starch is unimodal, bimodal, or trimodal.)

### 2.4.3 Confocal laser scanning microscopy

Confocal microscopy allows the evaluation of two-dimensional (2D) or three-dimensional (3D) topography of starch through high-resolution images using chromophore compounds with fluorescence properties, allowing a detail evaluation of surface area and depth. In addition, it is possible to identify how amylose is uniformly distributed in the starch granule (Jane, 2009).

The presence of other components is also possible through the use of chromophores, also known as dyes or markers, which form complexes with specific components of the material to be evaluated, either with proteins (3-(4-carboxybenzoylquinoline-2-carboxaldehyde or fluorescein isothiocyanate), lipids (NILE RED, 5-hexadecanoylaminofluorescein, fluorescein octadecyl ester) and carbohydrates (Rhodamine B) (Han and Hamaker, 2002; Achayuthakan et al., 2012). A wide range of dyes has also been used with promising results.

This technique has been widely used to evaluate the interaction between starch granules and various components, including hydrocolloids such as xanthan gum (Gonera and Cornillon, 2002), gum acacia and dextran (Achayuthakan et al., 2012), proteins such as gelatin, whey protein, and soy protein isolates, and lipids (Davanço et al., 2007; Matignon et al., 2014).

### 2.4.4 Atomic force microscopy (AFM)

Atomic force microscopy (AFM) has been used to observe the nanoscale of the structure of starch granule from different plant sources Baldwin et al. (1996), Baldwin (1997), Baker et al. (2001), Ridout et al. (2002) observed a blocket structure, which has been studied by Gallant et al. (1997). AFM allows observing that the size of the blocket structure can vary according to the starch granule. For example, the surface of native wheat starch had small protrusions of 10 to 50 nm, while potato starch exhibited larger spherical protrusions of 200 to 500 nm (Baldwin et al., 1996; Baldwin, 1997).

According to Ridout et al. (2002), the AFM images of the growth rings and the blocklet structures of native potato and maize starch, unstained starch and unmodified starch, and found that the images were affected by the embedding resin used. Ridout et al. (2002) found similar results and reported that the fixation techniques can lead to possible unintended reactions with embedding resin such as epoxy resins or adhesives. Other authors reported modifications in obtaining the images, as follows:

- Szymonska and Krok (2003) performed AFM studies on starch granules by high resolution non-contact atomic force microscopy (nc-AFM), which did not affect the sample surface, and observed the blocklet model of the potato starch granule subjected to multiple freezing and thawing. They observed surface sub-particles that might correspond to the single amylopectin side chain clusters bundled into larger blocklets packed in the lamellae within the starch granule.
- Park et al. (2011) developed a novel AFM protocol, using starch subjected to iodine vapor under humid conditions. They found vertical fiber-like structures, which are extensions of the glucan polymers from either amylose or amylopectin that are free to complex with iodine in the presence of moisture. In addition, they reported that the morphology and location of the hair-like extensions were different in corn and potato starches, likely reflecting the organization of polymers within the granule.

The advancement of AFM images can provide new findings, such as the chemical, enzymatic, and physical modifications in the native granule and the interactions between starch granules and other compounds, including the studies of:

- Dimantov et al. (2004), who studied films obtained with a blend of pectin and high amylose maize starch by AFM, and found the increased roughness with the increase in starch concentration in the films.
- Simão et al. (2008), who found growth rings in starch granules during ripening of mango.

### 2.4.5 Differential scanning calorimetry

The differential scanning calorimetry (DSC) is a technique that evaluates the changes in starch granules in relation to the molecular mobility and the ordering/disordering processes during heating and cooling. It is widely used to evaluate several factors, including temperature, heating, and degree of gelatinization, glass transition, structural organization of starch, melting and crystallization of starch components (important for the retrogradation measurement) among others, such as resistant starch type 2 (naturally present) and resistant starch type 5 (amylose-lipid complex) (Pérez et al., 2009).

In general, the determination is made by measuring the temperature difference between the sample placed in a hermetically sealed container and an empty container referred to as a reference. The lower the variation between the beginning and end temperatures of the thermal event, the greater the organization of the starch structure. Then, the parameters gelatinization temperature ($T_{on}$ = onset temperature), final gelatinization temperature ($T_{end}$), temperature variation (between $T_{end}$ and $T_{on}$), and gelatinization enthalpy (area under the curve – $J.g^{-1}$) are obtained, as you can see in Figure 2.1.

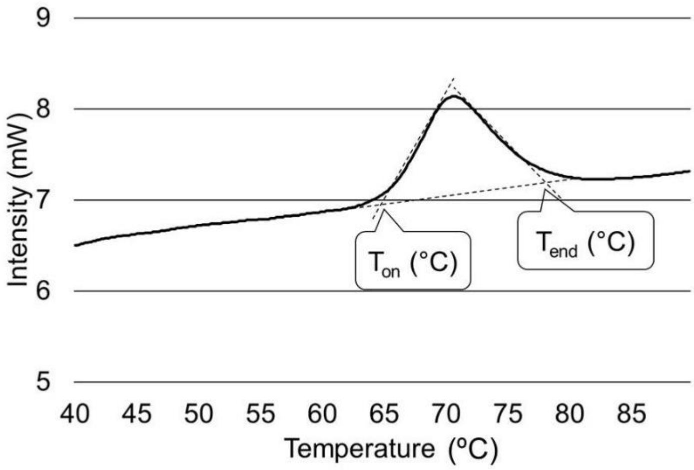


**Figure 2.1** Differential scanning calorimetry curve for maize starch.

The glass transition is one of the most important parameters for the study of the properties of amorphous or partially crystalline polymers, changing the mechanical properties, the behavior of the material during the processing, and the stability.

### 2.4.6 Nuclear Magnetic Resonance (NMR)

NMR is a qualitative and quantitative method, which shows the shape and structure of the molecules, and its most important application is in the study of hidrogen atoms in organic molecules. The hydrogen atom is perhaps the easiest to understand from the point of view of its physical properties (Robinson, 1995). For starch analysis, the following isotopes are used: $^{1}H$, most used and distinguishing H from α- 1,4, and α-1,6 glycosidic bonds; $^{13}C$ distinguishing C associated with the non-reducing endings, and $^{31}P$ indicating the phosphodiester crosslinks, in the naturally occurring or chemically modified starches.

NMR has been used to evaluate the formation and localization of new functional groups in modified starches, for example, the carboxyl groups found in oxidation processes. Teleman et al. (1999) showed that the hypochlorite oxidation of potato starch occurred at positions C-2 and C-3 of a glucose unit, and the introduced carboxyl groups caused ring cleavage between the carbons C-2 and C-3.

The authors studied the anion-exchange chromatography after enzymatic hydrolysis to isolate the pentamer and hexamer containing one glucose unit, which was oxidized to a dicarboxyl residue, and further evaluated through $^{1}H$ and $^{13}C$ NMR spectroscopy.

NMR has been used along with other techniques to evaluate the physical, chemical, and enzymatic modifications, type of starch, behavior during cooking, retrogradation, among others.

### 2.4.7 X-Ray Diffraction

X-Ray diffraction has been used to determine the crystalline structure and extent of starch granules aimed to know its plant origin. Starch granules are analyzed in the native form, and the crystalline pattern may change or even disappear in case of modification reactions.

The degree of crystallinity of the native starch ranges from 15 to 45% (Cheetham and Tao, 1998) and it is directly related to the amylopectin content and chain length, and inversely proportional to the amylose content (Hoover, 2001).

The X-ray diffraction patterns of starch granules are classified according to the packing of the double amylopectin helices and present basically four types: A, B, C, and V. The A-type structure is associated with cereal starches (maize, rice, wheat, and oat), while B-type is associated with root and tuber starch (potato, cassava), except for starch with high amylose and amylopectin contents, and modified starch (Cheetham and Tao, 1998; Eliasson, 2006). The C-Type structure is formed by the mixture of A-type and B-type, representing legume starches (beans, peas) and some mutant starches, with A-type around the pericarp and A-type in the center of the bead (Buleon and Colonna, 2007). The C-Type can also be subclassified into Ca, Cb, and Cc, according to its similarity with A-type and B-type. The V-type is associated with lipid-containing gelatinized starches (Eliasson, 2006) or emulsifiers, butanol, and iodine (Cheetham and Tao, 1998). It is still possible to find the E-type structure in extruded starch under different humidity and temperature conditions (Vandeputte et al., 2003).

These crystallinity patterns can also vary according to the size of the amylopectin chain; shorter chains ($CL \leq 19.7$) favor the formation of A-type crystals, longer chains ($CL \geq 21.6$) favor the B-type structure, while the association between the intermediate chain leads to the formation of C-type crystals (Hizukuri et al., 1983).

This behavior was also observed by Cheetham and Tao (1998), who studied maize starch with different amylose and amylopectin levels and found an increase in crystallinity with increasing the amylopectin content (Eliasson, 2006).

Diffraction methods and calculation of the crystalline region have been reported by Srichuwong et al. (2005), Tester et al. (2004), Nara and Komiya (1983), Rocha et al. (2008), and Hayakawa et al. (1997).

### 2.4.8 Fourier-transform infrared spectroscopy

Infrared radiation is electromagnetic radiation with longer wavelengths than those of visible light and extends from the nominal red edge of the visible spectrum at 780 up to about 50 μm. The main objective of IR spectroscopy is to qualitatively evaluate the presence of functional organic groups, such as carboxyls, carbonyls, hydroxyls, as each group absorbs a characteristic radiation frequency in the infrared region. After the sample is analyzed in the spectrometer, a graph gives the intensity of the emitted radiation per frequency, which will allow characterizing the functional groups of a standard or an unknown material.

The IR spectra, together with other spectral data, are useful for determining the molecule structure since each compound has a typical identity, which can be compared with known compounds and free database software. When the quantification of the functional groups is necessary, the analysis is done using Near-infrared spectroscopy (NIRS) with evaluation of the bands characteristic in the fingerprint region from 1350 to 910 $cm^{-1}$.

In addition to evaluating the presence of other compounds by the identification of functional organic groups, FTIR spectroscopy can be used to evaluate the chemical modifications of starch granules. Demiate et al. (2000) evaluated chemically oxidized cassava starch, commercial cassava sour starch, and native cassava starch by FTIR spectroscopy associated with chemo

metric data processing and qualitative and quantitative spectral analyses. The authors found the presence of carboxylate groups (1600 $cm^{-1}$) on cassava starch as well as some other changes in the region around 1060 $cm^{-1}$, and concluded that the degradative oxidation is assumed to take place on the C–O bond relative to carbon 1 and oxygen 5 of the cyclic part of glucose at 1060 $cm^{-1}$.

## 2.5 Rheological characterization

The rheological properties describe the behavior of materials subjected to shearing forces and deformation, which are considered viscoelastic complexes (Alcázar-Alay and Meireles, 2015). Rheological measurements were initially performed in a rheometer, obtaining the information of storage modulus (G "), which indicates the elastic behavior of starch, loss modulus (G”), which describes the viscosity behavior, and tan δ (G”/G’), indicating liquid-like (>1) or solid-like (<1) behavior (BeMiller and Whistler, 2009). Several analyses have been carried out to evaluate the behavior of modified or unmodified starches, as reported by Neder-Suarez et al. (2016), who evaluated the behavior of extruded resistant starch pastes during the refrigerated storage.

Acosta-Osorio et al. (2011) reported that the conventional rheometers cannot be used in the case of starch cumuli because the particles have similar dimensions than the gap used in this type of instrument. Also important are the changes taking place in the particles as a function of time and temperature. On the other hand, when the sample is placed in conventional rheometer geometries, the measuring process may result in the partial destruction of the internal structure. These authors corroborated the explanations of Bongenaar et al. (1973), who reported that the rheology of starch suspensions determined by conventional method is not appropriate for fluid systems with suspended solids. Therefore, the authors have proposed the use of a system consisting of a spindel coupled to a motor and a torque wrench, which measures the torque caused by the resistance of the starch suspension to agitation. However, the measured viscosity should be referred to as apparent viscosity, thus Metz et al. (1979) used an impeller viscometer system (as RVA), which measured the torque of the impeller, aimed to obtain information about the apparent viscosity of suspended-particle systems.

Therefore, the analysis of starch granules by conventional rheology was replaced by the empirical rheology, using viscoamylograph that can inform the pasting properties during cooking and cooling, and with stirring.

### 2.5.1 Empirical Rheology

The paste properties of starch are of great importance for the food industry to estimate the starch behavior during processing (Shelton and Lee, 2000). The paste profile is dependent on the starch origin, the water content in the system, the presence of other chemical compounds, and chemical, physical, and genetic modifications. The viscosity of the starch paste can still be influenced by the different temperatures and shear rates provided in the equipment (Zhu, 2015).

Brabender and RVA are the most widely used equipment for evaluating the paste properties of starch, which simulates heating and cooling of a starch-water suspension under constant agitation (Shelton and Lee, 2000; Ross et al., 2012; Zhu, 2015) aiming to obtain viscosity and temperature information under the conditions evaluated.

After many studies on starch characteristics, the pasting properties can be considered as a fingerprinting of starch, which can provide information about the starch origin, degree of purity, the processing characteristics, damaged starch content, enzymatic activity, presence of chemical compounds that promote oxidation reactions, use of high temperatures, as well as the effect of nutrients (Zhang and Hamaker, 2003), additives, organic compounds (Fu et al., 2015), minerals (Zaidul et al., 2007) and amylolytic enzymes (Wang et al., 2009; Uthumporn et al., 2013; Dura et al., 2014; Benavent-Gil and Rosell, 2017).

Brabender® viscoamylograph can record the system torque and rotation by providing the viscosity profile in Brabender Units (BU) with temperature of the sample is increased at a rate of 1.5 °C/min (Swinkels, 1985). Equipment such as RVA and more recently Microviscoamilograf Brabender® was developed with the aim of reducing analysis time and amount of sample, due to the needs of working with new starch-producing varieties and wheat lineages. RVA generally uses 3 to 4 g with a 20 min run time and a heating rate of 14 °C/min (Perten Instruments), while the MicroViscoamilograf uses between 2 and 15 g with a heating rate of 1.5 °C/min to 10 °C/min (Brabender, 2014). All the methodologies are described in AACCI and ICC.

The viscosity analysis of crude starch and starch flour can provide three different cooking phases. Figure 2.2 shows the pasting profile of cereal (maize) and root (cassava) starches analyzed by RVA.

**Phase 1:** Heating (starting at 25 or 50 ºC to 95 ºC), informing the peak time and pasting temperature required for starch gelatinization. The highest viscosity measured at 95 ºC is called peak viscosity. The initial temperature (25 or 50°C) can be changed according to the type of information required, once pre-gelatinized starches have cold viscosity at 25ºC. This step also allows to assess the initial viscosity of modified starches, for example, acid and enzyme-treated starches, oxidized starches or cross-linked starches.

The peak viscosity is the maximum resistance of the starch granules, between the particle stiffness and swelling, with maximum interaction with water, without the degradation of the granules (Shelton and Lee, 2000; Cozzolino, 2016). Native starch granules do not absorb water at room temperature, thus there is no cold peak viscosity. However, with the increase in temperature and agitation, there is a rupture of the amylose and amylopectin structure, allowing water to enter and swelling the granule, which increases viscosity, due to amylose leaching (Huang and Rooney, 2001).

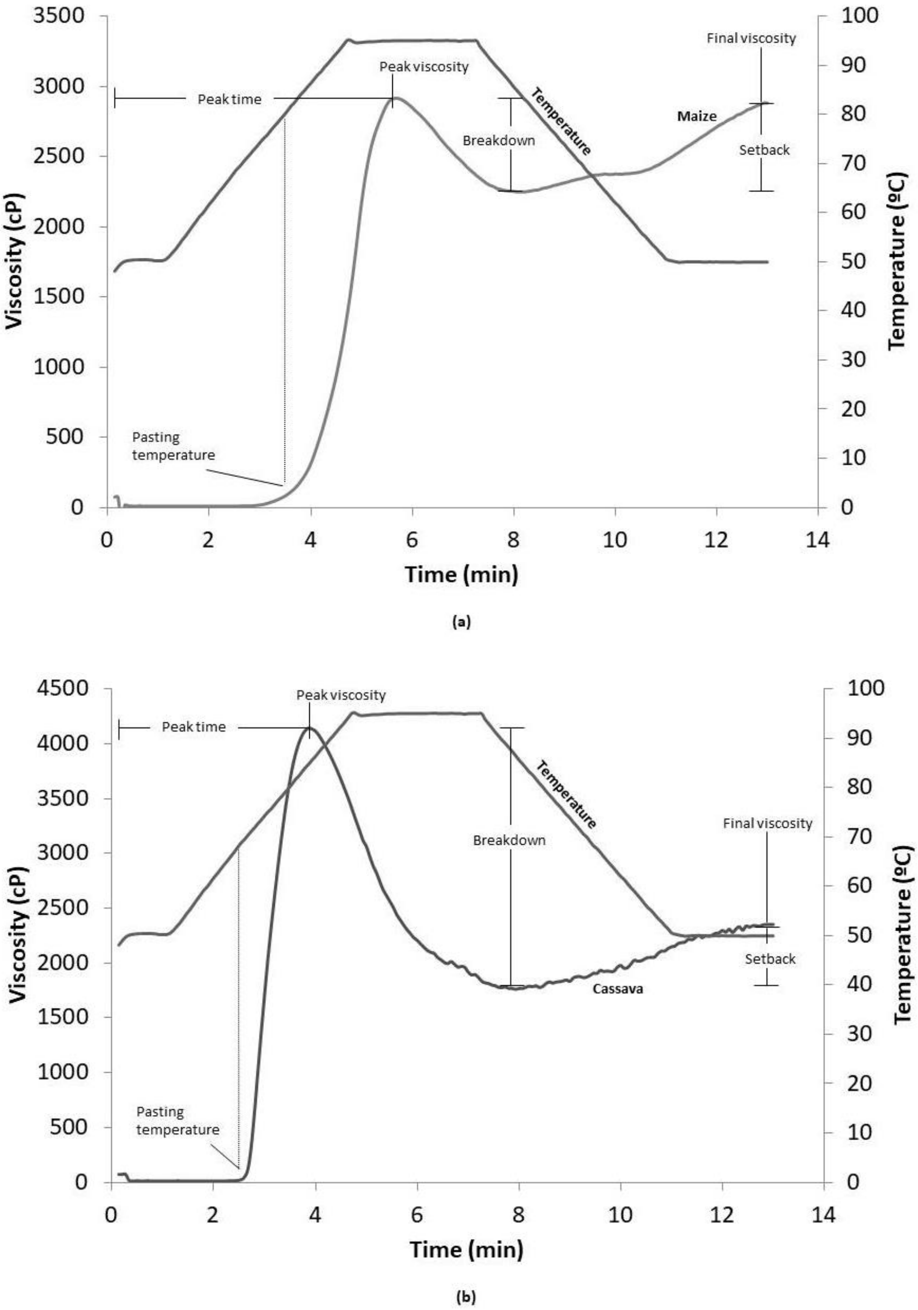


**Figure 2.2** Pasting profiles of (A) maize and (B) cassava starches by Rapid Visco Analyser.

The initial pasting temperature indicates the temperature of initial viscosity increase (Shelton and Lee, 2000; Cozollino, 2016). Thus, information about the viscosity during heating contributes to understanding the cooking processes involving starchy products, including soups, sauces, etc, as well as the equipment design, and the amount of water required for the process. The effect of temperature and agitation are fundamental for the industry to understand the rheological behavior of starch suspensions, which can generate changes in the velocity profiles in turbulent flow (Lagarrigue and Alvarez, 2001).

The pasting temperature is the most important parameter for studying the grinding, extraction, purification, and drying of starch. The pasting temperatures of root and tuber starches range from 50 to 60 °C, while cereal and legume starches exhibit a temperature above 60 °C for this parameter. Thus, starch should be isolated at temperatures below the pasting temperature in order to avoid physical changes and damages to the starch granule.

The time of initial viscosity increase is related to the cooking time of starch, i.e., the higher the initial pasting temperature, the longer the time. During hot gelatinization, the curve also indicates the time at which all starch has been gelatinized and is already in equilibrium with the aqueous medium.

**Phase 2:** At this step, the starch is heated to a constant temperature of 95 ° C, with a minimum viscosity and viscosity breakage.

The continuous heating and agitation of the system lead to the breaking of the granules with a consequent decrease in viscosity. The difference between peak viscosity and trough viscosity (minimum viscosity at 95 °C) provides the breakdown, which is related to the degree of disintegration of starch granules, ie, the starch paste stability (Shelton and Lee, 2000; Cozzolino, 2016).

**Phase 3:** Cooling process, which provides the final viscosity at 50 °C, retrogradation rate during cooling, and the tendency of retrogradation. Cooling begins after the starch paste reaches 95 °C. At 50 °C, there is a further increase in viscosity due to the tendency of retrogradation, with consequent reassociation of the amylose and amylopectin molecules, forming a gel. This measure is called the setback. The final viscosity refers to the ability of starch to form paste after heating and cooling (Shelton and Lee, 2000; Cozzolino, 2016).

This part of the curve is directly related to the hot filling starch products that are subjected to cooling in the pipes, and make the process difficult, which is a great challenge for the industry.

Cereal starches tend to form pastes with a higher retrogradation rate than those of tubers, due to differences in amylose and amylopectin contents, since amylose retrogrades rapidly while amylopectin needs a longer time to perform the rearrangement (Wang et al., 2015;

Mahmood et al., 2017). Amylose provides a more elastic and resistant gel when compared to amylopectin. A higher amylopectin concentration provides a softer gel, with low strength and good adhesiveness. Therefore, the lower availability of amylose to make hydrogen bonds can reduce the cohesion of the system (Wang et al., 2015). This phenomenon is perfectly visible in the cooling of soups, with the formation of coatings on the surface of soups containing cereal starches.

Phase 3 shows the starch behavior after cooling, i.e. in the final product, such as bread, cakes, puddings, soups among others. Starch with a high retrogradation rate, such as cereal starch, tends to form products with higher viscosity and rapid syneresis, which can impair the texture, appearance, and conservation of the product.

This effect is very common in gluten-free bread, once the characteristics of the final product depend on the starch properties, influencing the microstructure, rheology, water retention, storage, and product quality. To improve the quality of this product, different starch sources, modified starches, the addition of hydrocolloids or flour blends can be used (Witczak et al., 2016). Purhagen et al. (2012) used a commercial blend for a gluten-free bread with the addition of pregelatinized oats and barley and emulsifier and observed a reduction in amylopectin retrogradation evaluated by DSC, which interfered with the bread characteristics.

Native starch such as maize, cassava, and potato starches have a high peak viscosity, and potato starch has the highest values when compared to the others (Gunaratne and Corke, 2004) since according to Alexander (1995), it presents larger particle size, high-molecular-weight amylose, and presence of phosphate groups. Cereal starches generally have low peak viscosities and high setbacks, whereas waxy cereal starches have similar behavior to roots and tuber starches.

The amylose and amylopectin ratio and their distinct molecular structure have a great influence on paste properties. Ren (2017) investigated nineteen types of starch and found pasting temperature values ranging from 52 to 85 °C, with values of 83.10 °C, 64.45 °C, 70.90 °C and 72.65 °C for wheat starch, maize starch, cassava starch, and potato starch, respectively. Among these, peak viscosity was higher for cassava starch with 4.53 Pa×s followed by potato starch with 3.87 Pa×s. These differences are also due to crystallinity and particle size. Ren (2017) demonstrated a negative correlation between paste properties and peak viscosity, and a positive correlation with peak viscosity, through viscosity and breakdown, respectively, when analyzed by Pearson's correlation.

The presence of other components, as previously reported, can also affect the paste properties. The addition of hydrocolloids, such as xanthan gum, for example, may increase peak viscosity and final viscosity of starch, due to the interaction between amylose and this hydrocolloid (Hong et al., 2015).

Starch subjected to physical, enzymatic and/or chemical modifications, such as gelatinization, hydrolysis by amylases and/or acids, respectively, can present differences in cold solubility and initial pasting temperature when compared to native starch. Thus, the starch initial viscosity may indicate the degree of gelatinization and/or dextrinization, and can also serve to monitor the starch modification to the desired viscosity. Physical processes such as thermoplastic extrusion can lead to dextrinization, resulting in a cold peak viscosity (25 °C). This parameter indicates the ability of the material to absorb water at room temperature, thus allowing to evaluate the cooking degree by the starch gelatinization and dextrinization (Colonna et al., 1989).

### 2.5.2 Syneresis

The syneresis phenomenon corresponds to water exudation during amylose and amylopectin retrogradation during cooling (Doublier and Cuvelier, 2006) and storage of cooked starch, since temperatures of 4 °C favor maximal retrogradation (Wang et al., 2015).

To evaluate the syneresis, gel filtration is performed to measure the water content released (Radley, 1976), which is fundamental to study the stability of starch gels under freezing and thawing.

The quality loss by retrogradation of the starch gel can be evidenced by the syneresis or increased hardness, once the system is unstable and time and temperature dependent. Although after the gelatinization a crystalline structure can be formed during storage (Eliasson and Gudmundsson, 2006), it is different from the original structure. One of the practical applications of gelatinization and retrogradation events is the production of resistant starch type III, with an increase in resistance to enzymatic digestion using cooking and cooling cycles (Sievert and Pomeranz, 1989, 1990).

The syneresis is directly related to texture and conservation due to the increase in free water in the product, favoring microbial growth, thus various studies have been carried out from the use of ingredients and additives to new packaging systems. Some compounds may delay this phenomenon, such as proteins and lipids, by the interaction with amylose (Eliasson and Gudmundsson, 2006). To control retrogradation and syneresis in starch-based products such as bread, gluten-free bread, cakes and desserts, additives (Oliveira Filho and Mancim, 2009) and crumb softeners, modified starches (Cais-Sokolinska et al., 2006; Rodriguez-Sandoval et al., 2017), maltodextrins, and soluble fibers (Gomez et al., 2013, Huang et al., 2016, Miś et al., 2017) have been used as emulsifiers, among others.

## 2.6 Technological characterization

### 2.6.1 Swelling power and solubility

Water is an important component of the starch network, participating in various processes such as gelatinization, swelling, and dissolution. Starch granules subjected to heating in excess water can increase the volume, leading to a higher viscosity of the medium due to the rupture of the crystalline structure and formation of hydrogen bonds with the hydroxyl groups of amylose and amylopectin (Hoover, 2001).

The swelling power is generally measured by increasing the gel volume, starting at the initial pasting temperature (measured by DSC or by empirical rheology) and continuing with increasing temperature for a period of 5 to 10 minutes. The solubilized material can be determined by the reaction with iodine, microscopy (Eliasson and Gudmundsson, 2006) or gravimetry, as the water density increases with increasing the starch solubility.

Starch granules can be classified into four categories: high swelling (potato, cassava, waxy cereals, ionic starch derivatives), moderate swelling (native cereal), restricted swelling (cross-linked starch) and highly restricted swelling (> 55% amylose) (Gunaratne and Corke, 2004).

Several factors can affect the swelling power, including:

- Amylose and amylopectin ratio: higher amylopectin contents lead to increased swelling (Ross, 2012). For example, waxy rice starches subjected to high gelatinization temperatures show high swelling power at 80 °C, with low solubility in relation to waxy starch at low gelatinization temperatures (Tester and Morrison, 1990b).
- Formation of the amylose-lipid complex: it inhibits the swelling process and solubility (Tester and Morisson, 1990a).
- Occurrence in the granule: in some starch granules, such as potato and maize starch, the granules seems to swell to a similar degree in all directions, whilw wheat, rice, and barley starches swell in only one direction (Eliasson and Gudmundsson, 2006).

- Size: cassava starch, which presented larger granules had a greater swelling power (35.6 g/g) when compared to rice starch, which presented small granules (15.7 g/g) (Hamzah and Hill, 2010).
- Morphological structure: for example, potato starch has a poorly compacted structure and presents swelling power at room temperature, which is not observed for maize starch, which has a compact structure (Fonseca-Florido et al., 2017).
- Presence of other functional groups rather than hydroxyl groups: potato starch showed high swelling power and solubility when compared to root and tuber starches, such as cassava and yam, probably due to the presence of phosphate, which contributed to hydration due to the repulsion with amylopectin (Hoover, 2001).
- Starch modifications: changes in pH and physical modifications such as annealing processes (Adebowale and Lawal, 2002) and heat-moisture treatment (Hormdok and Noomhorm, 2007)

Tester and Morisson (1990a) evaluated the swelling power of native and waxy starches from wheat, barley, and corn, and observed that the onset of swelling was close to that of the initial gelatinization temperature ($T_o$) previously determined by DSC. A great exposure to high temperatures did not affect the swelling power, which increased with an increase in water: starch ratio (above 2.5: 1). Other factors that may contribute to the increase in swelling power and solubility include defatting and greater agitation during heating, since the amylose-lipid complex acts as an inhibitor of this process, not being leached, with dissociation only above 94 °C. A mild agitation can increase the swelling power, while a high agitation leads to the disintegration of the granules (Eliasson and Gudmundsson, 2006).

The swelling and solubility properties are of industrial importance as they provide important information on starch behavior in different food production systems. Factors such as the space occupied by starch, the loss of solids from suspensions or starch pastes and the fluid flow during the different unit operations, such as filtration and transport through the pipes must be taken into account, in addition to starch-containing products packaged before heat treatment, among others (Lapasin and Pricl, 1995).

Another important factor is the selection of the packaging material, once packaging with high permeability to water vapor, such as sacks, can lead to an increase in the specific volume of raw starch, including potato starch, in addition to reducing the shelf life of the product when stored in regions of high relative humidity.

### 2.6.2 Freeze-thaw stability

Knowledge about the starch stability during the freeze-thaw cycle is important to maintain the sensory quality of refrigerated and frozen products. This information will be useful for products direct for consumption or for those products containing gelatinized and retrograded starch that will be subjected to further processing operations. According to Vamadevan and Bertoft (2015), these cycles may lead to water exudation (syneresis) due to the reassociation of the starch molecules. Three methods can be used to verify the starch stability: measurement of the syneresis content, which is dependent on the centrifugal force applied to the sample; rheological characterization using a rheometer (Eliasson and Gudmundsson, 2006), and DSC analysis (Vamadevan and Bertoft, 2015).

Native starches present low freeze-thaw stability (Eliasson and Gudmundsson, 2006), with the exception of oat starch, which exhibits extensive retrogradation during the freeze-thaw cycles (Chotipratoom et al., 2015), thus chemical substitutions, genetic modifications, and use of hydrocolloids can reduce the quality loss of the starch gel (Vamadevan and Bertoft, 2015). Some authors have studied the starch stabilization, including:

- Use of hydrocolloids: Lee et al. (2002) evaluated the stability of sweet potato starch gels submitted to five freeze-thaw cycles, using several types of gum, and found that sodium alginate, guar gum, and xanthan gum were effective in reducing syneresis.
- Use of starch modification processes:
  - Ye et al. (2016) observed that extruded rice starch showed greater stability to freeze-thaw cycle than the native starch.
  - The use of high pressure hydrostatic technology combined with propylene oxide at different concentrations (4, 8, and 12% v/w) applied in maize starch led to better stability for the higher concentration (12%) at 400 MPa, which prevented adequate chain realignment, which could lead to retrogradation (Chotipratoom et al, 2015).

### 2.6.3 Other methodologies

Methods for measuring the gel strength, opacity, and clarity of the starch gel are also important for the starch characterization.

Paste clarity can be determined by the percentage transmittance from a dilute solution of starch (1% w/w) to a wavelength of 650 nm. Tuber starch forms clear pastes and has larger transmittance when compared to cereal starch, which forms less clear pastes (Craig et al., 1989). This analysis is important for confectionery using cereal starch as cake fillings. For toppings, such as fruit fillings, preference is given to tuber starch, once the pastes are clear and present lower retrogradation rates. The paste clarity can vary according to the origin of starch, type of modification, and storage time.

The gel strength or gel resistance is determined using a texture analyzer, in which the parameter peak force [N] defines the resistance of the three-dimensional network (Ulbrich et al., 2015). It has been used to evaluate the retrogradation and is very important for ready-to-eat starchy products since the starch gel must withstand storage and transport conditions.

## 2.7 Nutritional characterization

In its natural granular state (raw), starch degradation by enzymes is difficult; however, when cooked, it becomes an important source of energy in the diet, since it is hydrolyzed by amylases in maltose, dextrins, and maltotriose, which in turn will be hydrolyzed by the oligosaccharidases in glucose and then absorbed in the intestine (Jane, 2004). Although the amylases are found in both the saliva and intestine, the highest starch hydrolisys occurs in the intestine.

*In vivo* and/or *in vitro* studies can also be carried out with the use of enzymes to characterize starch from various sources. *In vitro* methods are preferable since they do not require ethics committee approvals, which is necessary for *in vivo* studies using humans and animals.

The classical method of assessing *in vivo* glycemia in humans, under fasting conditions or through the glycemic curve has been used for medical purposes. However, due to the different health conditions of the individuals, the evaluation of the starch digestibility in humans is limited to groups of research related to health area.

The methods used to evaluate the starch digestibility simulate a complete digestive process *in vitro*, in which starch is subjected to pH conditions and digestive enzymes that vary according to the human intestinal transit, thus simulating the gut environment. Starch is then classified according to its digestion rate and whether or not it is digested; the undigested starch is called resistant starch.

It is worth noting that resistant starch (RA) has a nutritional function similar to dietary fiber, thus the evaluation methods may be specific, such as those reported by Englyst et al. (1992), Goni et al. (1997), and AOAC (2006), or determined in food as dietary fiber.

The starch digestibility varies according to the interactions that occur during food processing, once there is a tendency to a lower digestion rate when complexed with lipids, fibers, proteins, which is determined by the glycemic curve for 120 minutes or more.

Although the methods *in vitro* do not reflect the reality of the digestive process *in vivo*, they provide preliminary information that guides the development of slow and resistant starch quickly and at a lower cost, besides the possible interactions with nutrients in the final product.

## 2.8 Biodegradation of starch-based packages

Packaging containing polymer compounds and edible starch-based films are evaluated for the biodegradation in the environment. Mergaet et al. (2000) and Accinelli et al. (2012) used the Lugol solution to evaluate the biodegradation of starch-based packaging and verified the blue zone clearing (starch-Lugol complex) over time since starch was used by the soil microbiota under study.

## 2.9 Future trends

With the greater concern about the preservation of the environment and the safety of laboratory analysts, the reduction or elimination of the use of chemical reagents has been widely discussed. Microscale analytical techniques have been used for those situations requiring the chemical reagents.

The use of green chemistry through non-destructive analytical techniques is an increasing demand, allowing the elimination of the generation of effluents (nontoxic or toxic), obtaining the results in a fast, safe and reliable way. However, these techniques require highly qualified technical-scientific knowledge, mainly concerning the interpretation of the results.

Other more recent starch evaluation techniques include the use of biosensors, crystallographic techniques, flow field-flow fractionation coupled to multi-angle light scattering, and refractive index detectors (FIFFF-MALS-RI), matrix-assisted laser desorption ionisation time-of-flight mass spectrometry (MALDI-TOF MS), sonic spray ionization (SSI), distributed expert systems (DES), among others.

## 2.10 Conclusion

Classical or modern techniques to evaluate starch granules, whether gravimetric, colorimetric, potentiometric, titrimetric or well-established instrumental or empirical methods are of fundamental importance to the food and non-food industries. The physicochemical, morphological, structural, rheological, and nutritional characterization allows identifying the starch properties such as plant origin, adulterations, behavior during food processing and sensory and nutritional properties.

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# CHAPTER IV

## PHYSICAL MODIFICATIONS OF STARCH

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This chapter presents a review of the main physical methods used to modify starches and their impacts on their technological properties

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# Physical modifications of starch

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## 6.1 Introduction

The use of starch in food products has increased, especially the reformulated products to improve the technological properties or products with reduction or absence of some ingredients, once starch has little impact on flavor, color, odor, and texture of the product, besides presenting important application as a body agent.

Knowledge about new kinds of physically modified starch has met the new consumer's demand, who looks for safe ingredients for consumption, obtained by environmentally friendly methods and that contribute to the trend of the clean label.

Bemiller (1997) has reported the reasons for increased investments in the development of physically modified starches. According to the author, hardly new chemical reagents or derivatives will be approved for use in chemical modification methods. In addition, the levels of the chemical reagents in the formulations should be reduced, for protection of both consumers and the environment, safety at work, and savings in production costs.

The main industrial reasons for modifying starches are based on their physicochemical properties and, from a nutritional point of view, the modifications have changed, once well-established processes for the production of rapidly digestible starches are giving rise to new methods of obtaining slowly digested and resistant starches.

The main reasons for the starch modification include:

- Raw starch is insoluble in cold water and can decant if kept in tanks without constant stirring. In addition, suspensions with high starch concentrations function as a non-Newtonian fluid, presenting dilating properties, which makes it difficult to pump through the pipes. This behavior is explained by Royer et al. (2016), who showed that these frictional contact forces and the hydrodynamic contributions fit accurately to the viscosity measured in a wide shear stress and particle size range. Currently, new uses for raw starch suspensions have been proposed, such as the use in bulletproof vests, due to the high capacity to absorb the impact energy and still be flexible during a movement like walking or running.

- When heated, starch-water mixtures do not form perfect pastes, which have high viscosity and cohesiveness, and exhibit retrogradation and syneresis after cooling. In case of freeze-thaw cycles, there is a weakening of the sponge-like paste.

This chapter presents the methods of physical modification of starch, reporting the conventional methods and pointing out new technologies, some still at experimental levels.

## 6.2 Conventional gelatinization processes

Pregelatinized starches are cold water soluble and have the ability to form a cold water paste (Colonna et al., 1984), and can be used in several formulations for thickening or water retention without heating, including puddings, instant milk mixes, and breakfast foods, and as an extender in the meat industry and in fruit pie filling, as they can increase the aroma retention (Powell, 1965).

In food, starch must be gelatinized to be digested by the amylolytic enzymes of the human digestive system and make it a source of energy. It can be subjected to home cooking or industrial food processing for the preparation of starchy foods, which has led to an increase of formulations containing dry and pre-cooked starches, aiming at rapid or instant preparation.

### 6.2.1 Conventional cooking

Conventional starch gelatinization methods are based on cooking under continuous stirring above the starch gelatinization temperature in a water starch ratio of 3:1 or more for more than 20 minutes, once there will be an increase in volume of the granule until breaking and exudation of the molecular components, which will affect the viscosity of the starch paste formed.

Cooking temperature, water content, agitation, and cooking time can vary according to the concentration and source of starch, gelatinization temperature, stirring speed, water volume used, time, and evaluation methods, and whether the starch is pure, in the form of flour or as a formulated product. Several classic publications such as Powell (1965), Leach (1965) and Greenwood (1976) have reported the characteristics of the process in more detail.

The gelatinized starch may be used hot, after cooling or after drying as a pregelatinized starch powder. In the industrial cooking process, heating equipment such as turbo-mixers, autoclaves, and heating tanks may be used, and the water removal can be performed using spray-drier, freeze-drying, drum-drying, flash-drying, drying oven, among others. The major challenges of these processes include high energy expenditure, time, temperature, and degree of agitation, control of the gelatinization and retrogradation of the starch paste. When starch is subjected to drying, the slurry of starch may exhibit low solids content (Leach, 1965, Greenwood, 1976).

The heat treatment of starch may be carried out for purposes other than gelatinization and changes in cold viscosity. Recent studies have focused on the starch retrogradation and the increase in regions resistant to the enzymatic digestion. Somes studies include:

- Many authors used high autoclaving temperature and long storing time, varying cooling time and cooling cycles to obtain resistant starch (RS). The authors showed that the factors that most affected the production of RS by autoclaving were temperature, moisture, number of cooking/cooling cycles and type of starch (Sievert and Pomeranz, 1989, 1990, Mangala et al., 1999, Teixeira et al., 1998).
- Tian et al. (2012) studied the dual-retrogradation treatment (gelatinization - retrogradation and gelatinization-retrogradation) for preparing slowly digestible starch (SDS) products from rice starch. The authors found 56% SDS in the 36 h interval between the gelatinization processes, which was higher than the starch obtained by single retrogradation, with 39.3% SDS. According to those authors, the dual-retrogradation treatment can increase the SDS yield, which is linearly related to the melting temperature range.

### 6.2.2 Cooking with the limitation of water

The main processes of high manufacturing efficiency of gelatinized and dried starch include drying rollers, using atomizers or thermoplastic extrusion. Although drying rollers are simple and commonly used, they have high product cost, difficult operation, constant maintenance, and adjustment of the rolls (Greenwood, 1976), besides affecting the quality of the final product, which may be heterogeneous.

The use of atomizers is economically limited, since cooked starch pastes have high viscosity, and the atomization systems operate at low solids contents (Chiang and Johnson, 1977). The most used process is the thermoplastic extrusion, considered a versatile process, with high production capacity and adjustable for all starch sources.

Historically, single-screw cooking extruders were developed in the 1940s to make puffed snacks from cereal flours or grits. An expanding demand for precooked cereals and starches required machines with larger capacity, so extruders with a nominal capacity of 5 ton per hour were developed in the 1960s, with numerous news applications: snacks, infant feeding, pet foods etc. In the 1970s, products containing more than one component, such as egg rolls and ravioli for co-extrusion were developed. Then, the use of two extruders in series, the first for cooking and the second for forming and structuring, resulted in several products. At the end of the 1970s, the twin-screw extruders for food processing were adopted, expanding the range of application (Mercier et al., 1989).

The thermoplastic extruders present temperature variations in the heating zones, and the single-screw type has a lower initial investment cost and maintenance when compared to the twin-screw extruders (Serna-Saldivar, 2008). Regardless of the type of extruder, the equipment is basically composed of a pre-conditioning system, a feeding system, screw or worm, barrel, die, and a cutting mechanism.

Inside the extruder, there are three different zones performing a series of specific functions, with different temperatures during the process: the former is responsible for mixing and transporting the raw material, the second confers high shear and cooking rate, and the third confers high pressure and has a higher temperature in relation to the other zones. The matrix at the end of the barrel provides back pressure and the mass expands to the final shape. Finally, the cutting system is located just after the die, which cuts the material into pieces (El-Dash, 1981, Mercier et al., 1989, Riaz, 2000, Steel et al., 2012).

During the thermoplastic extrusion, starch is converted into a viscoelastic fluid, which is forced out by a die, leading to a rapid expansion due to the difference in internal pressure between the equipment and the medium, with instantaneous water evaporation (El-Dash, 1981, Harper and Clark, 1979), which must undergo the drying and spraying process (Chiang and Johnson, 1977).

This technology involves several variables, thus the changes in both the composition of the raw material and the process will directly influence the quality of the final product. Many studies on the extrusion process have used the response surface methodology to make changes in the process. As reported by Yacu (1990), several independent variables can affect the process, including the composition of the ingredients, particle size, feeding, screw and die configuration, processing temperature, pressure, and the residence time of food in the extruder. The analyzed responses are called dependent variables and include the expansion, sensory characteristics, paste and thermal properties, water absorption index (WAI) and water solubility index (WSI) of extruded starch, fiber content, glycemic index, among others.

Starch will be gelatinized during the extrusion process at high temperature and high pressure, with a moisture content of 15 to 30%, which can also degrade to dextrinized starch. Thus, the water solubility index (WSI) and the water absorption index (WAI) will be indicative of the process conditions, once high temperature and pressure conditions and low moisture can lead to the formation of dextrinized starch, which increases WSI and reduces WAI (Mercier and Feillet, 1975, Yacu, 1990).

The gelatinization mechanisms were studied by Gopalakrishna and Jaluria (1991) in simulation studies on a single-screw extruder, who found that the changes in moisture levels provide an estimate of the extent of cooking, due to the moisture loss, besides the formation of starch hydrogen bonds with water. This phenomenon leads to the gelatinization and dextrinization of starch, initially by the decrease in free space along the thread of the extruder, leading to an increase in temperature and viscosity, thus promoting heating.

The versatility of the twin-screw extrusion cooking, which can operate in both low and high moisture and with various ranges of temperature, pressure, shear, speed, and numerous thread configurations, allows its use in the thermomechanical gelatinization and liquefaction of cereal starches and grains for the subsequent production of various starch-based syrups and ethanol (Linko, 1991).

The simultaneous use of the extrusion process and a chemical reagent to obtain modified starch has been studied for more than 30 years, such as the production of phosphate starch (Chang and Lii, 1992) and alcohol (Chang and El-Dash, 2003). These processes were advantageous due to the lower use of chemical reagents and the decrease in the volume of effluents, especially when the chemicals were safe. In addition, the reaction occurred in a short time, with processing at a lower moisture content and reduced drying time and temperature of the modified starch.

The stability of modified starches to the extrusion process was studied by Jaekel et al. (2015). The authors used hydroxypropylated cross-linked starch to study the behavior of starch against the thermoplastic extrusion. Traditionally, the cross-linked starch is obtained by presenting a greater resistance to the shear force under acidic conditions (low pH). The authors found a change in the technological properties of starch, resulting in a decrease in gelatinization temperature, peak viscosity, breakdown, setback, pasting temperature, and swelling power. In addition, an increase in the solubility of the extruded starch and the degree of gelatinization was observed.

Schmiele et al. (2016) have filed a patent using thermoplastic extrusion for the production of resistant starch type 5. RS 5 is usually obtained by debranching of amylopectin with isoamylase and pullulanase and subsequent complexation with saturated fatty acids (Hasjim et al., 2010, Hasjim et al., 2013). However, biotechnological processes with the use of enzymes are still a costly technology. In this context, thermoplastic extrusion presents an advantageous technology to replace the use of enzymes, due to higher shear rate during the process, leading to the dextrinization of starch, releasing linear molecules of lower molecular weight, which can complex with the fatty acids present in the sample. In addition, it is a process that does not present effluent generation (Steel et al., 2012).

Masatcioglu et al. (2017) studied the effect of thermoplastic extrusion on the production of resistant starch type 3 using high amylose maize starch as a raw material and the co-rotational twin-screw extruder. The authors concluded that the moisture conditioning at 60% combined with 2 extrusion cycles at 60 to 140 °C along the heating zones resulted in a 10% increase in resistant starch, and the high amylose starch presented 44% AR3.

Table 6.1 shows the isolated or combined use of starch, the use of reagents and the new tendency to produce resistant starches by the thermoplastic extrusion.

### 6.3 Microwave Cooking

The use of microwave cooking has increased in foods with a variety of purposes, including drying, enzymatic inactivation, moisture control in cookies, and pregelatinization of starch, due to its rapid cooking (Chang et al., 2010). The authors reported several studies showing that the gels formed by heating starch slurries by using microwave energy had significantly different properties than those heated by using conduction heat. The lack of granule swelling and the resulting soft gel are two key observations that highlight the differences in the two modes of heating. The significant differences in the other molecular properties, including enzyme susceptibility and amylopectin recrystallization, suggest a different mechanism of gelatinization during microwave heating.

Palav and Seetharaman (2007) studied maize starch subjected to microwave-heating and thermal conduction, with variations in moisture. The authors found differences in pasting properties, texture, and enzyme susceptibility, and explained that gelatinization differs from the conventional method, once during microwave heating the starch granules lose their birefringence much earlier than the gelatinization temperature due to the vibrational motion of the polar water molecules, with fast granule rupture and formation of film polymers, coating the granule surface. This event results in a soft gel even in the absence of a continuous network of amylose chains.

The use of microwave energy to obtain starches modified by annealing and heat/moisture treatments has been studied (Gupta and Kumar, 2003) which can also be used to produce chemically modified starches (Shogren and Biswas, 2006), with energy saving, shorter time, and reduced use of solvents and reagents.

Gonçalves et al. (2009) evaluated the effect of heat treatment at low moisture (25 to 35%) in sweet potato root starch using a conventional oven (90 °C/16 h) and microwave oven (35 to 90 °C/1 h). The conventional oven modified the starch properties, increasing the amylose content, with further reduction of the granule expansion factor and more pronounced modifications in the viscosity profiles and pasting properties when compared to the microwave oven treatment.

In chapter 7, Kong presents more details on the use of the microwave with a greater diversity of applications.

**Table 6.1** Recent physical modification by extrusion of starches and application to foods and feedstuff

| Raw material | Type of extruder | Die temperature (°C)/moisture conditioning (%) | Main results | Reference |
|---|---|---|---|---|
| Green banana flour | Co-rotating tween screw | 130/20 and 50 | Decrease in peak viscosity, water absorption and resistant starch content and increase in water solubility index and antioxidant capacity | Sarawong et al., 2014 |
| Sago and tapioca starch | Single screw | 160/20, 30 and 40 | Good floatability for mahseer aquafeed | Umar et al., 2013 |
| Potato starch | Single screw | 120/20 | Increase on hardness without affecting expansion index for fish aquafeed | Rodriguez-Miranda et al., 2012 |
| High amylose and normal maize flour | Co-rotating tween screw | 150/19 | Extruded high amylose starch showed lower hydrolysis extent and slower hydrolysis rate than normal maize flour | Zhang et al., 2016 |
| Cassava starch | Single screw | 70-130/18-26 | Intense pregelatinization of the hydroxypropylated cross-linked cassava starch, reducing peak viscosity and increasing the degree of gelatinization | Jaekel et al., 2015 |
| Pea and lentil starches | Co-rotating tween screw | 90/35 | Modified starch applied in noodles, promote firmer and less sticky and less bright in color for the end product | Wang et al., 2014 |
| Starches isolated from unripe banana (*Manguifera indica* L.) and mango (*Musa paradisiaca L.*) | Single screw | 90-150/15-40 | The resistant starch RS formation in the extruder for banana starch was affected positively by temperature and inversely by moisture. Moisture did not affect significantly RS formation in mango starch | Bello-Pérez et al., 2006 |

## 6.4 Heat moisture treatment (HMT)

Modified starch can be obtained by HMT processing at temperatures above the glass transition temperature (80 to 140 °C) of starch, combining low moisture (10-35%) and different periods of time (from 15 min to 16 h) (Biliaderis, 2009).

The HMT process presents varying results according to the process variables and the starch source, as examples:

- the lower the moisture used, the greater the changes in the surface of the starch granule, which may result in increased susceptibility to new chemical, physical, or enzymatic modifications (Gunaratne and Hoover, 2002);
- tuber and root starches are more sensitive to changes than cereal starches, as reported by Zavareze and Dias (2011) in a comparative study.

Several authors (Gunaratne and Hoover, 2002, Vermeylen et al., 2006) reported that the starch modification by HMT promoted changes in the crystallinity pattern of type B starches, resulting in type A crystals (evaluated by X-ray diffraction), which was not observed for the other starches. As reported by Genkina et al. (2004), type B starches have a hexagonal thermodynamic structure with about 36 water molecules inside every cell, while type A has a monoclinic structure with about 6 water molecules inside the helices.

Olayinka et al. (2008) reported that HMT starch gels showed a decrease in peak viscosity, breakdown, and setback when compared to native starch gels. Zavareze and Dias (2011) summarized the starch modifications by HMT including the reduction of swelling and solubility, production of gels with less firmness and greater opacity, probably due to the reorganization of the crystalline structure of the starch granule, the interaction between the amylose and amylopectin chains through hydrogen bonds in synergism with other bonds, and the amylose-lipid interactions. HMT starch has been widely used for infant foods and food products subjected to freeze-thaw cycles (Collado and Corke, 1999).

## 6.5 Cold water swelling (CWS)

According to Bemiller and Huber (2015), the starch modification by cold water swelling or cold water swellable (CWS) can be carried out using three main methods: (a) process 1, in which corn starch is heated in 75–90% ethanol to 150–175 °C (300–345 °F) for 0.5–2.0 h; (b) process 2, in which a starch slurry is very quickly heated in a special spray-drying nozzle and the droplets containing gelatinized granules are dried in a spray drier; and (c) process 3, in which a starch is treated with an alkaline, aqueous alcohol solution at 25–35◦C, as reported by Eastman and Moore (1984), Pitchon et al. (1991) and Jane and Seib (1991).

The initial CWS starch modification methods reported by Chiu and Solarek (2009) and Singh and Singh (2003) were:

- starch was placed in a mixture of water and an organic solvent, generally ethyl alcohol, in a proportion of 70-80%, heated in the range of 157 to 177 ° C, for 2 to 5 minutes (Chiu and Solarek, 2009); and
- starch was kept at room temperature in alkaline alcohol solution (water, alcohol, strong base). The alkaline solution may be composed of sodium hydroxide or potassium hydroxide at a concentration of about 2 to 3 moles / L (Singh and Singh, 2003).

Methods that concurrently use thermal and chemical gelatinization may be considered as mixed methods. Both methods for obtaining the modified starch require treatments above the atmospheric pressure. In view of this, a method was created at atmospheric pressure, wherein the starch granules are dispersed in water solution with polyhydric alcohol, heated in the range of 145 to 155 °C for about 15 minutes, which causes rearrangement of the granule structure, providing a cold solubility of 70-95% (Chiu and Solarek, 2009).

The final properties of the modified starch depend on several factors, including the plant source, the alcohol concentration, pH, and the reaction time. The starch modification is obtained by decreasing the dielectric constant of the medium, promoting the rupture of the crystalline structure of the starch, without disintegration of the starch granule. Thus, the starch suspension will exhibit substantial changes in physicochemical, morphological, thermal, and rheological properties, including an increase in hot viscosity, cold viscosity, higher processing tolerance, and smoother texture gels. In contrast, there is a decrease in amylose content. In addition, the modified starch granules have indentations and cracks, with changes in the granule morphology (Singh and Singh, 2003).

The starch modified by CWS has several advantages, including the dispersion in hot or cold water without formation of agglomerates, since it should have a cold viscosity, with the capacity to absorb water and gel at room temperature, presenting instant dispersion, heat tolerance, rapid development of viscosity, smooth and short texture, being used in foods of fast preparation.

**6.6 Annealing**

Annealing is a physical reorganization of starch granules, which promotes an increase in both the gelatinization temperature and enthalpy and a decrease in the temperature range at which this endothermic phenomenon occurs. The heat required for gelatinization of starch is inversely proportional to the area of the starch crystalline region, thus, it is a technique with greater viability for starches with larger amorphous regions. The modified starch can exhibit a decrease in swelling power and solubility (Tester and Debon, 2000, Biliaderis, 2009).

For annealing, it is necessary to heat the starch at a temperature higher than the glass transition temperature (Tg) and lower than the gelatinization temperature, that is, in the range of 40 to 55 °C, in the presence of moisture (about four times starch weight of water) for 12 hours or more. During the process, there is a reorganization of the semi-crystalline structure of the starch, resulting in greater stability of the granule as a function of the lower free energy on the molecule, and the interactions between the starch chains (Biliaderis, 2009, Zavareze and Dias, 2011). Tester et al. (2000) found that annealing can occur at up to 15 °C below the gelatinization temperature, but the closer the gelatinization temperature, the more efficient the process.

The higher interactions between the starch chains and the changes in the granule surface have been studied in relation to the digestibility of modified starches, as well as the formation of starches resistant to the enzymatic digestion. After annealing, starch presents greater resistance to digestibility as a result of the rearrangement of its crystalline structure, presenting higher levels of slowly digestible starch and resistant starch, and lower levels of rapidly digestible starch (Chung et al., 2009, Zavareze and Dias, 2011).

Many authors such as Lee and Osman (1991), Gomes et al. (2005), Liu et al. (2009) and Chiu and Solarek (2009) believe in a partial annealing during milling and extraction of starch (e.g. obtaining maize starch) in an aqueous medium. Once a slurry is formed in water with subsequent removal of the liquid phase, amylose can be leached, thereby altering the starch composition.

Table 6.2 shows a summary of studies on ANN and HMT modifications with different applications and changes in the technological characteristics.

**Table 6.2** Recent physical modification annealing (ANN) or heat moisture treatment (HMT) of starches

| Starch | Type of modification | Method - Solids concentration; Temperature; Time | Results | Reference |
|---|---|---|---|---|
| Potato, yam and wheat | ANN | 20%, w/v; 30 to 50°C; 24 h | Increase in gelatinization temperature for all starches; Wheat starch treated at 50ºC showed disruption of granule morphology; Yam and potato starch showed higher peak viscosity | Wang et al., 2017 |
| Buckwheat | ANN | 25%, w/v; 50°C; 24 h | Increase: amylose content, relative crystallinity, resistant starch content; Decrease: smooth surfaces, in vitro hydrolysis peak viscosity and retrogradation | Liu et al., 2015 |
| Potato | HMT | 76 to 88%, w/w;110°C; 1 h | Polymorphic change in X-ray B-type pattern to a mixture of A + B-types or totally A-type Higher moisture treatment results in: Increase in relative crystallinity and gelatinization temperature; Decrease in swelling power | Bartz, Zavareze and Dias, 2017 |
| *Pueraria lobata* (Willd.) Ohwi | ANN | 50%, w/v;50°C; 1 to 24 h | Slightly changes in crystal structure; Higher action in the amorphous regions of starch granules due the mobility and flexibility Increase: in resistant starch level | Zhu et al., 2018 |
| Canna | ANN | 10%, w/v; 50°C; 0 to 7 h | Swelling of amorphous regions and less compact structure observed by small-angle X-ray scattering analysis | Lan et al., 2016 |
| Acorn | HMT | 80%, w/w; 110°C; 24 h | Increase: in starch solubility and gelatinization temperature; Decrease: in swelling power amylose leaching, relative crystallinity, peak viscosity and retrogradation | Molavi, Razavi and Farhoosh, 2018 |
| Wheat | ANN | 10 to 50%, w/v; 40 and 50°C; 0.5 to 48 h | Higher moisture and time and lower temperature resulted in: Increase: in starch stability while storage; Decrease: in retrogradation degree; | Yu et al., 2016 |
| Acorn | ANN | 30%, w/v; 50°C; 20 h | Increase: in solubility and gelatinization temperature; Decrease: in on relative crystallinity, swelling power, peak viscosity and retrogradation | Molavi, Razavi and Farhoosh, 2018 |
| Buckwheat | HMT | 65 to 80%, w/w; 110°C; 16 h | Increase: amylose content, relative crystallinity, thermal stability and pasting temperature; Decrease in swelling power and solubility; Granules with less smooth surfaces | Liu et al., 2015 |
| Red adzuki bean | HMT | 70%, w/w; 120°C; 4 to 12 h | Increase: in starch hydrolysis and gelatinization temperature; Decrease: in resistant starch content, peak viscosity and retrogradation | Gong et al., 2017 |
| Breadfruit | HMT | 65 to 85%, w/w; 120°C; 4 h | Change in X-ray pattern from B-type to A-type Increase: in amylose content, thermal stability and pasting temperatures; Decrease: in viscosities and relative crystallinity | Tan et al., 2017 |

**6.7 High Hydrostatic Pressure (HHP)**

HHP has been used to promote changes in amylaceous sources through a combination of high pressures, dwell time, the botanical source of starch, crystallinity, and solids ratio (Kawai et al., 2007). Water enters the starch granule as a function of the high pressure (100 to 1000MPa, promoting the partial or total loss of birefringence, changing the granule shape and size, as reported by Leite et al. (2017) in pea starch. The process promotes the starch gelatinization, with low granule swelling, low viscosity and alteration in its physicochemical structure, such as the breaking of non-covalent bonds, mainly hydrogen bonds, which is responsible for the stabilization of the starch structure. Pea starch presents type C polymorphism, characterized by the overlap of type-A and B-type polymorphisms, thus greater changes are observed in the morphological structures and physicochemical characteristics due to the lower stability of B-type starch to the processing conditions (Pei-Ling et al., 2010).

For processing, the product is vacuum packed together with a determined amount of water or a mixture of water and alcohol, inserted into a cylindrical steel vessel, and pressure is generated indirectly, varying from 400-700 MPa with temperatures between 20 to 80 °C (Norton and Sun, 2008, Yang et al., 2017). Table 6.3 shows many applications of HHP for different starch sources.

**Table 6.3** Recent physical modification by high hydrostatic (HHP) pressure of starches or starchy flours

| Starch | Concentration | HHP conditions (pressure, temperature, time) | Results | Reference |
|---|---|---|---|---|
| Waxy corn | 30-50%, w/w | 300 to 600MPa, 22 to 25°C,30 min | Decrease: swelling power, gelatinization temperature and enthalpy<br>Increase: granule size | Pei-Ling et al., 2012 |
| Normal and waxy rice starch | 25%, w/w | 600MPa, 30°C, 30 min | Higher area of imperfect crystallites in relation to perfect crystallites and increase in slow digestibility, decrease in glucose release | Tian et al., 2014 |
| Waxy wheat starch | 10%, w/v | 300 to 600MPa, 20°C, 30 min | Increase: slow starch digestibility;<br>Decrease: relative crystallinity and in peak viscosity;<br>Gradually decrease in gelatinization enthalpy with higher pressure levels | Hu et al., 2017 |
| Waxy, normal and high-amylose corn | 40%, w/w | 100 to 600MPa, 25 to 35°C, 30 min | With higher amylose content was observed a lower lamellae thickness and higher granule swelling. Crystallinity X-ray pattern B-type and V-type were more resistant to HHP treatment in relation to A-type. Changes in starch morphology, lamellae and greater crystalline structures | Yang et al., 2016 |
| Potato | 25%, w/v | 400 and 600MPa, 21°C, 3 and 6 cycles of 10 minutes each | HHP associated with retrogradation provides a lower in vitro starch hydrolysis, slower glucose release and lower glycemic index | Colussi et al., 2018 |
| Quinoa | 10%, w/v | 100 to 600MPa, room temperature, 5 min | Increase in particle size;<br>Increase in swelling power and solubility at 55°C, while a decrease at 75°C for these parameters;<br>Total gelatinization at 600MPa | Li and Zhu, 2018 |
| Pea | 4%, w/w | 300 to 600MPa, 25°C, 15 min | Cold gelatinizatio due the loss of birefringence;<br>Total gelatinization at 500 and 600MPa;<br>Increase in retrogradation | Leite et al., 2017 |
| Mung bean | 20%, w/w | 120 to 600MPa, 25°C, 30 min | Increase in viscosity up to 480MPa, followed a decrease in viscosity at 600MPa, forming a weak gel and a high correlation between shear stress and shear rate data | Jiang et al., 2015 |
| Sorghum | 20%, w/v | 120 to 600MPa, room temperature, 20 min | Crystalline pattern changed from A-type to B-type at 600MPa, rough surface of granules;<br>Decrease: oil absorption capacity, swelling power, viscosity, relative crystallinity, rapidly starch digestibility<br>Increase: apparent amylose content, slow starch digestibility and resistant starch levels | Liu et al., 2016 |
| Red adzuki bean | 20%, w/w | 150 to 600MPa, 25°C, 15 min | Partial destruction of crystalline structure;<br>Increase in granule hydration;<br>At low pressure, the internal structure of starch granules were damaged firstly, at higher pressures, the proportion of damaged starch decreased;<br>Damaged starch granules could form intragranules–bonded forces under higher HHP level, complete gelatinization | Li et al., 2015 |

**6.8 Milling**

Milling can be used to alter the morphology, crystallinity, solubility and swelling power of starch granules. Even though this process is classified as a non-thermal modification, the changes promoted in the starch are consequences of the mechanical and thermal energy generated during milling, with an increase in temperature at the points of impact (Bemiller and Huber, 2015), as the starch undergoes action of various forces such as compression, impact, shear, and attrition (Karkalas et al., 1992).

The formation of damaged starch may be influenced by the following factors:

- Time:

✓ Morrison and Tester (1994) found a loss of shorter range crystalline order and double helix content in amylopectin in ball-milled wheat starches

✓ Tester (1997) reported that the native and waxy rice starch presented a damage of 30.3 and 36.0%, respectively, after 10 min of milling, with a damage increase of 73.6 and 76.5% by increasing the process time to 90 min. An increase in damaged starch was also observed in native maize starches (from 14.9 to 68.1%), waxy maize starch (from 23.8 to 85.3%), potato starch (from 38.2 to 94.3%), and pea starch (from 13.0 to 71.7%) subjected to similar processing conditions. The increase in damaged starch increased solubility and swelling, depending on the starch origin and process conditions.

✓ Chen et al. (2003) found that the ball milling time of rice starch causes reduction and disappearance of birefringence, X-ray diffraction peaks, and the endothermic event characteristic of gelatinization.

- Amylose to amylopectin ratio:

✓ Amylopectin is more susceptible to depolymerization than amylose (Bemiller and Huber, 2015). When comparing maize waxy starch with native starch, the former presented a higher rate of damaged granules, with the rheological properties being more affected, including the reduction of the apparent viscosity (Han et al., 2002).

- Water

✓ The addition of water in the system may have a plasticizing effect, reducing the breakage of starch granules. Shi et al. (2015) evaluated the physicochemical changes of maize starch with the addition of water (20 to 30%) in ball-milling. The starch granules remained largely intact, with oval-shaped or flat patterns. However, the broken granules formed larger particles in the presence of water. With respect to the pasting properties, an increase in paste temperature was observed, with a reduction in viscosity peak. The increase in paste temperature, in this case, may be due to the higher consumption of heat energy to solubilize the amorphous region, delaying the swelling of the granules.

✓ In relation to the quality of starch produced by milling, (Han et al., 2002) found that although it was possible to obtain damaged starches with lower molar mass in relation to the control, these starches were heterogeneous in quality and the process time was up to 60 min, indicating an increase in energy expenditure during the process.

✓ Dhital et al. (2011) studied commercial potato, maize, and two varieties of high-amylose corn starch subjected to cryo-milling, aimed to clarify whether the starch modification by milling can be considered mixed (thermal and physical) and whether the simultaneous action of heat and mechanical work can change the physicochemical properties of the damaged starch. The authors verified that the modified starch presented similar behavior in relation to the digestibility and other functional properties of starch subjected to ball-milling (Morrison et al., 1994), suggesting that changes in properties of ball-milled starch are predominately mechanically-induced rather than thermal.

Diop et al. (2012) studied the maize starch modifications by ball milling in ethanol medium. The authors observed that the increase in ethanol concentration increased the granule's susceptibility to physical damage due to the poor interaction between amylose and amylopectin chains. At low starch concentrations, the starch granules became more dispersed in the liquid, increasing the mobility in the system, allowing greater exposure of the particles during milling. In addition, ethanol can act as a damper, reducing the energy in the mill; however, at lower ethanol concentrations, the compacted sediment will reduce the process efficiency.

Dai et al. (2018) studied the physical and chemical modification of starch, who combined the use of sulfuric acid and milling to produce corn waxy starch nanoparticles, and found 19.3% yield, according to the process variables.

### 6.9 High-speed shear

Shahbazi et al. (2018) studied maize starch dispersions physically modified through a high-speed shear homogenizer with various shear-induced rates (56 e 400/s). The shear treatment produced a hydrogel with improved texture parameters and softer structure, and films with higher water resistance, tensile strength, and water barrier when compared to the native starch.

**6.10 Ultrasonic modification**

The ultrasound technique in food technology is considered as environmentally friendly and may be an alternative to various conventional processes, such as extraction, filtration, cooking, and depolymerization of polymers, such as starch. Its advantages include productivity, good yield, besides reducing the use of chemical reagents and heat, with shorter process time (Chemat et al., 2011), improving the quality of the products and reducing pathogens (Patist and Bates, 2008).

Ultrasound consists of mechanic waves of high frequency (> 20 kHz) that have the ability to traverse the medium (air, liquid, or solid), propagating in sinusoidal waves. The sound waves propagating in the medium will generate a vibration, which will lead to the displacement of the particles, causing an increase and a reduction of pressure and density. Therefore, the mechanical energy is the only type of energy imparted into the medium. The changes in the chemical and physical properties of the material are due to the cavitation phenomenon, which is dependent on the process parameters, such as frequency and power (Mcclements, 1995, Bermúdez-Aguirre et al., 2011).

The US technique can be classified according to the frequency and power as a) high-intensity ultrasound → processed at low frequency (20-100 kHz) and high power (>10W/cm$^2$); b) low intensity-high frequency ultrasound → processed at high frequency (20-200 MHz) and low power (<1W/cm$^2$). The main difference between these two conditions is that the first one aims at the modification of the material, while the second does not cause permanent changes in the material (Mcclements, 1995).

Basically, the ultrasonic equipment consists of an electrical generator, a transducer, and an emitter. Thus, it is assumed that the generator is responsible for supplying the energy to the system, the transducer is responsible for converting the electric energy into sound energy through the mechanical vibration, while the emitter radiates the waves in the medium (Bermúdez-Aguirre et al., 2011). Further details of the process can be seen in the studies of Povey and Mason (1998) and Kentish and Feng (2014).

The application of ultrasound in starch granules is carried out in a two-phase liquid-solid system, using water as the medium (Zhu, 2015), once water is a good bubble-forming fluid due to its low vapor pressure and viscosity (Bemiller and Huber, 2015). The mechanism of action is based on the emission of sound waves and the production of hydroxyl radicals during cavitation that damage and/or hydrolyze starch (Zhu, 2015), which can also cause erosion and formation of pores on the granule surface, leading to an increased water adsorption and swelling power (Bemiller and Huber, 2015).

The main effects of the ultrasonic process on starch properties include:

- Porosity

The porosity and particle diameter are related to the texture of foods, which can affect the technological and sensory properties of the products. The presence of pores may allow a larger reactive surface (Sujka, 2017), favoring a short processing time and lower amounts reagents used in the chemical modification.

The particle size of the starch granules influences the porosity, and the starches with larger granules are more susceptible to the exposure of the sound waves (Carmona-García et al., 2016), which was confirmed by Sujka (2017), who reported a higher porosity of potato starch granules when compared to wheat, maize, and rice starches during the ultrasonic process. In addition, the pore diameter may vary due to the use of other solvents such as alcohol and the frequency used in the process.

- Solubility and swelling power

Sujka and Jamroz (2013) observed an increase in solubility and swelling power in maize, rice, wheat, and potato starches, with more evident changes when using water instead of ethanol, with a proportional increase with the increase in the temperature range (60, 70, 80, and 90 °C).

As an example of association between chemical and physical modification, the researchers Amini and Razavi (2015) studied US in maize starch using acidic hydrolysis (3.16 and 4.5 M sulfuric acid), and observed an increase in solubility without damaging the crystalline structure and a tendency to reduce the swelling power with increasing temperature and exposure time.

- Paste clarity

Amini et al. (2015) studied the effect of different conditions of temperature, time, starch concentration and wave amplitude on the physical modification of maize starch and observed an increase in paste clarity with the reduction of enthalpy of gelatinization, without modification of the crystalline structure. The authors also found that damage of the starch surface may be influenced by process temperature, which was greater at low temperatures (25 and 55 °C) because high sonication temperatures reduce the energy transmission, resulting in a lower cavitation intensity.

- Paste properties

In general, the pasting viscosity is reduced due to depolymerization of the granule caused by cavitation, which induces the shear stress (Kang et al., 2016), and may have a higher depolymerization rate at high suspension temperatures (Ashokkumar, 2015, Bemiller and Huber, 2015).

Zuo et al. (2009) found a reduction of pasting viscosity when using a frequency of 211 kHz and power of 4.1 W in waxy rice starch, which exhibited lower peak viscosity and final viscosity values when compared to the control sample at a temperature close to the temperature of gelatinization ($T_{on}$).

Kang et al. (2016) observed that corn starch pastes with lower amylose concentrations presented less resistance to US treatment because of the small agglomerates. The disruption of these clusters leads to a reduction of the particle size, allowing a transition from the pseudoplastic behavior to a Newtonian fluid.

Hu et al. (2017) studied different frequencies (20 kHz, 25 kHz, and 20 + 25 kHz) under the same conditions of temperature (30 °C), power (400 W) and time (40 min), and observed a reduction in viscosity of 17.66, 18.87, and 19.61% for three frequencies used, respectively. However, they also observed an increase in the setback, indicating that the use of high frequencies may lead to starch instability.

- Digestibility

The effects of ultrasound on starch digestibility were also investigated by several authors. The reduction of the structure of the starch granules can lead to an increase in resistant starch content. Flores-Silva et al. (2017) studied native maize starch subjected to the ultrasonic treatment using a frequency of 24 kHz, and obtained an increase in resistant starch from 4.7 to 6.2% after 16 minutes of sonication, with no changes to rapid digestible starch (RDS) and slow digestible starch (SDS), due to the increase in crystallinity from 25% to 33% and a rearrangement of the amorphous region, making them more compact, which impairs the enzymatic hydrolysis, leading to a slow degradation of the amylopectin chains. When this sample was evaluated as a starch gel dispersion, the increase in RS varied from 2.1 to 4.0%, with an increase of RDS from 42.9 to 60%, due to the fragmentation of amylose and amylopectin that are more susceptible to the action of enzymes.

- Particle reduction (nanoparticles)

Boufi et al. (2018) observed a reduction of the particle size of the native and waxy maize starch granules suspended in water-isopropanol solution (50/50 wt%) from a micro to a nanoparticle scale, within 75 min of process at 25 °C in ultrasonic horn at 20 kHz and 400 W. The authors concluded that the system should contain a minimum of 50% water to aid in the disintegration of the granules. During the ultrasound treatment, the particle reduction by cavitation led to the progressive disintegration of the starch granule until the amylopectin was released and rearranged into 2D nanoparticles for the combined effect of the process with water.

Other examples can be seen in Chapter 7, reported by Kong, which deals with the unconventional starch modification methods.

## 6.11 Cold plasma

Plasma process can be distinguished into two main groups, i.e. high temperature (or fusion plasma) and low temperature (or gas discharges). In high-temperature plasma, all plasma species are in thermal equilibrium and generally at very high temperature, which is not recommended for the food industries. In general, a subdivision of cold plasma is made between low pressure and atmospheric pressure plasmas. Both these types of cold plasma have a wide range of applications in food processing. Cold plasma technology has drawn more attention in recent years, as it is chemical free, nontoxic, and environmental friendly (Thirumdas et al., 2017).

In cold plasma, plasma (ionized gas) can be generated by several processes (radio frequency, microwave, direct-current – DC - and pulsed DC), and the choice of process depends on the purpose of use. As examples, in the deposition of metallic thin films (by sputtering) and some ceramics (TiN, TaN etc) (by reactive sputtering), and also in polymer deposition (polymerization to plasma) starting from hydrocarbons and organometallic compounds, the plasma is generated by a glow in the precursor gas; discharge produced by continuous voltages (DC source). In contrast, in the deposition of insulating and semiconductor films, the gas is ionized (the glow is produced) by alternating, low (RF-KHz and MHz) or high-frequency (microwave-GHz) (Chapman, 1980, D'agostino, 1990).

Lii et al. (2002a) and Mozetič (2001) reported that the plasma treatment of polymers can cause physical changes in the surface of the material, besides the chemical modifications. The types of modifications depend on several factors, including:

a) energy and how it is generated: low-pressure, radio frequency, and microwave;

b) type of gas that will be excited to become ionized: $O_2$ has oxidizing properties; $H_2$ has reducing properties, $NH_3$ can promote the formation of amines on the surface of polymers. Other gases can also be used such as He, Ne, Ar, $CH_4$, and $CF_4$, etc.

c) water vapor once amounts below 5% can be beneficial for plasma reactions, which can affect plasma atomization and ionization above 5%.

Wongsagonsup et al. (2014) reported that the cold plasma can modify the starch by several ways, including the increase in surface energy, incorporation of functional groups, cross-linking, depolymerization, and change in hydrophilic nature.

When starch granules were subjected to cold plasma, an increase in acidity was found, mainly when starch was exposed to ozone and nitrous oxide in the plasma and mild dextrinization (Lii et al., 2002a, b, c). The authors concluded that plasma may be an alternative process for starch dextrinization, and the factors that most influenced the process were the botanical origin of starch, water content, the gas used, and time of exposure.

Zou et al. (2004) obtained plasma-modified starches using a soluble starch paste sample and Argon gas for 45 minutes. The authors verified the presence of cross-links between the starches, showing the viability of this process to promote cross-linking and oxidation reactions in starch.

Thirumdas et al. (2017) studied the effect of cold plasma treatment on the functional and rheological properties of rice starch using two different power levels (40 and 60 W), and reported depolymerization and changes in the starch properties, including an increase in gel hydration properties, syneresis, and turbidity, and a decrease in peak gelatinization temperature and pasting temperature. The SEM micrographs evidenced the formation of fissures on the granule surfaces.

- Lii et al. (2002c) found a higher content of reducing sugars in plasma-treated starch when compared to the original starch using the β-amylase enzyme. Plasma treatment consisted of placing 1 g starch in the plasma reactor for 30 minutes using ethylene gas and pressure of $10^{-7}$ torr ($1.33 \times 10^{-5}$Pa), achieving an increase in β-amylase digestibility of about 8%.
- Regarding starch modification in plasma, Lii et al. (2002b) found oxidation and a decrease in pH in plasma-treated starches. The authors used 1g sample and tested various gases, times from 10 to 30 minutes and agitation during the process.
- The patent JP63035604-A (starch modification) reported a decrease in starch viscosity after exposure to a gaseous plasma discharge. The authors of the patent analyzed the solubility and water absorption capacity of starch and found that the lower viscosity of treated starch resulted in an increase in water absorption due to the decrease in the crystalline area.

Clerici et al. (2011) obtained maize starches with different technological and physicochemical properties, such as WAI, WSI, paste viscosity, porosity, pH, acidity and enzyme digestibility by varying potency, time, and temperature conditions and gases in a cold plasma reactor by radio frequency (RF) using low pressure and vacuum. In this patent, the authors showed that the use of cold plasma can also produce resistant starches because starch can be of high digestibility or become partially resistant, as a function of the variables used in the process.

Observed hydrophobic coatings on starch films subjected to low-pressure plasma polymerization in 1-butene atmosphere. Similar morphological characteristics were observed for uncoated and plasma-coated films in AFM topographic images at low magnification, showing that roughness of uncoated and coated films exerts an important role in the evolution of contact angles with time, once more homogeneous surfaces showed a lower water absorption rate.

To increase the production scale of tapioca starch modified by cold plasma, Chaiwat et al. (2016) used a semi-continuous downer reactor designed to provide a production rate of 0.1–0.5 kg per cycle using argon gas. The authors observed modifications in the first treatment cycle, in which the paste clarity and breakdown viscosity decreased and the gel strength increased, in relation to the native starch. With increasing the number of cycles, the properties were different to the first cycle.

Other examples of the application of cold plasma can be seen in Chapter 7, described by Kong.

## 6.12 Other physical modification methods

### 6.12.1 Radio frequency (RF)

Zhang et al. (2018) studied a pilot-scale free running oscillator RF system heating performed by a 6KW, 27.12 MHz to modify potatoes and found that the starch granules were completely gelatinized when the RF heating reached 80 °C.

### 6.12.2 Pulsed electric fields (PEF)

Reported that PEF technology is a non-thermal or chemical free modification technique to improve the stability and function of biomacromolecules. The PEF-induced modifications follow two primary mechanisms, i.e. electrochemical reactions and polarization of the structural moieties. Critical PEF treatment intensity (EC) is required for the onset of the microstructure changes in biomacromolecules, which can be affected by the settings and configuration of the

PEF equipment, product characteristics (molecular weight, pH, conductivity), and temperature of the system. The main effects pointed out in the study were the increase in starch damage, loss of starch shape, and lower degree of crystallinity, due to the rearrangement and destruction of the molecular structure.

Han et al. (2009) studied corn starch–water suspensions (8.0%) subjected to PEF with different electric field strength up to 50 kV $cm^{-1}$. The authors found through SEM analysis that the modified starch exhibited dissociation and granule damage. The pasting viscosity analysis showed a decrease in peak, breakdown, and final viscosity with increasing the electric field strength due to the loss of crystalline structure (analyzed by X-ray diffraction), indicating a rearrangement and destruction of the starch molecular structure during the PEF treatment.

### 6.12.3 Freezing/thawing process (FT)

Multiple FT treatments have been shown to alter the structural and functional properties of wheat starch. When the starch suspensions were frozen, a pressure was developed due to a phase transformation from water to ice crystals. The freezing pressure induced irreversible changes in granule integrity, thus leaching amylose, proteins, and lipids. The freezing/thawing treatment resulted in the reorganization of double helices, which increased the relative crystallinity of starch, gelatinization temperatures, and changes in pasting behavior. The increase in damaged starch and the modified granule surface of the freezing-treated starch was likely to be a result of a high level of in vitro digestibility, with an increase in RD and SDS and a decrease in RS content (Tao et al., 2015).

Tao et al. (2016) studied FT to modify wheat starch after reconstitution with gluten for the production of bread. The authors found that the modified starch led to an increase in water absorption and a decrease in water available for dough formation, and an increase in bread firmness during storage.

Wang et al. (2017) studied FT in the pre-treatment of starch to increase the reaction efficiency of starch and octenyl succinic anhydride (OSA). The results were not promising, due to the decrease in the reaction efficiency and degree of substitution, as observed by the authors.

Szymońska et al. (2003) reported that FT (total of 5 cycles) in potato starch modified the granule surface and led to a weakening of double helices, with an irreversible disruption of the crystalline order within the potato starch granule. The first cycle (from 1 to 5) promoted the most pronounced changes in starch properties, such as gel formation, retrogradation, water solubility and the water holding capacity.

Vernon-Carter et al. (2016) investigated the repeated application of freeze (-20 °C)-thaw (90 °C) cycles (FT) on the properties of cornstarch gels (5% w/w), The increased number of FT cycles led to more compact and firmer starch gels as reflected by the increased storage and loss moduli in rheological tests and reduced the hydrolysis and the in vitro digestibility of the modified starch gels.

### 6.12.4 Gamma and Electron Beam Irradiation

Bhat and Karim (2009) investigated the impact of radiation processing on starch granules. According to those authors, the radiation processing via gamma irradiation involves the use of a radioactive isotope, either in the form of cobalt-60 or cesium-137, which emits high-energy gamma rays or photons capable of intruding in-depth into the target product, up to several feet. In the case of electron beam irradiation, the technology uses a stream of high-energy electrons generated from machine sources (such as linear accelerators, microtron). In a compilation of studies by other authors, they observed a chemical and/or enzyme modification of starch with greater intensity, using gamma and electron beam, ultraviolet light (UV), and x-ray radiation. In addition, the combination of irradiation with other physical treatments like X-rays, infrared rays, laser irradiation, and ultrasound can be studied to improve the cross-linking.

As can be seen in Chapter 7, Kong has reported the gamma and electron beam irradiation with a greater diversity of applications.

### 6.12.5 Combination of physical methods

Pinto et al. (2015), Andrade et al. (2005) and Giteru et al. (2018) studied the modification of *pinhão* starch by annealing (ANN), heat-moisture (HMT) or sonication (SNT) treatments, using a combination of these treatments. Native and single ANN-treated *pinhão* starch presented a CA-type crystalline structure, while the other modifications promoted the migration of crystalline structure from CA to A-type. The relative crystallinity decreased in starches subjected to HMT and SNT alone, as well as HMT–SNT and SNT–HMT starches. The ANN, HMT, and SNT did not provide visible cracks or grooves to *pinhão* starch granules. SNT-treated starch presented the highest breakdown, which indicates a weaker physical structure of the granules, making them more susceptible to collapse when subjected to thermal and shear treatments. In general, HMT in a single or dual modification had a stronger effect on the gelatinization temperatures and enthalpy when compared to ANN and SNT.

### 6.12.6 Production of starch nanoparticles

In food products, the starch nanoparticles can be used as a food additive such as emulsion stabilizer, fat replacer, thickener, or rheology modifier (Kim et al., 2015, Kaur et al., 2016). In packaging, they can promote the improvement in mechanical and physical properties, such as permeability to water vapor (Le Corre et al., 2010).

Le Corre et al. (2010), Le Corre and Angellier-Coussy (2014), Kim et al. (2015) and Kaur et al. (2016) reported various methods for the production of nanostarch, using chemical, physical, and enzymatic methods. Concerning the physical methods, the high-pressure homogenization, ultrasonication, reactive extrusion (combination of a physical method and use of chemical reagents), and gamma irradiation stood out, in addition to other methods that combine enzymatic treatments and physical methods.

Le Corre and Angellier-Coussy (2014) reported that the increasing scientific and industrial interest for starch nanoparticles (SNP) has led to the development of numerous methods for preparing sub-micron starch fillers for nanocomposite applications. Several authors have shown that the method may be promising; however, it is necessary to optimize the production process and to develop continuous processes and techniques that facilitate its extraction as an aqueous suspension.

## 6.13 Future perspectives

The physically modified starches have been subjected to instrumental methods of analysis, which can improve the knowledge about the physicochemical, morphological, structural, and technological properties of starch granules.

In addition to studies aimed at obtaining new technological properties, some authors have studied the digestibility of modified starches, and found mainly an increase in the slowly digestible fraction, resulting in a lower glycemic index and modified starches with healthy properties. These studies tend to increase since slowly digestible starches and resistant starches can aid in the control of chronic noncommunicable diseases such as obesity, diabetes, and others.

Physical methods, still experimental and promising, in a laboratory or discontinuous system, can become continuous and large-scale production methods if they are successful for food application.

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# CHAPTER V

## BAMBOO FIBER APPLICATION FOR FOOD AND FEED

This chapter presents a review of the characteristics of young bamboo shoots and culms and their applications for food and feed

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(Annex 4)

# Bamboo fiber application for food and feed

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## 1. Introduction

Bamboo is a valuable natural resource that many people living in producing areas depend on for their livelihood and energy source. China has the most bamboo resources, whose forests cover a total area of 6.8 million hectares, and whose industry directly employed nearly 10 million people; and moved in 2019 more than 2 billion dollars in exports, according to China Bamboo Industry Association (1).

Although Latin America has the greatest diversity of bamboo species in the world, there is no data on its economic contribution, as it has been employed in the informal trade of handcrafted products (decoration and food items), agriculture and civil construction (2).

Bamboo is a giant grass that belongs to the subfamily *Bambusoideae* of *Poaceae*. This grass has more than 1250 species, and its main advantages are its fast growth and distribution across continents, such as Africa, Asia and Latin -America. Usually, a bamboo clump reaches maturity between 3 and 5 years (3,4). After this period, bamboo shoots and mature culms are partially removed. This practice can extend its life for over 100 years, without the need for replanting, provide a large annual yield per area unit, allow it to be a source of food, and, therefore, a good investment for the economic adversities that impact the populations of the mentioned continents.

Bamboo has an organized production chain with investments, dissemination, and valorization of its products. Many studies and processes are carried out, ranging from industrial to regional use by small farmers. This chapter presents the potential of bamboo for food and feed, especially in this severe economic moment due to the Covid-19 pandemic, aiming to enhance the visibility of the bamboo's potential for other continents.

## 2. Nutritional aspects of bamboo

Promoting healthier eating habits remains a challenge, as it goes through processes that require social changes and economic investments allied to high scientific dissemination. For example, comparing the number of information disclosed in the Google Academics database of bamboo **(Table 1.1)** with grasses already commercialized, such as wheat, corn, and rice, we observe that the investments are 3 to 5 times higher for the large one's commodities. This shows the need for scientific and financial investments to promote the bamboo culture.

**Table 1.1** - Papers available on Google Academic on April 28$^{th}$, 2021.

| Keyword | Total citations | Food | Feed |
|---|---|---|---|
| Bamboo | 986.000 | 403.000 | 169.000 |
| Bamboo shoots | 92.800 | - | - |
| Bamboo young culm | 7.960 | - | - |
| Wheat | 3.600.000 | 3.150.000 | 2.180.000 |
| Rice | 4.080.000 | 3.350.000 | 2.360.000 |
| Corn | 4.780.000 | 3.500.000 | 2.590.000 |

The starch content of wheat, rice and corn **(Table 1.2)** was fundamental for the development of humanity since, over time, physical work required a caloric diet. For this reason, the milling industry was being developed and updated to improve starch cooking, making it more available in the digestive process.

In comparison with other nutrients (proteins and fats), starch became our primary source of energy, and for economic reasons, it became present in all of our meals. The consequence of all this was that industrialization and changes in professions, from the 20th century, with the greater use of mental activity for management, development of machines and systems, led to a significant reduction in physical activities, corroborating the increase in sedentary lifestyles. As a result, the WHO issued a public health warning regarding the high prevalence of obesity and diabetes in the population. Physical activity and a change in diet can be a way to prevent these diseases (5).

**Table 1.2** – Physicochemical composition (g.100g$^{-1}$) of young bamboo culm flour, fresh bamboo shoot and commercial wheat, corn, and rice flours

| Samples | Moisture | Protein | Lipids | Ash | Dietary fiber | Total Carbohydrates | References |
|---|---|---|---|---|---|---|---|
| Young bamboo culm flour | 3.2-8.2 | 1.3-2.0 | 0.1-0.9 | 0.8-2.8 | 64-90 | 89-94 | (6) |
| Fresh bamboo shoot | 84-93 | 2.3-3.7 | 0.2-0.6 | 0.6-1.4 | 2.3-4.5 | - | (7) |
| Refined Wheat flour | 13 | 9.8 | 1.4 | 0.8 | 2.3 | 75.1 | (8) |
| Yellow corn flour | 11.5 | 7.2 | 1.9 | 0.5 | 4.7 | 78.9 | (8) |
| White Rice | 13.2 | 7.2 | 0.3 | 0.5 | 1.6 | 78.8 | (8) |

Similar human's problems have appeared in pets, which also started to be raised in small spaces, with limitations in physical activities and a high-calorie diet. This fact clearly shows that a family's eating habits can even affect the health of pets, not only in a beneficial way.

A wide range of weight-control products is available in the current market to regulate satiety and blood glucose. Many of these products have different types of fibers in their composition, which benefits depend on the type of fiber and type of animal directed: carnivores, herbivores and/or omnivores. In addition, there are different types of digestive systems, which for humans and birds are monogastric, while for ruminants, they are polygastric. Therefore, the use of fiber in the diet should always be evaluated to achieve the desired benefits.

For humans, dietary fiber is defined by the AACCI (9) as "edible parts of plants or carbohydrate analogues that are resistant to digestion and absorption by the human small intestine with complete or partial fermentation in the large intestine". Besides the aspects involving the health benefits of fiber consumption (reduction in LDL cholesterol levels, blood pressure, increased satiety, encouraging the growth of intestinal microflora, among others) (10,11), the food industry can take advantage of its physicochemical properties to improve viscosity, texture, sensory aspects, and shelf life of the product, as reviewed by Elleuch et al (12). For polygastric animals, cellulolytic bacteria digest the fiber to provide the necessary energy, requiring nitrogen for good bacterial growth.

Bamboo shoots and young culm flour are sources of dietary fiber **(Table 1.2)** and have different applications in food. Shoots can be consumed in cooked, roasted or dehydrated form, as an ingredient in salads, soups, and meat accompaniment **(Figure 1.1)** (13), while young culm flour (not industrially commercialized yet) has been investigated and successful used in cookies (14) and pasta (15).

**Figure 1.1 –** Bamboo shoot as ingredient in different types of foods

In animal feed, we always remember of panda, an omnivorous animal. Pandas consumes 10 to 40 kg of bamboo shoots, culms and leaves, to take advantage of the nutrients present and complete its diet since the meat in their diet does not reach 1% of their nutritional requirement **(Figure 1.2)**. Furthermore, pandas do not have a digestive system capable of digesting cellulose, and consequently, 50% of the bamboo consumed is excreted.

## 2.1 Properties of bamboo shoot

Bamboo shoots are the initial part of the culm in formation and vary in flavor, weight, yield and color, according to the species and time of year. For human consumption, bamboo shoots can be processed into slices, small pieces or even whole through different methods, such as fermentation, roasting, boiling, blanching, canning, dehydrated, fried, among others (16).

Previous treatments for detoxification of bamboo shoots are crucial to allow correct and safe consumption. Cyanogenic glycosides are compounds used for plant protection against insects and microorganisms. When hydrolyzed, release hydrogen cyanide, also known as hydrocyanic acid, which is toxic to humans and animals (17). Satya et al. (18), an extensive review, showed that bamboo shoots have a wide concentration range of cyanogenic glycosides, linamarin and taxiphyllin (110 to 8000 ppm), according to the species and the position analyzed in the shoot (base, middle and top). Cooking, fermentation, drying, and other processes, reduce these concentrations, making them suitable for consumption. They also reported a decrease in cyanogenic compounds as shoots turns into culms, since these are a form of protection against insects and microorganisms.

Conservation of bamboo is still challenging due to its high moisture (90%) and sugar contents that lead to the fermentation process. For this reason, transport and processing must begin immediately after harvesting. One of the proposals that have emerged is the dehydration of the shoots to extend their stability.

The health benefits of bamboo shoots have been evidenced by prebiotic fibers, which modulates the gut microbiota and improves host metabolism. Some studies showed anti-obesity effects of insoluble bamboo shoot fiber by identifying higher amounts of lipids in the feces of rats fed this fiber compared to other fibers in a high-fat diet (13).

## 2.2 Properties of bamboo culm

Bamboo culm is an aerial culm with distinct nodes and internodes **(Figure 1.2)**, usually hollow, that provide support for the branches, leaves and reproductive structures of the plant. One of the anatomical characteristics of bamboo culm refers to the presence of parenchymal cells responsible for storing and mobilizing the compounds necessary for the growth of the shoot, especially starch. Starch is a challenge for culm conservation after harvest, as this compound is of interest to fungi and insects, especially *Dinoderus minutus* (9).


**Figure 1.2 –** Panda (A), *Phyllostachys edulis bicolor* (B), *Gigantochloa atroviolacea* (C), and *Indosasa hispida* cv. *rainbow* (D)

Toledo et al. (19) determined the chemical composition of *Guadua flabellata* culm and reported 27% starch, 16% pentosans and 7.8% soluble sugars. Recent studies by our research group (6,20–22) have shown that young culms from *Dendrocalamus asper*, *Bambusa vulgaris* and *B. tuldoides* can generate new food ingredients for use in several products, and with potential capacity to be produced on an industrial scale. This raw material can be harvested from clumps, between 2 months and 3 years old, with significant contents of fiber, starch and sugars. Also, according to Felisberto et al. (21), xylans in the fibers contribute to their prebiotic potential.

The young bamboo culm flour is obtained after removing the culms from the clump, followed by cutting into smaller portions, and removing the outer layer that covers the culm, since it has silicified cells, and, therefore, it is not edible (9). Afterwards, the material must undergo a drying process and successive grindings to reduce the particle size. The flour obtained by Felisberto et al. (6) also presented a low detection limit of cyanogenic compounds (< 5 ppm), proving to be safe for consumption (20).

Chemical characterization **(Table 1.2)** of young culm flours from *D. asper*, *B. vulgaris* and *B. tuldoides*, showed a great variation, suggesting that more studies should be carried out to expand knowledge. Depending on culm age, the flour can be used as a source of fibers and also for the extraction of starch and fibers (6). Regarding technological properties, the flours had a slightly acidic pH and light-yellow color (6), demonstrating an advantage over whole grain flours, which generally lead to darker, rancid-tasting products, and the possibility of consumer rejection.

## 3. Food applications

Despite some applications of bamboo fibers by the food industry, their scientific study is still scarce. Below, we provide some applications of these fibers in food products found in the literature. However, as the study for the use of young bamboo culm is recent, the examples indicated in this section are mostly from bamboo shoot fiber, being called here just as bamboo fiber.

### 3.1 Bakery products with gluten

Most studies in bakery were carried out with the association between wheat and bamboo flour, where it was possible to observe that bamboo fiber affected the formation of the gluten network. Bamboo fiber competed for the water in the system and directly influenced the final volume of bread, crumb firmness and also affected the flavor of the final product.

Pessanha (23) used 3% and 5% bamboo fiber to replace wheat flour in bread formulation partially. He observed an increase in stability time to mixing and water absorption, but a smaller volume of bread, as a consequence of the increase in consistency and resistance of the dough to extension. Thus, the bread samples added with bamboo fiber had an average of 7 points (liked moderately) on the hedonic scale of a sensory acceptance test carried out with 90 tasters.

In the same way, as observed for bread, fiber granulometry also influenced the dough of cookies. Mancebo et al. (24) noted an increase in sugar-snap cookie dough consistency, compared to the control, when using 350 µm bamboo fiber. With this particle size, the dough of cookies presented a greater capacity for absorbing and retaining water, as well as for absorbing oil, compared to the fiber with a particle size of 50 µm.

Choudhury et al. (25), when adding bamboo shoot powder to biscuits, observed a product browning, reduced in diameter and thickness, increased in hardness, and lower product acceptance with an additional content above 10% of fiber. Similar results were also observed by Farris et al. (26) in Amaretti-type cookies.

Felisberto et al. (14) founded that replacement of 25% to 50% of sugars and/or fats in cookies with young bamboo culm flour, preserved the texture properties of the final product. Reaching 50% of fat replacement allowed an increase in the spreadability of the dough during baking and a more significant reduction in the energy value. It was also noted, in general, an increase in the height of cookies from 5 to 9 mm, but with no undesirable changes in color, moisture or water activity content over the 30-day storage.

**3.2 Gluten-free bakery products**

Gluten-free products can benefit from bamboo flour, becoming more healthier for the consumers. Zardo et al. (27) evaluated the addition of bamboo fiber in gluten-free bread, at concentrations of 2, 4 and 6%. After 24 h of baking, an increase in hardness and a reduction in chewing was observed compared to the control. However, when comparing only the formulations containing bamboo fiber, no significant differences were found. On the other hand, Zhang et al. (28) verified an improvement of frozen dough with 1, 1.5 and 2% of bamboo fiber after thawing, with a better distribution of water in the dough and thermal stability.

Parameters such as fiber morphology and particle size must also be verified, as these characteristics may directly influence the dough rheology as identified by Martínez, Díaz and Gómez (29). They observed an increase in consistency and elasticity, with more irregular structures in the dough, when using bamboo fibers with 350 µm particles. On the other hand, fiber with 50 µm reduced hardness and increased specific volumes. These differences happened because the fibers of large granulometry remained almost unaltered in the mass, with more rupture points and less gas retention.

**3.3 Dairy products**

Dairy products are another food category widely produced and consumed. Milk is used as raw material for several products, such as yogurt, cream, dessert, butter and ice cream. The enrichment of these products with dietary fibers is an effective way to enhance nutritional and functionality aspects(30) by influencing rheological and thermal properties of the final products (31).

Staffolo et al. (32) studied the impacts of adding 1.3% dietary fiber (apple, bamboo, inulin and wheat) to yogurt formulation based on whole milk. The yogurt with the addition of bamboo fiber remained stable without syneresis and color change for more than 21 days of storage. The consistency index and apparent viscosity were similar to the control, but the maximum compression force increased with time. However, this increases favored product acceptance because, according to the authors, consumers prefer firmer consistency yogurt.
Subsequently, the authors evaluated the use of 1.3% of the same dietary fiber (apple, bamboo, inulin, and wheat) plus psyllium fiber in low-calorie dairy desserts. The products were prepared with prior addition of carrageenan and corn starch, which according to the authors (33), combined with bamboo fiber, allowed a balance about the water lost from the product by reducing the levels of fat and sugar. Thus, there was no statistically significant difference for syneresis between the fiber-added formulations compared to the control.

Zheng et al. (34), when studying the effects of adding bamboo fiber to milk pudding, found an increase in hardness, viscosity and gumminess with the addition of up to 2% of the fiber. They observed that the structure of the product became more compact due to the increase in particle aggregation favored by the addition of bamboo fiber. For substitution levels above 2%, an inverse behavior was observed, with milk pudding flocculation. According to the authors, these behaviors result from the generation of a continuous phase, with a strong interaction of the fiber with the water present in the system, whose structure was damaged upon reaching the 2.5% level of fiber addition.

Other studies have been carried out emphasizing dairy products containing probiotics and their supplementation with prebiotic fibers to improve the stability of bacteria during storage. With this objective, Akalin et al. (35), added bamboo fiber and several others in probiotic ice cream to investigate the impacts on technological, sensory and viability characteristics of probiotics for 180 days. The addition of bamboo fiber increased acidity, viscosity and consistency, and influenced the product hardness after 30 days. Compared to the control, the addition of bamboo fiber benefited the reduction of melted weight during storage and did not negatively influence the taste-flavor. Technological aspects would be correlated with the water retention capacity of the fibers, change in the freezing point of the product, which is affected by its chemical composition and fiber hardness. Considering the Lactobacillus acidophilus viability parameter of ≥ 7 log cfu.g$^{-1}$ during storage, the addition of fiber did not favor this property.

### 3.4 Meat products

Meat products present a high protein content that plays an important role in the human diet. However, these products are generally deficient in complex carbohydrates, and therefore, the addition of dietary fiber represents a strong trend in the sector (36). From a technological point of view, fibers can improve the quality of meat products by increasing water retention, acting as binders and/or fat replacer, contributing to the stability, and reducing cooking losses or purge in vacuum packages (37).

Li et al. (38), when adding bamboo fiber to pork bater, at the level of up to 4%, noticed a better water distribution, gel, and rheological properties. Fiber acted as a filler and binder, contributing to the formation of a stronger gel network.

Park & Kim (39) investigated the quality of fried pork loin batter with wheat, oat and bamboo fiber, and they observed that the insertion of 3% bamboo fiber promoted an increase in water content and a fat reduction compared to the control. According to the authors, the addition of fibers also provided less oil absorption in the fried batter coating during the deep-frying process. The final product also presented a stable shape during frying, leading to higher yield due to the increase in viscosity promoted by the insertion of the fiber.

Huber et al. (40) applied a mix of fibers (bamboo, wheat and pea fibers) as fat replacer (chicken skin), at different concentrations in chicken burger. Most of the products presented lower hardness, elastic resistance, and chewiness than the control, while the adhesiveness was similar. The products were well accepted in the sensory analysis, and the formulation containing the fiber combination of 0.4% bamboo, 1.6% wheat and 1.6% pea, was the most accepted.

Magalhães et al. (41) evaluated the effect of adding 2.5 and 5% bamboo fiber in reduced salt and sodium tripolyphosphate-free Bologna sausage to develop a healthier product. There was an improvement in emulsion stability, increased brightness and hardness with fiber insertion. Upon reaching 5%, these parameters were reinforced, and the structure was more compact with a more heterogeneous product surface. As for sensory characteristics, the products were described as mild aroma, unflavored, pale, opaque, dry, and gritty.

In the meat products market, another concern that is present is related to the unsustainable production and consumption of meat. Therefore, the industry and researchers are studying alternatives to develop plant-based meat analogues that meet the characteristics expected by consumers in terms of nutrition, flavor, and texture (42). To develop this type of product, in addition to the type of protein, it is necessary to think about the other ingredients that will constitute this new food matrix to provide properties like meat. Fibers can contribute to the formation of this new structure; however, there is still a lack of data in the literature about the action of bamboo fibers in meat analogues.

### 3.5 Other products

Other works evaluated the insertion of fibers from bamboo in pasta, sauces, fruit jellies, beer and in Pickering emulsion, whose work will be presented below. Ferreira (15) found that the pasta with 1.75% bamboo shoot fiber and 3.5% young bamboo culm fiber had the best technological characteristics, whereas another pasta with 1.75% of each fiber. The cooked pasta presented desirable technological characteristics of texture and mass increase, with low loss of solids and no major changes in the color of the pasta, compared with traditional pastas, without the addition of fibers. In the sensory analysis, the pasta received acceptance scores, between 6 (liked slightly) and 7 (liked moderately) points on the hedonic scale, demonstrating its significant acceptance among the tasters. In a deeper investigation on the characteristics and quality of these pasta by near infrared (NIR) spectral, Badaró et al. (43,44) managed not only to identify the type of fiber present, but also to quantify and evaluate its form of distribution in the final product.

Szafrańska & Sołowiej (45) evaluated different fiber sources (acacia gum, bamboo, citrus and potato) at concentrations ranging from 1% to 4% in acid casein processed cheese sauces. The addition of bamboo fiber led to an increase in hardness and a reduction in the stickiness and viscosity of the sauce, as the fiber concentration increased. According to the authors, fiber chemical composition and its ability to interact with water were responsible for these results. As for the sensory aspects, these sauces were described as too bright, even white, due to the fiber color. In addition, sauces with 2 and 3% bamboo fiber were more accepted in flavor than the other fibers evaluated.

Figueroa & Genovese (46), when developing fruit jelly with the addition of 3% of four types of dietary fiber (apple, psyllium, bamboo, and wheat), observed, in this order, a reinforcing effect on the gel strength. Regarding the jelly containing bamboo fiber, this reached consistency stability after one day of production, and remained stable until the thirtieth day of storage. According to the authors, insoluble fiber acted as a filler with a reinforcing effect in the gel matrix, but with fewer interactions in the pectin network, even if these interactions were strong. Syneresis also increased with bamboo fiber, and a “floury” mouthfeel was also observed. However, these technological and sensory aspects were eliminated by combining bamboo and psyllium (1:1). This combination ends up allowing, according to the authors, an increase in insoluble fibers in the final product without loss of quality.

Paulino (47) developed an ale-type beer using young bamboo culm flour from *D. asper* (0.85%) as a clarifier for the brewer's must. The bamboo flour was chemically modified with citric acid to interact with the proteins from the must. The modified flour adjusted the pH of the musts to the ideal and increased the turbidity of the beer, which, according to the author, may be due to the partial gelatinization of the starch in the boiling stage. The clarifying effect of bamboo flour was in the fermentation stage. Although the beer has a lower clarity than the control, flour did not change the other sensory aspects, even receiving statistically equal scores to the control.

He at al. (48) elaborated solid particles from bamboo fibers as stabilizers in Pickering emulsions, replacing proteins. The suspension containing the bamboo fiber particles showed two behaviors according to the applied shear rate. At a low shear rate, the particles were dispersed, while at a high shear rate, the particles were broken down, with a consequent reduction in viscosity. The authors also observed that increasing zeta potential of these particles improved emulsion stability due to the electrostatic repulsion existing between the particles; and that a higher concentration of bamboo fiber particles (0.3%), allowed the formation of smaller and more uniform droplet sizes, thus reinforcing the three-dimensional network structure. Furthermore, the emulsions showed excellent storage stability, not being influenced by temperature, pH or iron strength.

Through the studies cited, it is noted fibers from bamboo have different functionalities in their use. Depending on the type of product, they can act as a structuring ingredient, emulsifier or even substitute for other nutrients and act together with the ingredients to achieve both a technological and nutritional improvement.

## 4 Feed applications

As shown in **Table 1.3**, there are few published studies on the use of bamboo in animal feed. Although not reported, it is believed that much of it is used by local communities. Bamboo charcoal is a product that can be obtained by dry distillation of thick mature culms, which is then powdered. It has been used as an additive in animal feed formulation because of its high ammonia and nitrogen absorption power, promoting the elimination of poisons and impurities that are present in the gastrointestinal tract of animals or in their environment, through less nitrogen excretion, in the case of fish, as reported by Quaiyum et al. (42).

Chu et al. (52) fed 144 pigs for 42 days with a diet based on bamboo charcoal or bamboo vinegar, in addition to the control diet, and they observed increased growth performance, feed efficiency and fecal beneficial microflora, while decreased noxious gas emissions and fecal harmful microflora in fattening pigs. Bamboo vinegar may be obtained from the fermentation of sugars present in the bamboo. It is a brown-red transparent liquid produced as a by-product during pyrolysis of bamboo charcoal. It contains more than 200 types of chemical components, in which acetic acid is the main component. Acetic acid and phenolic compound contents show fungicidal, termiticidal and antibiotics properties in animal production, and improve the growth performance of animals, when added to the feed (52,54).

Bamboo leaf is a traditional folk medicine in East Asian countries, including China, Japan and Korea, whose flavonoid composition has led to bamboo leaf extract. It showed a positive effect on broiler carcass performance and the meat quality of heat-stressed broilers, and to reduce the incidence of diarrhea in weaned pigs (55,56). Additionally, in a preliminary study Li et al. (57) observed that the use of bamboo extract in the diet of non-heat stressed cows improved lactation performance, antioxidant capacity and immunity, among others. When evaluating the effect for heat-stressed cows, of bamboo leaf extract as a dietary supplement, Li et al. (58) observed an increase in milk production and milk fat content, besides improved rumen acetate concentration, and performance of cows. They also observed that the addition of this extract in the diet reduce the risk of mastitis during periods of heat stress, since the extract play an inhibitory action on DNA and RNA synthesis, and consequently inhibiting the growth of *Staphylococcus aureus,* the first cause of cow mastitis. Bamboo leaf extract has proven to be a good strategy for relieving the deleterious effects of stressed dairy cows, whether they are stressed from the heat or not.

The interesting point is that only leaf extracts present soluble fibers (when compared to the culm), and therefore there is a promising field for the use of bamboo fiber in animal feed. Its study should be encouraged since both shoots as young culms, on a dry basis, fiber content above 80%, which can be considered as fiber concentrates.

**Table 1.3** - Potential application of bamboo in animal feed.

| Product | Animal specie | Highlights | Reference |
|---|---|---|---|
| Bamboo charcoal (BC) | *Pangasius hypophthalmus* | • Reduction in ammonia concentration, with increasing BC in the diet.<br>• It was observed on histological sections that the height and area of the villi in all intestinal segments tended to increase with the increase of BC supplementation in the diet. | (51) |
| Bamboo charcoal (BC) and vinegar (BV) | *Fattening pigs* | • Better growth performance, immune responses and faecal microflora populations were observed with reasonable inclusion of BC or BV in the pig diet. | (52) |
| Bamboo charcoal (BC) and vinegar (BV) | *Laying hens* | • Better performance in egg production in the final feeding stage and lower rate of damaged eggs when supplemented with BV and/or BV/BC combination.<br>• Reduction in abdominal fat in laying hens with BC-included diet. | (53) |
| Bamboo vinegar (BV) | *Weaned piglets* | • Impact on the fecal bacterial community of piglets supplemented with BV in the diet.<br>• Antimicrobial action of BV, included in the feed, resulted in better performance of the piglets.<br>• Additive potential of BV in animal production as an antibiotic alternative. | (54) |
| Bamboo leaf extracts (BLE) | *Heat-stressed broilers* | • Positive impact on the growth and slaughter yield of broilers, with the dosage of 1.6 g/(kg.d) of bamboo-leaf-flavonoid on diets | (55) |
| Bamboo leaf extracts (BLE) | *Weaned piglets* | • Improve the growth performance with an increase the average daily feed intake, average daily gain, and reduce feed/gain and diarrhea rate. | (56) |
| Bamboo leaf extracts (BLE) | *Non-heat stressed cows* | • Improve lactation performance, antioxidant capacity and immunity. | (57) |
| Bamboo leaf extracts (BLE) | *Dairy cows* | • BLE supplementation increased milk yield, milk fat content and rumen total volatile fatty acid concentrations (acetate, butyrate and valerate), in addition to reducing somatic cells ($p < 0.01$). | (58) |

## 5 Future trends

Even though it is an ancient grass, scientific research focused on the food use of bamboo are still few. Nevertheless, we believe that with the new food challenges, to increase healthiness and environmental sustainability, we will have a greater incentive to consume and use of bamboo fibers in food, mainly in the countries where it finds a favorable climate and soil for its growth.

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# CHAPTER VI

## TECHNOLOGICAL AND PREBIOTIC ASPECTS OF YOUNG BAMBOO CULM FLOUR COMBINED WITH RICE FLOUR TO PRODUCE HEALTHY EXTRUDED PRODUCTS

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This chapter presents results from the process of obtaining flour from young bamboo culms and its impacts on the technological properties in rice-based extrudates

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**Technological and prebiotic aspects of young bamboo culm flour (*Dendrocalamus latiflorus*) combined with rice flour to produce healthy extruded products**

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**Abstract**

Young bamboo culm flour (YBCF) has proved to be a healthy and sustainable ingredient, due to its high fiber content and high yield of bamboo crops. The present study evaluated the effects of YBCF from *Dendrocalamus latiflorus* on the physicochemical, technological properties and prebiotic activity of rice-based extrudates aiming to expand its application. The extrudates were produced in a twin-screw extruder with different RF:YBCF concentrations (100:0; 95:5, 90:10, and 85:15%). During the process, the specific mechanical energy increased as YBCF content increased because of the high shear favored by YBCF particles. With increasing RF replacement by YBCF, the extruded products presented a significant ($p<0.05$, by the Scott-Knott test) increase in hardness (57.37 to 82.01 N) and water solubility index (12.80 to 34.10%), as well as a decrease in color luminosity (L*=85.49 to 82.83), expansion index (2.68 to 1.99), and pasting properties. In addition, all extrudate samples presented bifidogenic activity. Therefore, YBCF exhibited attractive technological and functional properties and can be a potential ingredient to be used in extruded products.

**Keywords:** bamboo, dietary fiber, rice flour, prebiotic, particle size, extrusion cooking

## 1. Introduction

Asian countries, in 2017, got around US$60 million from bamboo, with applications ranging from civil construction to food processing (INBAR, 2017). In contrast, although Latin American and African countries produce many bamboo species, the economic policies are not organized, and bamboo is often linked to the informal economy. Also, promoting the sustainable use of bamboo in food products has been a challenge. For example, bamboo shoots consumption is still not well established worldwide, despite many studies have shown the health benefits and prebiotic potential of its fibers (Li, Guo, Ji, & Zang, 2016; Wu, Hu, Gao, Chen, Fang, Mu, & Han, 2020; Chen, Chen, Yang, Yu, Wei, & Ding, 2019; He, Wang, Zhang, Wang, Sun, Wang et al., 2016).

To improve bamboo consumption practices, our research group produced and analyzed young bamboo culm flour (YBCF) of three species, *Dendrocalamus asper, Bambusa tuldoides* and *Bambusa vulgaris*, with commercial potential (Felisberto, Beraldo, & Clerici, 2017), aiming to identify it as a new sustainable source of starch, dietary fiber (Felisberto, Miyake, Beraldo, & Clerici, 2017), and xylans (Felisberto, Beraldo, Sentone, Klosterhoff, Clerici, & Cordeiro, 2021), and also to provide an alternative to the bamboo shoot sustainability, since for the clump maintenance, only 20-30% of the shoots can be annually removed (Pereira & Beraldo, 2016). The YBCF used as an ingredient in cookies and pasta increased the fiber content (Badaró, Morimitsu, Ferreira, Clerici, & Barbin, 2019) while also acting as a fat and sugar substitute (Felisberto, Miyake, Beraldo, Fukushima, Leoni, & Clerici, 2019) without affecting the color and flavor of the products.

Now our team is moving forward to produce and study the YBCF from *Dendrocalamus latiflorus*, that has potential for food application. This specie naturally present in China and Twain can achieve 25 m of height and 20 cm of diameter, being frequently used for the production of bamboo shoots, since the Asian communities appreciate their taste. Our purpose is to use YBCF as a new fiber source in extruded products, in addition to the conventional whole grains, that effect negatively the texture, flavor, and color of the products, as reported in several studies (Meza, Sinnecker, 1Schmiele, Massaretto, Chang, & Marquez, 2019; Oliveira, Schmiele, & Steel, 2017; and Mkandawire, Weier, Weller, Jackson, & Rose, 2015).

Rice (Oryza sativa L.) is a widely used raw material for cereal-based extruded products. Likewise, it is considered one of the most important crops and carbohydrates sources in the world (FAO, 2021), mainly consumed as cooked polished rice grains. Meanwhile, broken rice, presents similar nutritional properties to polished rice, with a mild flavor and neutral color, but has low dietary fiber content and low economic value in the market. In industry, the broken rice grains obtained from processing are used to produce gluten-free flour used in bakery products (Qian & Zhang, 2013) and as a base for extruded products.

Consumption of breakfast cereals and snacks containing fiber to increase satiety has been a trend since, according to WHO (2021), obesity triggers heart disease, diabetes, and hypertension, moreover, it is among the chronic diseases in the risk group for patients with Covid-19. Therefore, using YBCF can be a viable alternative for inclusion in dietary patterns and improving rice product quality.

This study aimed to obtain, to produce, and to characterize the properties of YBCF from *D. latiflorus* and also to evaluate its application in rice extruded products and its effects on the technological and functional properties of the final product.

## 2. Materials and methods

### 2.1 Raw materials

Rice flour (RF) was supplied by SL Alimentos (*Mauá da Serra*-PR, Brazil). Young bamboo culms (*Dendrocalamus latiflorus*) were harvested at *Fazenda de Bambus*, in *Pardinho*-SP, Brazil, in 1st April 2019. Fig. 1 shows the young bamboo culm flour (YBCF) processing: the culms were harvested at approximately 4 months of age, 4 m high, and 15 cm in diameter, and processed following the procedure described by Felisberto et al. (2017) but without dividing the culms into bottom, middle, and top fractions. The culms were subjected to a first milling step in a wood chipper (Nicolletti-SP, Brazil) and two milling cycles in a knife mill using a sieve with a 2 mm aperture.


**Fig. 1.** Young bamboo culm flour processing

## 2.2 Physicochemical and technological properties of raw materials

### *2.2.1 Morphological characteristics*

The raw materials and extrudates were visualized, when convenient, by an unaided eye and in an optical microscopy (OM) (Olympus, model BX51/BX52) under polarized light (10x magnification) to identify starch, fiber, and the shape of the particles. For scanning electron microscopy (SEM) analysis (TM 4000 Plus, HITACHI), the raw materials and extrudates (cross-section pieces and powders) were attached to a circular stub with double-sided carbon tape, removing the excess of the sample. The observations were performed at 10 kV, low vacuum, and 100 or 600x magnification.

### *2.2.2 Proximate composition*

RF was analyzed according to the official methods of AACCI (2010) (moisture, method 44-15.02; ash, method 08-01.01; protein, method 46-13.01 using conversion factor = 5.95; ether extract, method 30-25.01; total dietary fiber, method 32-07.01; and total starch, method 76.13-01); and YBCF was analyzed according to the official methods of AOAC (1998) (moisture, method 925.09; ash, method 923.03; protein, method 960.52 using conversion factor = 6.25; lipids, method 920.39; total dietary fiber, method 992.16; and total starch, method 996-11). The total sugars content was also determined for the raw materials by the Lane-Eynon (method 31.034-6, AOAC, 1984). Carbohydrates were calculated by difference. The results were expressed in g/100 g, on a dry basis.

### *2.2.3 pH and titratable acidity*

The pH and titratable acidity of the raw materials and extrudates were determined according to methods 02.52-01 and 02.31-01 of AACCI (2010), respectively.

### *2.2.4 Particle size and fineness modulus*

The particle size of the raw materials was determined according to method n. 66.20-01 (AACCI, 2010) using a Produtest vibrator with 14, 20, 32, 60, 80, and 100 mesh sieves opening sizes, representing 1,410, 841, 500, 250, 180, and 150 μm, respectively. This analysis was performed in triplicate. The fineness modulus was calculated by the ratio of cumulative percentages of material retained on sieves divided by 100.

*2.2.5 Bulk density*

The bulk density of the raw materials was determined according to Onuma, Okezie, & Bello (1988). For that, 10.0 g of sample was placed in a 100 mL graduated cylinder and manually packed with 30 strokes on a flat rubberized base. The analysis was performed in triplicate, and the density (g/mL) was calculated by the ratio between mass and volume.

*2.2.6 Instrumental color*

The color of the raw materials and extrudates were measured using a MiniScan HunterLab CR-400 colorimeter (Konica Minolta Sensing Americas), according to the CIELab system, for the parameters L*, a*, and b*. The L* parameter represents the brightness of the sample, with values between 0 (totally black) and 100 (totally white), and the coordinates a* and b* represent the color itself, ranging from red (+a*) to green (- a*) and from yellow (+b*) to blue (-b*), respectively. Illuminant D65 10º was used, and readings were taken in triplicate. L*, a*, and b* values were converted to RGB using Nix Color Sensor program.

*2.2.7 Hydration properties*

The swelling capacity of the raw materials was determined according to Rosell, Santos, & Collar (2009). For that, 1.5 g of sample was mixed with 30 mL of distilled water in a Falcon tube (50 mL). The sample remained for 16 h under hydration at room temperature. The swelling capacity was expressed as the difference between the final and initial volume divided by the initial sample weight. The water absorption (WAI) and water solubility (WSI) indexes of raw materials and extrudates were determined according to Anderson, Conway, & Peplinski (1970). Briefly, 2.5 g of sample was placed in a 50 mL Falcon tube, and 30 mL of distilled water was added. After 30 min of intermittent shaking, the samples were centrifuged (3000 × g/10 min). A 10 mL aliquot of the supernatant was removed for evaporation in an oven (105 ºC/4 h), and the centrifuge residue was weighed. The analysis was performed in triplicate, and the results were expressed on a dry basis.

**2.3 Extrusion cooking process of rice flour and rice-young bamboo flours**

Four samples were prepared using different proportions of RF:YBCF, 100:0 (control flour), 95:5, 90:10, and 85:15%. Distilled water was slowly added to the blends (2.5 kg) to reach 18% moisture and mixed for 10 minutes. The blends were then transferred to plastic bags and stored at 7 ºC for 24 h to ensure moisture uniformity. The formulations were extruded in a co-rotating twin-screw extruder (Werner & Pfleidere ZSK 30, Ramsey, USA) of 29 L/D (length/diameter ratio) (Fig. S1 - Supplementary material). The temperatures of the four zones were programmed for 65, 85, 110, and 130 ºC, and screw speed (250 rpm), circular die diameter (4.8 mm), and feed rate (11.7 kg/h) were fixed. After processing, the extrudates were dried in an oven with forced air circulation at 50°C until reaching moisture content lower than 10%. The products were packaged in plastic bags and stored at a controlled temperature of 21 ºC. They were identified as RB0, RB5, RB10, and RB15, according to the YBCF%. During the extrusion process, torque was recorded for each sample at least five times and the specific mechanical energy (SME) was calculated according to Pansawat, Jangchud, Jangchud, Wuttijumnong, Saalia, Eitenmiller et al. (2008).

$$SME\ (W.h/kg)\ =\frac{SS\ (rpm)\times P\ (W)\times T\ (\%)}{SS_{max}\ (rpm)\times Q\ (kg/h)\ \times 100} \tag{1}$$

where SME is specific mechanical energy; SS, screw speed (rpm); $SS_{max}$, maximum screw speed (500 rpm); P, the power rating of the extruder (9,000 W); T, average torque recorded over sampling time (%); and Q, the mass flow rate (kg/h).

## 2.4 Physicochemical and technological properties of the extrudates and their respective flours

### *2.4.1 Expansion index (EI) and bulk density (BD)*

Ten random samples of the extrudates from each trial were measured for diameter, using a caliper (accuracy of 0.05 mm), and weight. EI was determined by the ratio between the mean diameter of the sample and the diameter of the extruder die (Faubion & Hoseney, 1982). BD was obtained from the mean diameter of the samples and the mean weight of each extrudate, and calculated according to Equation (2) (Fan, Mitchell, & Blanshard, 1996).

$$\rho\ (g/cm^3) = \frac{4\ x\ m}{\pi\ x\ D^2\ x\ L} \tag{2}$$

*Where: $\rho$ is bulk density (BD); m is the mass of the extrudate (g); $\pi$ = 3.1416; D is the diameter (mm); and L is the standard length at 50 mm.

*2.4.2 Texture profile*

The cutting test of the extrudates was determined using a texture analyzer (TA-XT2i, Stable Micro Systems Ltd., Godalming, Surrey, UK) with a Warner-Bratzler shear blade in a "V" shape, using 1 mm/s pre-test speed, 2 mm/s test speed, 10 mm/s post-test speed, 20 mm probe distance, and 0.05 N force. Cutting was performed perpendicular to the main axis of the extrudates, previously standardized at 5 cm in length. The results represented the average of 10 repetitions, and the force to cut was the peak force (N) achieved during the test, representing the hardness.

*2.4.3 Cross-section image analysis*

The image evaluation of the extrudates was carried out according to Oliveira, Rosell, & Steel (2015). The cross-section images were captured using a scanner equipped with the HP PreciseScan version Pro 3.1 software (HP Scanjet 4400C, Hewlett-Packard, USA), using a black background paper. The diameter (mm), perimeter (mm), area ($mm^2$), air cell number, porosity (%), extrudate circularity (0-1), and air cell circularity (0-1) were determined using the Image-J software. Circularity value indicates the scale from 0, without circularity, to 1, a perfect circle. The results were obtained by the average of ten extrudates.

*2.4.4 Pasting properties*

Rapid Visco Analyzer (RVA 4500, Perten Instruments, Australia) was used with the software Thermocline for Windows to determine the pasting properties of the samples. Configuration *Standard 1* was applied for RF and *Extrusion 2* for extrudates (AACCI, 2010). A flour dispersion containing 3.0 g of sample (14% wet basis) and 25 mL of distilled water was prepared. The analysis was performed in triplicate. According to the configuration chosen, the parameters measured from de RVA curve were: cold peak (cP), raw peak (cP), hold peak (cP), breakdown (cP), final viscosity (cP), setback (cP), time (min), and pasting temperature (ºC).

**2.5 Glucan and xylan quantification of YBCF and the extrudates flours**

Glucan and xylan content were determined according to the methodology proposed by the National Renewable Energy Laboratory (NREL) (Sluiter, Hames, Ruiz, Scarlata, Sluiter, Templeton et al., 2008) with some modifications. Firstly, the extrudates were milled in a blender and then sieved to physically separate the YBCF particles, which have different lower density and large particle size compared to RF. About 0.3 g of YBCF and YBCF particles obtained from extrudates after milling and sieving were subjected to acid hydrolysis with 72% (w/w) sulfuric acid for 1 hour, followed by dilution to a final concentration of 4% (w/v) by adding distilled water. The mixture was autoclaved at 121°C for 1 hour, cooled, and filtered through a porous bottom crucible (4-5 µm). Aliquots of 1 mL of the resulting hydrolysate were filtered through a syringe filter of porosity of 0.22 µm, and stored in vials to determine the concentrations of glucose, cellobiose, xylose, arabinose, acetic, and formic acid using high-performance liquid chromatography (HPLC). A Thermo Scientific® Accela chromatography was used for the runs, equipped with a Bio-Rad® HPX-87 H separation column maintained at 45°C, a flow rate of 0.6 mL/min, using a solution of $H_2SO_4$ (0.05mM), pH 2.6 as the mobile phase, during 25 minutes. After obtaining the compounds, the glucan and xylan contents were estimated according to equations (3) and (4).

$$Glucan\ (\%) = \left(\frac{(0.90 \times [\,]glucose) + (0.95 \times [\,]\ cellobiose) + (3.53 \times [\,]\ formic\ acid)}{0.3}\right) \times 0.087 \quad (3)$$

$$Xylan\ (\%) = \left(\frac{(0.88 \times [\,]xylose) + (0.88 \times [\,]\ arabinose) + (0.72 \times [\,]\ acetic\ acid)}{0.3}\right) \times 0.087 \quad (4)$$

**2.6 Bifidogenic activity in vitro of YBCF and the extrudates**

The bifidogenic activity was evaluated according to the method reported by Li, Kim, Jin, & Zhou (2008) and Moro, Celegatti, Pereira, Lopes, Barbin, Pastore et al. (2018) with some modifications. De Man Rogosa Sharpe (MRS) supplemented with 0.5 g/L L-cysteine, 0.2 g/L sodium thioglycolate, and 0.1 g/L calcium chloride was used as a basal medium. A blank control (MRS+bacteria), a positive control (MRS+bacteria+Raftiline GR® (Sweet mix Ltda, Sorocaba, Brazil)), and a negative control (blank control, positive control, and samples without bacteria to eliminate the effect of sample turbidity) were used. Positive control (Raftiline), YBCF, and extrudates were added to MRS broth at a concentration of 3% (w/v) (0.3 g sample to 10 mL of MRS) and inoculated with 1 x $10^6$ cells/mL of *Bifidobacterium bifidum* (*B. bifidum* G90®: BioGrowing Co. Ltd., Shanghai, China), previously activated for 24 h (10 mL of MRS and 50 mg of *B. bifidum*) under anaerobic conditions. The extrudates were prepared in two ways: (A) grinding the extrudate and (B) separating YBCF particles from extrudate by milling and sieving, which was possible due to the bulk density and particle size characteristics of this raw material. The B samples were named as YBC5, YBC10, and YBC15. The samples were incubated at 37 ºC for 24 h in anaerobic jars. After this period, the tubes were centrifuged (3000 × g/10 min/4 ºC), the supernatant was discarded, and 15 mL of phosphate-buffered saline (PBS)

(0.1M phosphate buffer pH 7.4, 0.9% saline) was added. Turbidity was measured in a spectrophotometer (Orion AquaMate 8000, Thermo Scientific) at 600 nm, in quadruplicate, and repeated twice. The bacterial growth was calculated by the difference in turbidity of the sample inoculated with bacteria and the negative control.

### 2.7 Data analysis

For the physicochemical and technological properties, and bifidogenic activity, analysis of variance (ANOVA) was used, and means were compared by the Scott-Knott test ($p$ value $< 0.05$), using the software Analysis of Variance for Balanced Data (SISVAR 5.6). Pearson's correlation was used to determine possible correlations between YBCF concentrations and the technological properties of the final products. Correlations with a 70% confidence level were subjected to principal component analysis (PCA). These two analyses were conducted using Statistic 8.0 software.

## 3. Results and Discussion

### 3.1 Characterization of the raw materials

*3.1.1 Morphological characteristics*

RF presented a higher profusion of starch granules (Fig. 2-1c) characterized by smaller diameters with a polyhedral shape corresponding with the descriptions previously provided by Bao & Bergman (2018). On the other hand, YBCF showed a high amount of fibers and low quantity of starch granules inside the parenchyma cells (Fig. 2-2c). According to Liese & Tang (2015), parenchymal cells have a very important role in the bamboo structure, presenting small cells toward the outside and large and longer cells toward the inside. The elongated cells undergo the lignification process during the intermodal development of the plant, while the shorter cells remain with their original shape.

Starch from *D. latiflorus* also had similar characteristics to other bamboo species, such as *B. tuldoides* (Felisberto, Beraldo, Costa, Boas, Franco, & Clerici, 2019a), *D. asper* (Felisberto, Beraldo, Costa, Boas, Franco, & Clerici, 2019b) and *B. vulgaris* (Felisberto, Beraldo, Costa, Boas, Franco, & Clerici, 2020). However, there is a starch content difference between these species, which can be explained by the culm ages harvested (Felisberto, Beraldo & Clerici, 2015). Aiming to facilitate bamboo processing, we have harvested culms at 4 months old, while Felisberto et al. (2019a, 2019b, 2020) have harvested culms at almost 3 years old to obtain culms with a high starch concentration.

*3.1.2 Proximate composition*

RF presented a proximate composition (Table 1) similar to that found by Seetapan, Limparyoon, Yooberg, Leelawat, & Charunuch (2019). The YBCF showed a higher protein content when compared to the YBCF from *Dendrocalamus asper* studied by Felisberto et al. (2017), a fact probably connected to differences between environment conditions, species and culm ages.

**Table 1 –** Proximate composition of rice flour (RF) and young bamboo culm flour (YBCF) in dry basis (g/100g)[1,2]

| | **RF** | **YBCF** |
|---|---|---|
| Ash | 0.51 ± 0.00 | 2.37 ± 0.09 |
| Protein | 8.51 ± 0.03 | 3.65 ± 0.04 |
| Ether extract | 0.55 ± 0.03 | 0.52 ± 0.06 |
| Total sugars | 0.94 ± 0.38 | 4.95 ± 0.04 |
| Total starch | 70.02 ± 0.26 | 3.68 ± 0.14 |
| Total dietary fiber | 0.75 ± 0.03 | 81.61 ± 1.28 |
| Other carbohydrates[3] | 18.72 | 3.22 |

[1]Mean value ± standard deviation of triplicates

[2]Initial moisture: RF = 10.82±0.18 g/100g; YBCF = 5.07±0.13 g/100g;

[3]Carbohydrates = 100 – (ash + protein + ether extract + total sugar + total starch + total dietary fiber)

Concerning the carbohydrates contents of the raw materials (Table 1), the starch content was higher in RF than YCBF, because of the natural formation of the rice grain, in which the endosperm corresponds to 89-94% of its weight, consisting mainly of starch. The low content of dietary fiber in our RF can be explained by the removal of the outer layers of rice grains during grain processing, polishing, and milling, as reported by Juliano & Tuaño (2019).

The total sugars and total starch contents of YBCF were lower than the results reported by Felisberto et al. (2017) in young culms of *D. asper*. Brito, Tomielis, Silveira, & Cereda (2017) reported stability of sugar content in culms of *B. vulgaris*, *B. multiplex*, and *B. vulgaris* var. *vitatta* during autumn-winter, with changes in the starch contents. According to Banik (2015), the highest starch contents in the culms are observed just before sprouting and gradually increase after the rhizome growth, with a reduction in starch and sugar contents during bamboo shoot growth, which coincides with the autumn-winter season, when the culms of this study were collected. Thus, our young bamboo culms were harvested before emitting branches and leaves, then according to Pereira & Beraldo (2016), it was expected to have low starch concentration.

As expected, YBCF had an expressive dietary fiber content (above 80%), indicating its potential use as a concentrate fiber ingredient. According to Liese & Tang (2015), the fibers present in the culms can contribute between 60 and 70% of the total tissue weight, with culm lengths varying between species and different parts of the culm. YBCF presents higher fiber contents when compared to other cereal flours and bran, such as whole wheat flour (12.6 g/100g) (Dhingra, Michael, Rajput, & Patil, 2012), wheat bran (43 g/100g), rice bran (21 g/100g), and oat bran (15 g/100g) (USDA, 2022).

Unlike the extensive literature and production of big commodities, such as wheat, rice, and corn that have been done, the production of young bamboo culm and bamboo shoot flours containing a range of fibers, starch and sugar is still in their early stages. More data and understanding of the correlation between physicochemical and technological properties, such as fiber content and bulk density (item 3.1.2), are required to achieve a desired flour.

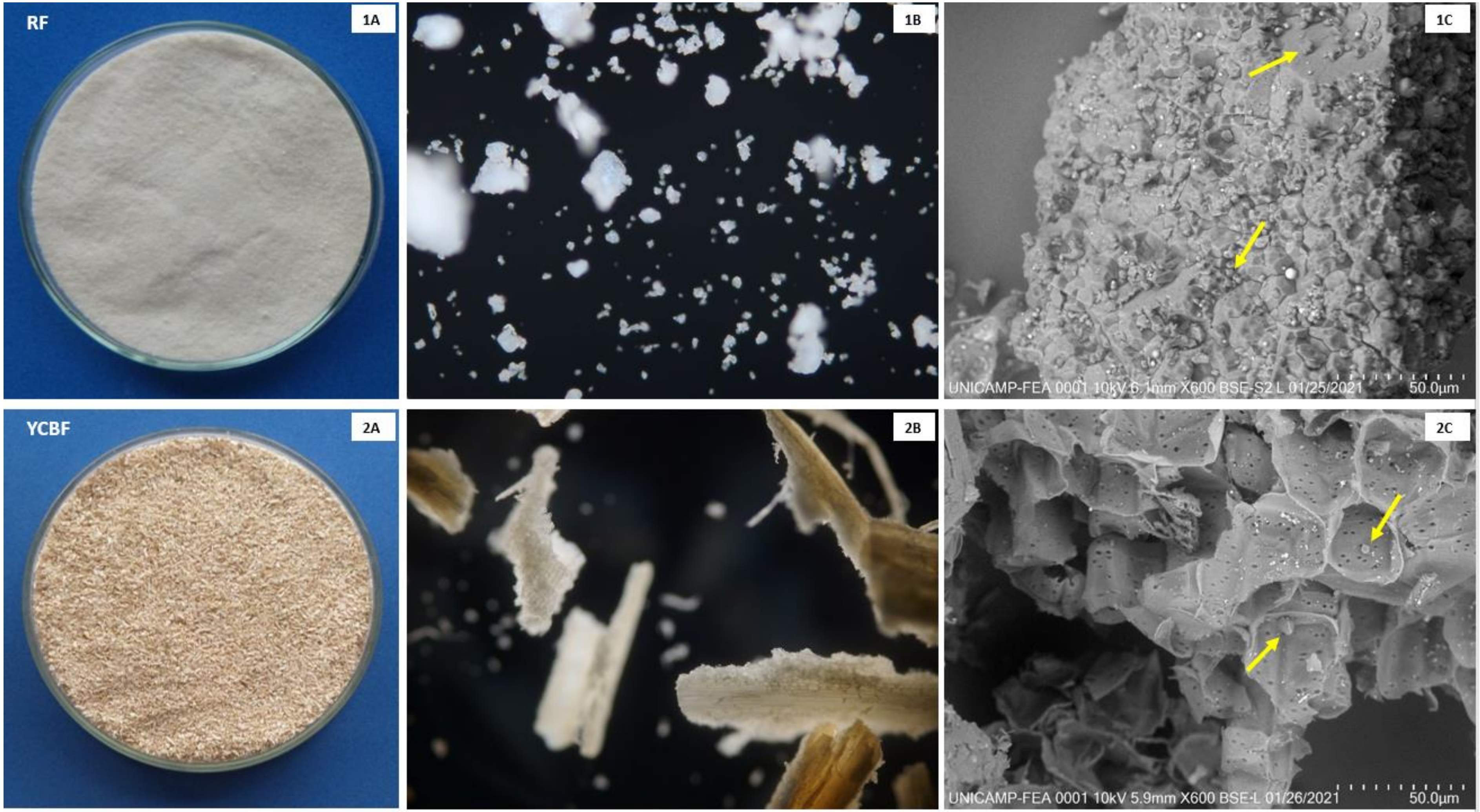



**Fig. 2.** Unaided eye (A), optical microscopy (10x polarized light) (B), and scanning electron (600x) (C) of rice flour (RF) and young bamboo culm flour (YBCF)

*3.1.3 Physicochemical characteristics*

The physicochemical properties of the raw materials are shown in Table 2. The pH of RF was close to neutrality, therefore it can be considered a low-acid food (pH > 5.2), while YBCF was shown as an acid food, with a pH between 4.0 and 4.6 (Erkmen & Bozoglu, 2016). The results of titratable acidity confirmed these characteristics. The pH of YBCF was lower than that reported by Felisberto et al. (2017) in three bamboo species. Probably, pH and acidity may vary according to culm age, thus, further studies are required on this topic since it is already known that bamboo shoot has a rapid natural fermentation due to sugar content, and even the same process can occur in young culms. Therefore, in this study, the young culms were refrigerated and processed within the first 24 hours to avoid increasing acidity by natural fermentation.

Regarding the particle size, RF presented 77% of its particles between 500 µm and 180 µm, while YBCF had 75% of its particles between 1,410 and 500 µm (Table 2). The particle size is an important property in the extrusion process, as the particles can interfere directly in the water distribution and local shear stress, as explained in the item 3.2.1.

The bulk density value of RF is similar to the one reported by Juliano & Tuaño (2019), varying between 0.78 and 0.85 g/mL. This result is due to the removal of the outer layers during grain milling and is also related to the compaction properties of rice starch granules, their shape, and small diameter (Fig. 2). The bulk density of YBCF was similar to sweet sorghum bagasse (0.24 g/cm$^3$) and fiber sorghum bagasse (0.26 g/cm$^3$) (Cardoso, Oliveira, Santana Junior, & Ataide, 2013). The low density of YBCF can be explained by the insoluble fibers, which are naturally porous, and the lignin levels presented in young culms. According to Banik (2015), new and immature culms contain lower lignin levels and density, which makes it easier to cut, mill, and process them into flour. Thus, the determination of this analysis can be used as a quality standard for the raw material to distinguish the ideal young bamboo culm for processing by indirectly estimating the age and the lignin content. In addition, as indicated by Bouvier & Campanella (2014), bulk density becomes essential to assess the flow behavior of the raw materials in the feeding system of the extruder equipment.

Regarding color, RF presented brightness (L*) values close to 100, while the other parameters (a* and b*) were closer to zero (Table 2, as also can be confirmed in Figure 2), indicating a whitish appearance, characteristic of the polished rice grain, which are in agreement with the description of Zhou, Yun, & He (2019) for rice flour. YBCF presented a lower L* value (81.89) compared to RF, but close to 100, tending to a yellowish appearance, as the inner layer of the culm has a different visual color from the thin outer layer (as can be seen in Figure 1). Similar results were reported by Feliberto et al. (2017) for *D. asper*, *B. vulgaris*, and *B. Tuldoides* flours.

**Table 2 –** Technological properties of rice flour (RF) and young bamboo culm flour (YBCF)[1]

| **Properties** | **RF** | **YBCF** |
|---|---|---|
| pH | 6.62 ± 0.08 | 4.44 ± 0.02 |
| Titratable acidity (latic acid) % | 0.36 ± 0.00 | 1.14 ± 0.10 |
| Particle size (%) | | |
| <∞ - 1410 μm | 0.09 ± 0.00 | 0.25 ± 0.02 |
| <1410 - 841 μm | 0.22 ± 0.00 | 30.61 ± 0.63 |
| < 841 - 500 μm | 0.21 ± 0.00 | 45.34 ± 0.51 |
| <500 - 250 μm | 42.01 ± 0.94 | 18.20 ± 0.63 |
| <250 - 180 μm | 35.67 ± 1.41 | 2.68 ± 0.35 |
| <180 - 150 μm | 7.55 ± 0.56 | 0.85 ± 0.01 |
| <150 μm | 13.80 ± 0.53 | 1.98 ± 0.14 |
| Fineness modulus | 2.08 ± 0.07 | 3.97 ± 0.14 |
| Bulk density (g/mL) | 0.84 ± 0.00 | 0.28 ± 0.01 |
| Color in RGB system | | |
| Color parameters | | |
| L* | 96.06 ± 0.28 | 81.89 ± 0.28 |
| a* | -0.08 ± 0.01 | 0.44 ± 0.02 |
| b* | 7.67 ± 0.14 | 16.98 ± 0.01 |
| Hydration properties | | |
| Swelling capacity (mL/g) | 0.15 ± 0.00 | 10.35 ± 0.74 |
| WAI[2] | 2.23 ± 0.02 | 10.15 ± 0.02 |
| WSI (%)[3] | 1.24 ± 0.04 | 9.62 ± 0.46 |
| Pasting properties | | |
| Raw peak (cP) | 2396.00 ± 45.00 | - |
| Hold peak (cP) | 1741.00 ± 45.00 | - |
| Breakdown (cP) | 655.00 ± 10.00 | - |
| Final viscosity (cP) | 3849.00 ± 70.00 | - |
| Setback (cP) | 2108.00 ± 32.00 | - |
| Time (min) | 6.10 ± 0.00 | - |
| Pasting temperature (ºC) | 84.50 ± 0.03 | - |

[1]Mean ± standard deviation of triplicate
[2]WAI: water absorption index; [3]WSI: water solubility index

Comparing the hydration properties of samples, RF presented a lower swelling capacity, WAI, and WSI than YBCF (Table 2). These results can be related to the differences in starch and fiber content of the raw materials shown in item 3.1.2. As explained by Vandeppute, Derycke, Geeroms, & Delcour (2003) and Eliasson & Gudmundsson (2006), native starch swelling and water absorption capacity are almost non-existent at room temperature. However, as Hasjim, Li, & Dhital (2012) explained, damaged starches obtained during rice flour milling can increase its WAI. Similar results were observed by Seetapan et al. (2019), as the WAI of rice flour was 2.49 g/g, while the WSI was lower 0.07 g/g. Comparing the hydration properties of YBCF, Rosell et al. (2009) found a lower swelling capacity in commercial bamboo shoot fiber (5.69 mL/g), while Wang, Ma, Zhu, Wang, Ren, Zhang et al. (2017) found high values between 13.90-28.75 mL/g for bamboo shoot fibers (*Phyllostachys praecox*). Felisberto et al. (2017) reported low WAI and WSI values for the young bamboo culm flour from *D. asper* at approximately 3 years of age compared to our YBCF from *D. latiflorus*.

Besides the chemical composition of YBCF, parenchymal cell content, and its porous fibers, the hydration behavior of fibers is dependent on several factors, including particle size, pH, temperature (Elleuch, Bedigian, Roiseux, Besbes, & Blecker, 2011), molecule size, degree of chain branching, and types of intermolecular bonds (Meuser, 2001).

### 3.2 Extrusion process and extrudates properties

*3.2.1. Torque and specific mechanical energy (SME)*

The partial replacement of rice flour by YBCF above 10% increased the torque and the SME during processing (Table 3). In a first approach, these results suggest a higher shear/friction between RF-YBCF particles and between particles, screws, and extruder barrel, which may have led to the formation of more dextrinized starches in extrudates containing YBCF compared to RF alone (discussion in items 3.2.3 and 3.2.4).

However, we also need to consider the amount and particle size of the raw material source of dietary fiber. According to Robin, Théoduloz, Gianfrancesco, Pineau, Schuchmann, & Palzer et al. (2011), for extrudates with low moisture content (18-22%), a minimum amount of fiber and particle size are required to increase local mechanical stress, starch conversion and melt viscosity at a constant shear. They observed the behavior of wheat bran particles at two concentrations (12% and 24.4%) and two particle sizes (224 µm and 317 µm) when replacing refined wheat flour. Even though reducing particle size could increase the extent of shear imparted to starch, they did not notice any significant effects that indicated starch conversion using these process conditions. However, they verified that fiber mainly affects the solubility of proteins.

**Table 3 –** Extrusion process parameters and technological properties of extrudates and respective flours containing rice flour and young bamboo culm flour (*D. latiflorus*)[1]

| **Properties** | **Assays[2]** | | | |
|---|---|---|---|---|
| | **RB0** | **RB5** | **RB10** | **RB15** |
| *Extrusion process* | | | | |
| Torque (%) | 43.20±1.30[c] | 42.20±1.30[c] | 49.60±1.14[b] | 51.40±2.07[a] |
| SME (W.h/kg) | 166.38±5.02[b] | 162.53±5.02[b] | 191.03±4.39[a] | 197.96±7.97[a] |
| *Extrudates* | | | | |
| Cross-section | | | | |
| Diameter (mm) | 14.07 ± 0.58[a] | 13.17 ± 0.37[b] | 11.62 ± 0.40[c] | 11.80 ± 0.47[c] |
| Perimeter (mm) | 47.30 ± 1.98[a] | 42.73 ± 0.95[b] | 38.74 ± 1.12[c] | 38.95 ± 1.56[c] |
| Area ($mm^2$) | 157.95 ± 12.76[a] | 137.90 ± 6.47[b] | 109.59 ± 6.74[c] | 111.32 ± 8.62[c] |
| Cell number | 9.40 ± 1.07[c] | 13.20 ± 2.15[b] | 16.00 ± 3.19[a] | 17.70 ± 5.58[a] |
| Porosity (%) | 45.47 ± 7.02[n.s.] | 39.58 ± 8.02[n.s.] | 40.08 ± 6.56[n.s.] | 41.16 ± 4.88[n.s.] |
| Extrudate circularity | 0.89 ± 0.01[c] | 0.91 ± 0.01[b] | 0.92 ± 0.01[a] | 0.92 ± 0.01[a] |
| Cell circularity | 0.33 ± 0.03[n.s.] | 0.33 ± 0.04[n.s.] | 0.31 ± 0.05[n.s.] | 0.30 ± 0.04[n.s.] |
| Expansion index | 2.68 ± 0.12[a] | 2.51 ± 0.18[b] | 2.43 ± 0.14[b] | 1.99 ± 0.12[c] |
| Bulk density ($g/cm^3$) | 0.25 ± 0.03[a] | 0.19 ± 0.02[b] | 0.17 ± 0.02[b] | 0.25 ± 0.03[a] |
| Texture (N) | 57.67 ± 3.80[c] | 59.65 ± 6.80[c] | 82.01 ± 3.48[a] | 76.17 ± 5.28[b] |
| Color in RGB system | | | | |
| Color parameters | | | | |
| L* | 83.83 ± 0.01[b] | 85.49 ± 0.24[a] | 81.52 ± 0.07[c] | 83.71 ± 0.64[b] |
| a* | 0.83 ± 0.01[a] | 0.62 ± 0.05[b] | 0.60 ± 0.04[b] | 0.44 ± 0.04[c] |
| b* | 17.22 ± 0.01[b] | 17.44 ± 0.52[b] | 17.04 ± 0.13[b] | 18.59 ± 0.34[a] |
| *Extruded flours* | | | | |
| pH | 6.10 ± 0.03[a] | 6.05 ± 0.01[b] | 5.81 ± 0.01[c] | 5.64 ± 0.01[d] |
| Titratable acidity (lactic acid) % | 0.18 ± 0.00[b] | 0.18 ± 0.00[b] | 0.36 ± 0.00[a] | 0.36 ± 0.00[a] |
| WAI[3] | 7.15 ± 0.09[a] | 6.83 ± 0.15[b] | 6.13 ± 0.05[c] | 5.48 ± 0.08[d] |
| WSI (%)[4] | 25.20 ± 1.07[d] | 36.16 ± 1.52[c] | 40.80 ± 0.47[b] | 55.09 ± 1.39[a] |

[n.s.]- not significant [1]Mean value ± standard deviation, Different lowercase letters in the same line indicate statistically significant differences by Scott-Knott test. [2]RF:YBCF ratio: RB0 – 100:0, RB5 - 95:5, RB10 - 90:10, RB15 - 85:15; [3]WAI: water absorption index; [4]WSI: water solubility index

*3.2.2 Image analyses of extrudates*

The parameters analyzed for the cross-section (Table 3) showed no significant differences ($p<0.05$) for porosity (%) and cell circularity between the samples. The diameter, perimeter, and area of the extrudates decreased while the number of air cells increased by YBCF incorporation. The higher number of air bubbles with small diameters improved the extrudate circularity (Fig. 3). According to Barrett (2003) and Sozer & Poutanen (2013), fibers interfere with the air cell wall viscosity and act as a filling agent in the starch matrix. Chanvrier, Desbois, Perotti, Salzmann, Chassagne, Gumy et al. (2013) also observed this when enriched wheat, corn, and soybean-based extrudates formulations with oat bran concentrate and wheat bran (10-20%).

RB0 extrudates presented thin, smooth, and continuous walls without distinction between macronutrients, such as starch and proteins. In the extrudates containing YBCF (RB5, RB10, and RB15), there were observed fibers adhered to the smooth walls, forming protrusions visible to the ImageJ (white dots) and SEM (yellow arrows) (Fig. 3).

To the unaided eye (Fig. 4a), powdered extrudates showed few differences and an increase of heterogeneous particles in samples containing YBCF (RB5, RB10, and RB15). The images obtained for all samples under the polarized light OM (Figure 4b) did not allow the identification of the Maltese cross, suggesting a starch gelatinization/dextrinization during the extrusion process. Fiber particles were easily recognized in RB5, RB10, and RB15 samples by both OM and SEM techniques (Fig. 4c). Although these particles had adhesion points in the starch/protein matrix, their presence resulted in heterogeneous powdered extrudates.

*3.2.3 Physicochemical properties*

The physicochemical properties of the extrudates samples are shown in Table 3. The samples containing YBCF showed a significant reduction in expansion index and increase in bulk density when compared to the control (RB0). Fiber particles act as fillers and create collapse regions, reducing the air bubble diameters and consequently affecting expansion and bulk density (Robin, Schuchmann, & Palzer, 2012). This phenomenon can be explained by the low physicochemical compatibility between insoluble fibers and starch granules. The changes in starch structure during extrusion can also be affected by the difference in hardness between particles and the lower uniformity of water distribution in the system (Robin et al., 2012).

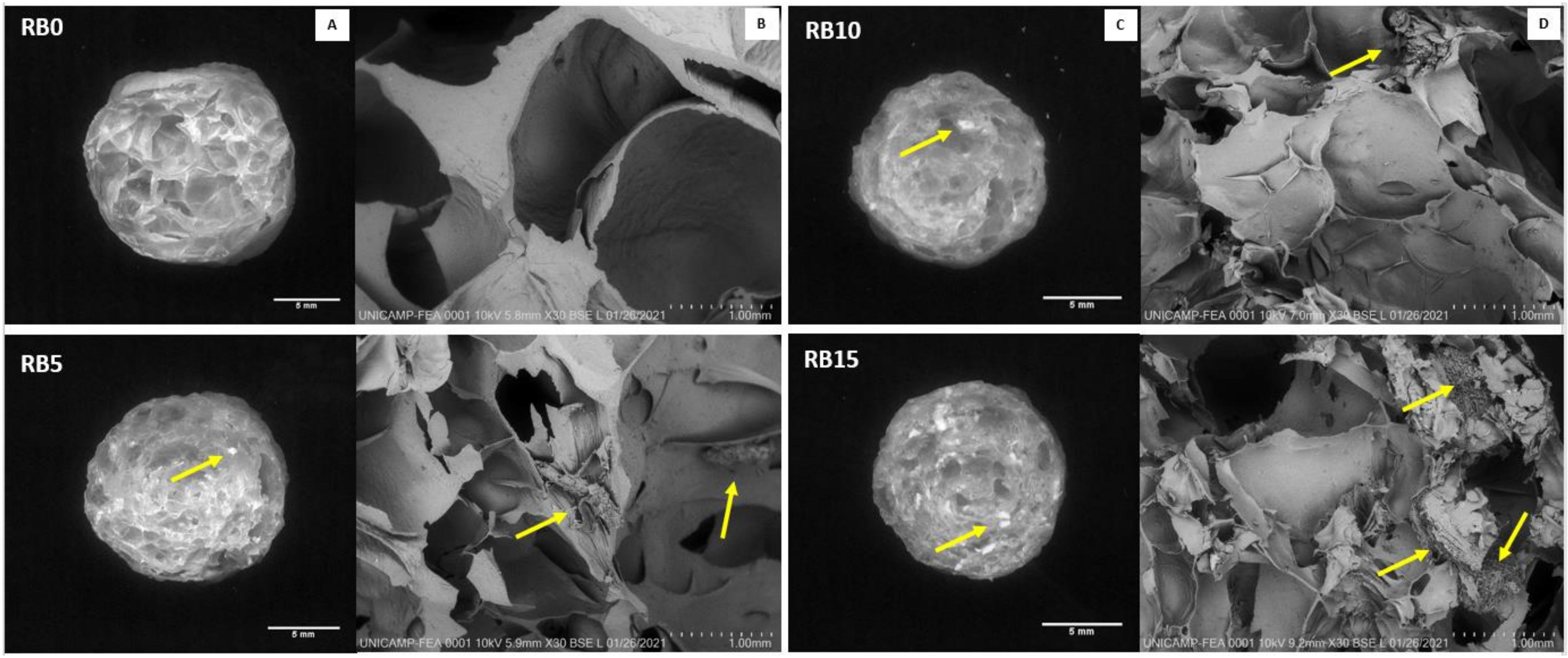



**Fig. 3.** Cross-sectional area of extrudates by ImageJ (A and C) and SEM (B and D)

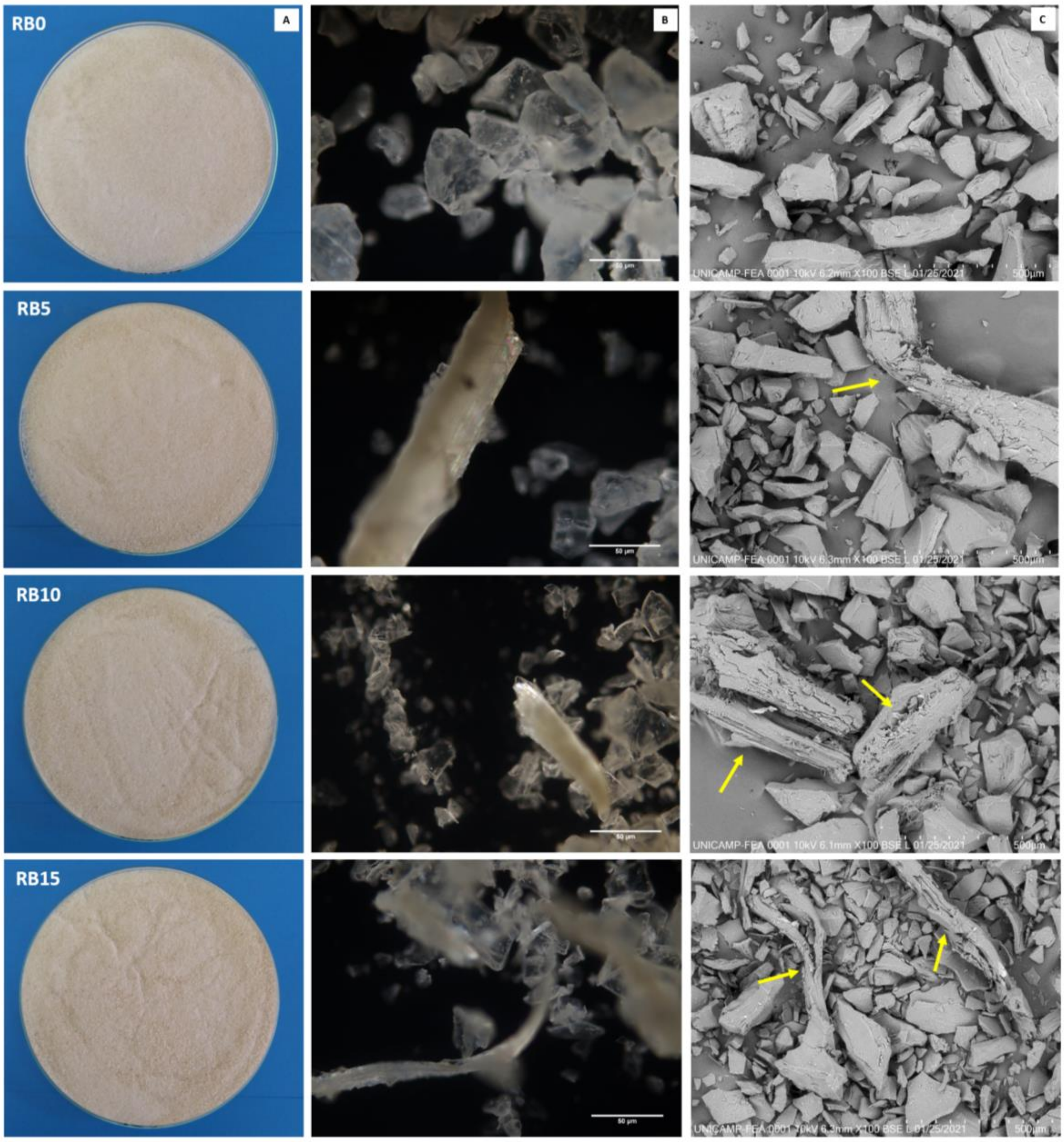



**Fig. 4.** Unaided eye (A), optical microscopy (10x polarized light) (B) and scanning electron (100x) (C) of extruded flours

A higher compression force to break the rice extrudates containing YBCF was required compared to the control (Table 3). RB10 and RB15 had the highest hardness values. Barret (2003) explained that the cell structure developed during expansion in the presence of fibers increases the extrudate resistance to deformation. The water absorption capacity of YBCF can also be related to those results, as it can prevent water evaporation from the matrix, leading to a lower expansion, higher density, and hardness of the products. Several studies observed similar behavior in expansion, bulk density, and hardness when using whole cereal flours or cereal bran (Oliveira et al., 2017; Makowska, Polcyn, Chudy, Michniewicz, 2015; Fleischman, Kowalski, Morris, Nguyen, Li, Ganjyal, & Ross, 2016)

Significant differences ($p<0.05$) were observed in the color parameters between the formulations. YBCF incorporation reduced the lightness (L*) and redness (a*) and increased yellowness (b*) in extrudates. Variations in the extrusion process, such as residence time, and the formation of melanoidins by Maillard reaction, can also be linked to these results. Martinez, Calviño, Rosell, & Gómez (2014) studied rice flour extrusion under different process conditions and also reported changes in the color parameters, except for the parameter a*, which increased after extrusion.

At YBCF concentrations of 10% and 15%, the pH was reduced by up to 4.3%, while the titratable acidity doubled in value. Despite this result, the extrudates can still be considered low-acid food (pH > 5.2) (Erkmen & Bozoglu, 2016). The contribution of YBCF to these parameters was expected, and all these factors, combined with the low moisture content, can favor a longer shelf life of the final product.

In terms of hydration properties, WAI indicates starch integrity, whereas WSI refers to the soluble solids content released into the water. These parameters assess the degree of starch damage, gelatinization, and dextrinization after the extrusion cooking. As shown in Table 3, increasing the YBCF concentration resulted in a significant reduction of WAI and a significant increase in WSI. The decrease in WAI can be related to the lower starch content available in the extrudates containing YBCF compared to the others and the disruption of the starch structures.

The increase in WSI is probably related to starch dextrinization and protein solubilization rather than a change in fiber solubility. The gelatinization and disruption of starch structures during extrusion make them more soluble in water (Ek, Kowalski, & Ganjyal, 2020), while the solubility of dietary fibers is slightly modified (Wang & Klopfenstein, 1993). In addition, the processing conditions in this study remained unchanged, except for the YBCF concentration. These suggest a possible contribution of YBCF particles to increase starch dextrinization by increasing local shear besides the screw configuration effect (Figure S1 - supplementary material). Robin, Dubois, Pineau, Labat, Théoduloz, & Curti (2012) also stated that the increase in WSI (23.1-37.0%) of whole wheat flour extrudates was due to starch degradation provided by the increase in mechanical stress and specific energy.

### 3.2.4 Pasting properties

The RVA is applied to evaluate starch behavior. In their native form, starch granules exhibit low water solubility at room temperature, but when the gelatinization temperature is reached, it can be observed an increase in viscosity. Since only RF has enough starch content to form a paste, YBCF pasting properties will be briefly discussed here. RF showed a peak viscosity at 2396.00 ± 45.00 cP and at a temperature of 84.50 ± 0.03 ºC (Table 2). This peak represents the maximum swelling capacity of starch granules before their disruption. A new increase in viscosity at 50 °C occurs when the amylose and amylopectin molecules reassociate. Our results agree with the literature on rice flour (Guha & Zakiuddin Ali, 2011; Wani, Singh, Shah, Schweiggert-Weisz, Gul, & Wani, 2012). On the other hand, the pasting properties of YBFC showed a low viscosity profile (< 200 cP) (Table S1 - supplementary material). As explained by Mudgil & Barak (2019), insoluble fibers, such as cellulose and hemicellulose, cannot increase the viscosity of the medium and form a gel, as they have a complex crystalline molecular structure, with the presence of intermolecular hydrogen bonds. YBCF also presented lower viscosity compared, for example, to whole wheat flour (Robin *et al*, 2012; Oro, Limberger, de Miranda, Richards, Gutkoski, & de Francisco, 2013).

As expected, the extrusion process and the partial replacement of RF by YBCF led to a drastic reduction in paste viscosity in the samples (Table 4), showing a peak viscosity value 6 times lower when compared to unprocessed RF. All extrudates showed cold viscosity peaks, corroborating with the WSI results, and a maximum peak viscosity (95 ºC), probably owing to partially gelatinized and/or intact starch granules. However, a significant difference was observed for the breakdown of the extrudates containing YBCF when compared to the control. YBCF particles seem to become a point of mechanical stress, increasing the torque of the system, which may have accelerated the breakdown of the remaining starch granules. This same effect in RVA can also suggest the material behavior in the extruder, which explains the results of the hydration properties and viscosity profile. The proportional increase in torque recorded by the equipment and the mechanical specific energy (Table 4) suggest that the higher starch conversion/dextrinization was attributed to increased attrition between the YBCF and RF particles.

**Table 4 –** Pasting properties of extrudates containing rice flour and young bamboo culm flour*

| Assays[1] | Cold peak (cP) | Raw peak (cP) | Hold peak (cP) | Breakdown (cP) | Final viscosity (cP) | Setback (cP) | Time (min) |
|---|---|---|---|---|---|---|---|
| RB0 | 123.50±4.95[c] | 307.50±4.95[n.s.] | 251.50±2.12[a] | 56.00±2.83[b] | 256.00±1.41[a] | 4.50±0.71[c] | 8.00±0.00[a] |
| RB5 | 168.00±7.07[a] | 341.00±4.24[n.s.] | 162.50±4.95[b] | 178.50±9.19[a] | 193.00±7.07[b] | 30.50±2.12[b] | 6.37±0.05[b] |
| RB10 | 139.00±2.83[b] | 322.00±7.07[n.s.] | 121.00±1.41[c] | 201.00±5.66[a] | 151.50±2.12[c] | 30.50±0.71[b] | 6.13±0.00[c] |
| RB15 | 164.50±3.54[a] | 359.50±44.55[n.s.] | 110.50±2.12[d] | 249.00±42.43[a] | 157.00±0.00[c] | 46.50±2.12[a] | 6.10±0.05[c] |

[n.s.]- not significant

[1]RF:YBCF ratio: RB0 – 100:0, RB5 - 95:5, RB10 - 90:10, RB15 - 85:15.

*Mean value ± standard deviation. Different lowercase letters in the same column indicate statistically significant differences by Scott-Knott test.

### 3.3 Principal component analysis (PCA)

Results shown in Fig. 5 may be used to predict the behavior of raw materials during processing and the characteristics of the final products. For example, a negative correlation above 0.95 was observed between SME, water absorption index (WAI), and cell circularity (CC), and above 0.80 between SME and expansion index (EI), diameter (DI), perimeter (PE), and area (AR). While a positive correlation above 0.80 was observed between SME, cell number (NC), and water solubility index (WSI). Also, positive and negative correlations above 0.95 were noticed between pasting properties and physical properties, such as NC and hold peak (HP), breakdown (BD), and final viscosity (FV) (Supplementary material – Fig. S2). These correlations showed the main impacts of YBCF particles in a starch-based formulation, in which a higher starch conversion occurs during the extrusion process, as a consequence of the increase in SME, that also led to high water solubility index and low peak viscosity.

The principal component analysis allowed assessing 88.69% of the results, where the response variables and samples were distributed in the four quadrants. As revealed in Figure 5, RB0 and RB15 affected the outcomes since they are closer to Factor 1 axis (75.17%), whereas RB5 and RB10 are closer to Factor 2 axis (13.52%). Therefore, a concentration of up to 10% YBCF can be used to increase dietary fiber content and produce extruded products similar to the control RB0 with less impact on technological quality. Then, an advantage of RB5 and RB10 having less influence on technological results is that probably the production chain, from processing to the final consumer, will not require major investments for adjustments or modifications.

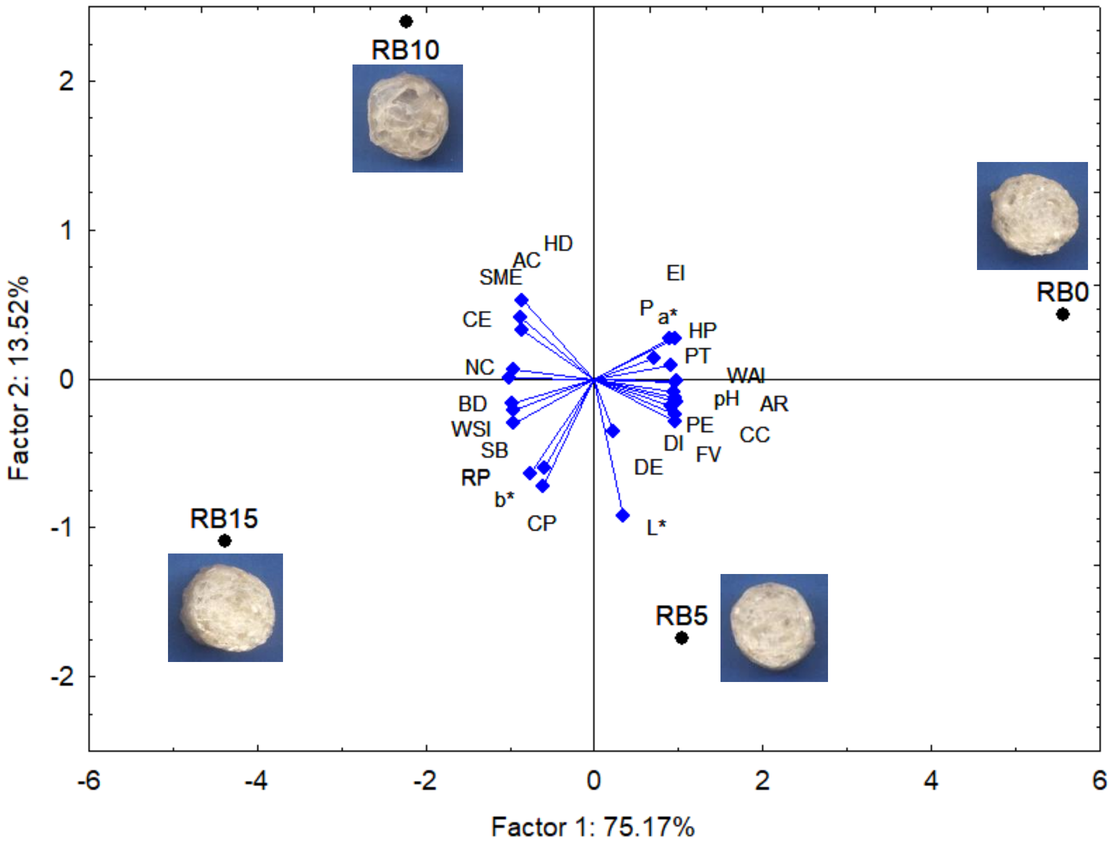
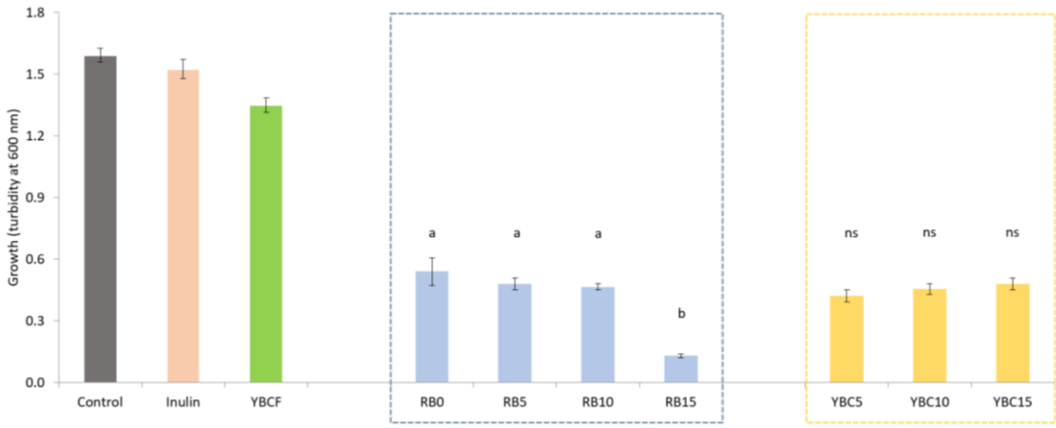



**Fig. 5.** Principal component analysis of technological properties of extrudates

### 3.4 Glucan and xylan quantification and Bifidogenic activity in vitro

YBCF presented above 7% and 20% glucan and xylan content (Fig. 6), respectively, showing a great potential to be a prebiotic source for food products. When hydrolyzed, xylan and glucan produce xylo-oligosaccharides and cello-oligosaccharides, respectively, which promote the growth of probiotic strains, releasing short-chain fatty acids and consequently promoting physiological functions in the body (Farias, Mélo, Silva, Bivilaqua, Ribeiro, Goldbeck et al., 2022; Ávila, Silva, Martins, & Goldbeck, 2021).

No significant difference ($p<0.05$) was observed for glucan content between the extruded YBCF particles (YBC5, YBC10, and YBC15), while xylan content was varied. The glucan content in these samples was higher when compared to YBCF (Fig. 6), which indicates a change in the fiber structure during the extrusion process, thus promoting an extraction of xylan that composes the hemicellulosic fraction and the consequent maintenance of the integrity and exposure of the glucan that composes the cellulosic fraction. Felisberto et al. (2021) also reported an expressive content of xylans and arabinoxylans in other young bamboo species known for their prebiotic activities.




| | Control | Inulin | YBCF | RB0 | RB5 | RB10 | RB15 | YBC5 | YBC10 | YBC15 |
|---|---|---|---|---|---|---|---|---|---|---|
| Glucan (%) | - | - | 7.84±0.24 | - | - | - | - | 50.77±2.58$^{ns}$ | 39.53±0.09$^{ns}$ | 44.38±0.06$^{ns}$ |
| Xylan (%) | - | - | 22.00±0.14 | - | - | - | - | 0.00±0.00$^{c}$ | 8.68±0.01$^{a}$ | 0.99±0.01$^{b}$ |

**Fig. 6.** Bifidogenic activity and polysaccharides content from YBCF, extrudates (RB0, RB5, RB10, and RB15) and extruded YBCF particles obtained after milling and sieving (YBC5, YBC10 and YBC15). Different lowercase letters in the bars/row indicate statistically differences by Scott-Knott test

Regarding bifidogenic activity, YBCF stimulated bacterial growth, presenting turbidity of 1.35 ± 0.03 at the end of 24h of incubation; the control blank was 1.59 ± 0.03, and inulin was 1.52 ± 0.05 (Figure 6). Despite the difference in bacterial growth between YBCF and the others, this is a promising result, as YBCF was not subjected to extraction and hydrolysis procedures to obtain prebiotic oligosaccharides from the isolated fiber fractions. In addition, possibly the 24h cultivation time and/or the other cultivation conditions may not have been sufficient for the probiotic strain to secrete cellulolytic and hemicellulolytic enzymes capable of efficiently degrading the polysaccharides present in the flour when compared to the control and inulin.

Similar bacterial growth was observed for the extrudates group with an exception for RB15, and for the sieved YBCF particles group (YBC5, YBC10, and YBC15) (Figure 6). Compared to YBCF, bifidogenic activity was reduced after thermal and mechanical treatment. However, this result demonstrates that *B. bifidum* strains consumed all carbohydrates available, and YBCF was still viable for bacterial growth after the extrusion cooking. The lower bacterial growth in RB15 may be due to the available sugar content, while similar growth observed between the milled and sieved group may be due to the stability of the YBCF particles during the extrusion process.

The prebiotic activity of bamboo shoots was investigated previously (He et al., 2016; Chen et al., 2019) since this product is already consumed in Asian countries and has significant importance in their population's diet. Recently, Ge, Li, Zheng, Yang, Li et al. (2022) demonstrated that insoluble dietary fiber from bamboo culm (*Dendrocalamus membranaceus Munro*), average three years old, could be utilized by human gut microbiota and produce SCFA in an *in vitro* test.

Prebiotics have gained attention from researchers for their health benefits, but they must be stable for food processing, maintaining their intact structure. Nevertheless, few studies have reported the stability of commercial prebiotics during extrusion cooking using complex food matrices. Duar, Ang, Hoffman, Wehling, Hutkins, & Schlegel (2015) noticed better GOS and resistant starch stability at 2% addition to breakfast cereal formulation compared to inulin and FOS. Ferreira, Capriles, & Conti-Silva (2021) found a reduction in fructans after the extrusion of a corn-based breakfast cereal containing inulin at different concentrations (5.1-24.9%).

According to Steed & Macfarlane (2009), various other factors may affect the stability of prebiotic fibers during the processing, including the composition of sugars, polymerization degree of the prebiotics, availability of other carbohydrates, as well as the chemical reactions, such as the Maillard reaction. Data reported in the literature and those obtained in our study show the need for further investigation of prebiotic fiber properties in complex food matrices and different unit operations. The results also indicate a future for *in vivo* studies, that confirm short chain fatty acids (SCFA) production and identify genetically the human gut microbiota.

## 4. Conclusion

Young bamboo culm proved to be a potential ingredient in the manufacture of extruded products, containing more than 80% of dietary fiber and potential prebiotic activity, which makes it a promising raw material for the development of the local economy.

In the rice-based extrudates, the YBCF favored a higher starch conversion during the extrusion, increasing the specific mechanical energy. The partial replacement of RF by YBCF also led to a reduction in the expansion index and an increase in hardness, indicating that up to 10% replacement can be ideal for ready-to-eat products.

As the first application of YBCF in extruded products, the results point to future studies, that can confirm the benefits of using and consuming young bamboo culm fibers. Adjustments are still required to improve the texture and bulk density of extruded products for immediate consumption. Further studies on particle size, particle dispersion in an aqueous medium for the use of extruded flour in other formulations, such as beverages and soups, and thermal stability of YBCF are also required.

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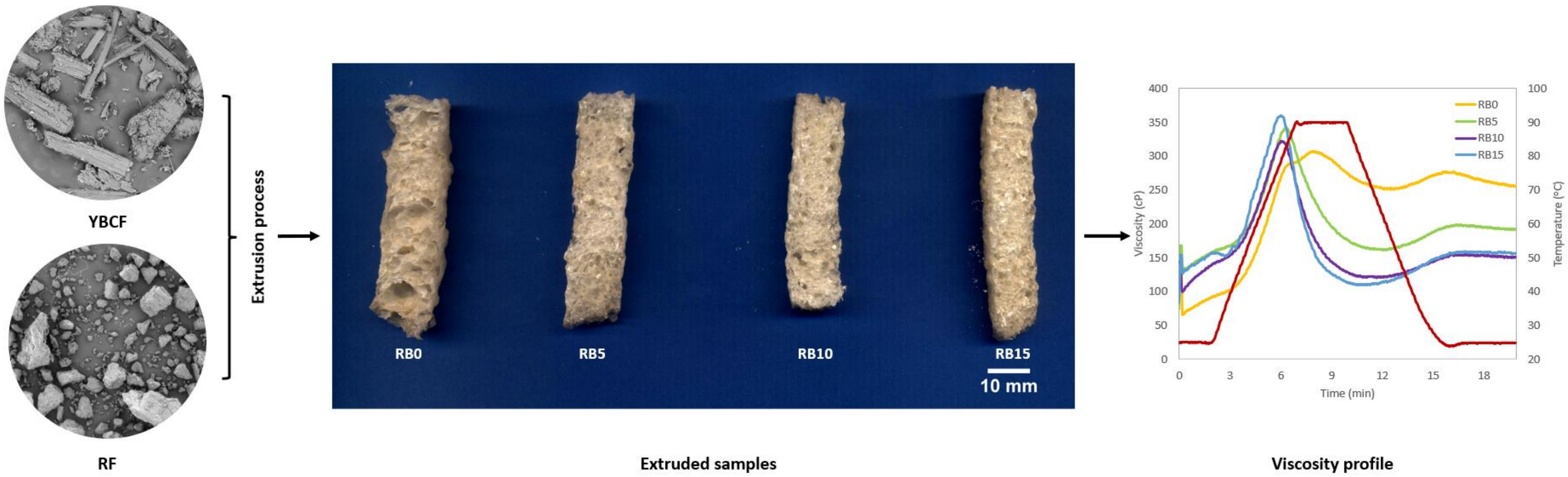
## Graphic abstract



**Supplementary material**

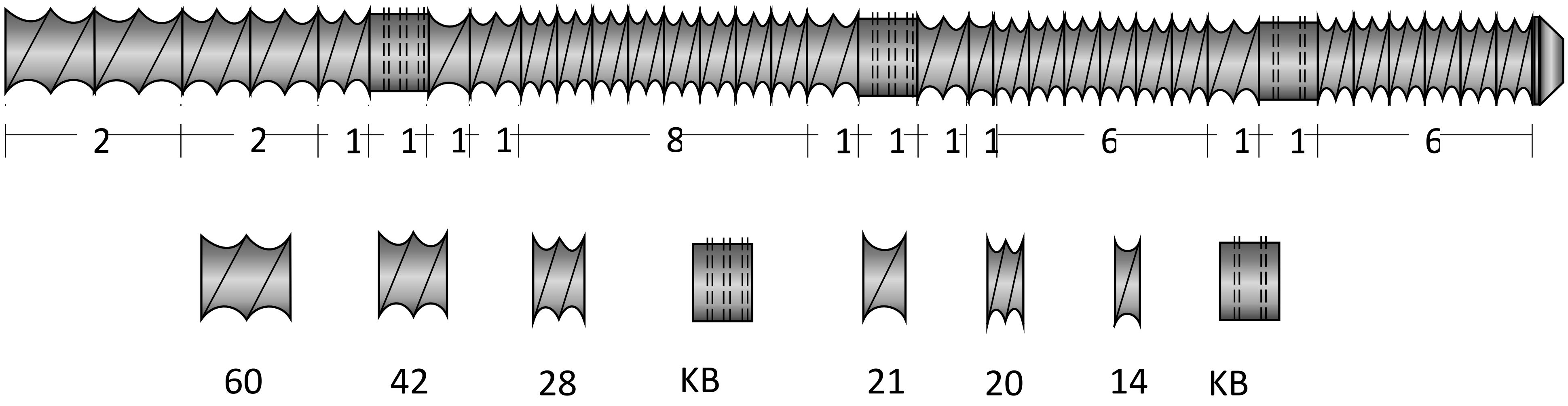



**Figure S1 -** Twin screw configuration

| | | CP | RP | HP | BD | FV | SB | PT | EI | DE | HD | L* | a* | b* | WAI | WSI | DI | PE | AR | P | CE | CC | NC | pH | AC | SME |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Pasting properties** | **CP** | 1.000 | 0.918 | -0.652 | 0.761 | -0.573 | 0.821 | -0.747 | -0.626 | -0.175 | 0.120 | 0.508 | -0.779 | 0.621 | -0.471 | 0.672 | -0.394 | -0.531 | -0.440 | -0.754 | 0.627 | -0.291 | 0.604 | -0.394 | 0.163 | 0.163 |
| | **RP** | 0.918 | 1.000 | -0.733 | 0.848 | -0.647 | 0.917 | -0.740 | -0.874 | 0.087 | 0.319 | 0.317 | -0.911 | 0.860 | -0.731 | 0.876 | -0.547 | -0.639 | -0.587 | -0.604 | 0.693 | -0.589 | 0.760 | -0.673 | 0.421 | 0.468 |
| | **HP** | -0.652 | -0.733 | 1.000 | -0.982 | 0.993 | -0.944 | 0.974 | 0.798 | 0.417 | -0.828 | 0.323 | 0.938 | -0.463 | 0.876 | -0.897 | 0.952 | 0.989 | 0.966 | 0.834 | -0.998 | 0.809 | -0.981 | 0.849 | -0.821 | -0.768 |
| | **BD** | 0.761 | 0.848 | -0.982 | 1.000 | -0.953 | 0.989 | -0.964 | -0.863 | -0.302 | 0.734 | -0.164 | -0.983 | 0.598 | -0.885 | 0.941 | -0.894 | -0.948 | -0.916 | -0.817 | 0.969 | -0.793 | 0.975 | -0.848 | 0.757 | 0.728 |
| | **FV** | -0.573 | -0.647 | 0.993 | -0.953 | 1.000 | -0.897 | 0.965 | 0.738 | 0.489 | -0.870 | 0.414 | 0.891 | -0.365 | 0.850 | -0.850 | 0.970 | 0.996 | 0.979 | 0.834 | -0.998 | 0.799 | -0.965 | 0.830 | -0.844 | -0.774 |
| | **SB** | 0.821 | 0.917 | -0.944 | 0.989 | -0.897 | 1.000 | -0.927 | -0.902 | -0.188 | 0.649 | -0.045 | -0.996 | 0.699 | -0.879 | 0.959 | -0.829 | -0.893 | -0.856 | -0.772 | 0.922 | -0.773 | 0.949 | -0.836 | 0.697 | 0.691 |
| | **PT** | -0.747 | -0.740 | 0.974 | -0.964 | 0.965 | -0.927 | 1.000 | 0.698 | 0.541 | -0.710 | 0.177 | 0.901 | -0.380 | 0.755 | -0.818 | 0.873 | 0.941 | 0.894 | 0.935 | -0.978 | 0.657 | -0.916 | 0.713 | -0.678 | -0.604 |
| **Physical properties** | **EI** | -0.626 | -0.874 | 0.798 | -0.863 | 0.738 | -0.902 | 0.698 | 1.000 | -0.213 | -0.637 | 0.117 | 0.936 | -0.886 | 0.953 | -0.982 | 0.754 | 0.768 | 0.774 | 0.425 | -0.756 | 0.894 | -0.887 | 0.932 | -0.757 | -0.821 |
| | **DE** | -0.175 | 0.087 | 0.417 | -0.302 | 0.489 | -0.188 | 0.541 | -0.213 | 1.000 | -0.334 | 0.267 | 0.123 | 0.562 | -0.036 | 0.024 | 0.380 | 0.431 | 0.377 | 0.742 | -0.475 | -0.072 | -0.244 | -0.055 | -0.144 | 0.038 |
| | **HD** | 0.120 | 0.319 | -0.828 | 0.734 | -0.870 | 0.649 | -0.710 | -0.637 | -0.334 | 1.000 | -0.789 | -0.678 | 0.222 | -0.840 | 0.718 | -0.960 | -0.901 | -0.944 | -0.494 | 0.837 | -0.887 | 0.855 | -0.863 | 0.978 | 0.924 |
| **Color** | **L*** | 0.508 | 0.317 | 0.323 | -0.164 | 0.414 | -0.045 | 0.177 | 0.117 | 0.267 | -0.789 | 1.000 | 0.089 | 0.259 | 0.401 | -0.175 | 0.590 | 0.460 | 0.547 | 0.013 | -0.351 | 0.549 | -0.357 | 0.466 | -0.725 | -0.663 |
| | **a*** | -0.779 | -0.911 | 0.938 | -0.983 | 0.891 | -0.996 | 0.901 | 0.936 | 0.123 | -0.678 | 0.089 | 1.000 | -0.736 | 0.916 | -0.981 | 0.843 | 0.895 | 0.868 | 0.717 | -0.913 | 0.823 | -0.960 | 0.879 | -0.739 | -0.745 |
| | **b*** | 0.621 | 0.860 | -0.463 | 0.598 | -0.365 | 0.699 | -0.380 | -0.886 | 0.562 | 0.222 | 0.259 | -0.736 | 1.000 | -0.713 | 0.797 | -0.363 | -0.396 | -0.393 | -0.131 | 0.402 | -0.633 | 0.583 | -0.683 | 0.401 | 0.528 |
| **Hydration properties** | **WAI** | -0.471 | -0.731 | 0.876 | -0.885 | 0.850 | -0.879 | 0.755 | 0.953 | -0.036 | -0.840 | 0.401 | 0.916 | -0.713 | 1.000 | -0.969 | 0.902 | 0.886 | 0.909 | 0.472 | -0.849 | 0.981 | -0.953 | 0.996 | -0.919 | -0.945 |
| | **WSI** | 0.672 | 0.876 | -0.897 | 0.941 | -0.850 | 0.959 | -0.818 | -0.982 | 0.024 | 0.718 | -0.175 | -0.981 | 0.797 | -0.969 | 1.000 | -0.846 | -0.870 | -0.866 | -0.577 | 0.866 | -0.903 | 0.956 | -0.944 | 0.805 | 0.834 |
| **Cross section** | **DI** | -0.394 | -0.547 | 0.952 | -0.894 | 0.970 | -0.829 | 0.873 | 0.754 | 0.380 | -0.960 | 0.590 | 0.843 | -0.363 | 0.902 | -0.846 | 1.000 | 0.986 | 0.999 | 0.682 | -0.955 | 0.894 | -0.959 | 0.901 | -0.947 | -0.893 |
| | **PE** | -0.531 | -0.639 | 0.989 | -0.948 | 0.996 | -0.893 | 0.941 | 0.768 | 0.431 | -0.901 | 0.460 | 0.895 | -0.396 | 0.886 | -0.870 | 0.986 | 1.000 | 0.993 | 0.783 | -0.991 | 0.846 | -0.977 | 0.871 | -0.886 | -0.826 |
| | **AR** | -0.440 | -0.587 | 0.966 | -0.916 | 0.979 | -0.856 | 0.894 | 0.774 | 0.377 | -0.944 | 0.547 | 0.868 | -0.393 | 0.909 | -0.866 | 0.999 | 0.993 | 1.000 | 0.706 | -0.968 | 0.891 | -0.971 | 0.904 | -0.935 | -0.883 |
| | **P** | -0.754 | -0.604 | 0.834 | -0.817 | 0.834 | -0.772 | 0.935 | 0.425 | 0.742 | -0.494 | 0.013 | 0.717 | -0.131 | 0.472 | -0.577 | 0.682 | 0.783 | 0.706 | 1.000 | -0.853 | 0.350 | -0.715 | 0.418 | -0.410 | -0.297 |
| | **CE** | 0.627 | 0.693 | -0.998 | 0.969 | -0.998 | 0.922 | -0.978 | -0.756 | -0.475 | 0.837 | -0.351 | -0.913 | 0.402 | -0.849 | 0.866 | -0.955 | -0.991 | -0.968 | -0.853 | 1.000 | -0.786 | 0.968 | -0.824 | 0.816 | 0.751 |
| | **CC** | -0.291 | -0.589 | 0.809 | -0.793 | 0.799 | -0.773 | 0.657 | 0.894 | -0.072 | -0.887 | 0.549 | 0.823 | -0.633 | 0.981 | -0.903 | 0.894 | 0.846 | 0.891 | 0.350 | -0.786 | 1.000 | -0.902 | 0.994 | -0.962 | -0.990 |
| | **NC** | 0.604 | 0.760 | -0.981 | 0.975 | -0.965 | 0.949 | -0.916 | -0.887 | -0.244 | 0.855 | -0.357 | -0.960 | 0.583 | -0.953 | 0.956 | -0.959 | -0.977 | -0.971 | -0.715 | 0.968 | -0.902 | 1.000 | -0.934 | 0.883 | 0.861 |
| **Physico-chemical properties** | **pH** | -0.394 | -0.673 | 0.849 | -0.848 | 0.830 | -0.836 | 0.713 | 0.932 | -0.055 | -0.863 | 0.466 | 0.879 | -0.683 | 0.996 | -0.944 | 0.901 | 0.871 | 0.904 | 0.418 | -0.824 | 0.994 | -0.934 | 1.000 | -0.941 | -0.969 |
| | **AC** | 0.163 | 0.421 | -0.821 | 0.757 | -0.844 | 0.697 | -0.678 | -0.757 | -0.144 | 0.978 | -0.725 | -0.739 | 0.401 | -0.919 | 0.805 | -0.947 | -0.886 | -0.935 | -0.410 | 0.816 | -0.962 | 0.883 | -0.941 | 1.000 | 0.983 |
| **Extrusion process** | **SME** | 0.163 | 0.468 | -0.768 | 0.728 | -0.774 | 0.691 | -0.604 | -0.821 | 0.038 | 0.924 | -0.663 | -0.745 | 0.528 | -0.945 | 0.834 | -0.893 | -0.826 | -0.883 | -0.297 | 0.751 | -0.990 | 0.861 | -0.969 | 0.983 | 1.000 |

100% significance
95% significance
90% significance
85% significance
80% significance
75% significance
70% significance

CP - cold peak, RP - raw peak, HP - hold peak, BD - breakdown, FV - final viscosity, SB - setback, PT - peak time, EI - expansion index, DE - density, HD - hardness, L* - lightness, a* - redness, b* - yellowness, WAI - water absorption index, WSI - water solubility index, DI - diameter, PE - perimeter, AR - area, P - porosity, CE - extrudate circularity, CC - cell circularity, NC - number of cells, AC - acidity, SME - specific mechanical energy

**Fig. S2 -** Pearson's correlation

**Table S1 -** Pasting properties of young bamboo culm flour*

| **Parameters** | **YBCF** |
|---|---|
| Cold peak (cP) | 111.33 ± 3.30 |
| Raw peak (cP) | 52.67 ± 1.25 |
| Hold peak (cP) | 34.33 ± 1.70 |
| Breakdown (cP) | 18.33 ± 0.94 |
| Final viscosity (cP) | 73.00 ± 5.10 |
| Setback (cP) | 38.67 ± 3.40 |
| Time (min) | 2.07 ± 0.00 |

*Mean value ± standard deviation.

# CHAPTER VII

## CONTINUOUS MICROWAVE NON-THERMAL PLASMA SYSTEM TO MODIFY STARCH PROPERTIES

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This chapter presents results of commercial starches modified by non-thermal plasma

This chapter was filded as a patente number BR 10 2019 027106 0 in 18/12/2019 (Annex 5)

# Continuous microwave non-thermal plasma system to modify starch properties

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**Abstract:** The techniques used to modify starches are constantly improving as it is evident that innovation, especially in physical treatments, is crucial to meet consumer demands for healthy and sustainable products and industrial process scale-up needs. A continuous non-thermal argon plasma system (NTP) was used to modify the functional properties of native corn, cassava, and potato starches. The changes in starch microstructure, moisture, pH, damaged starch, particle size distribution, gel strength, and pasting properties were assessed, and a t-test ($p<0.05$) was applied to compare the means. All the starch samples showed fissures on their granule surface after treatment. An increase in the damaged starch content of approximately 10% was observed in corn and potato starches. Overall, the NTP treatment decreased moisture content due to the release of water molecules bound to starch and changed pasting viscosity due to crosslinking formation. Also, because of the crosslinking reaction favored during NTP treatment, treated cassava starch formed a stronger gel network. Therefore, NTP can be a viable technology to scale-up for starch modification compared to chemical treatments, as it induces changes in its functionalities without generating waste.

**Keywords:** cold plasma, modified starches, green technology, scale-up

## 1. Introduction

Non-thermal plasma (NTP), also called cold plasma or non-equilibrium plasma, is an innovative and environmentally friendly alternative to modify food structures that does not generate chemical residues, toxic substances, and deleterious effects resulting from heat [1, 2]. NTP is generated when a gas or a mixture of gasses, such as argon, helium, nitrogen, oxygen, or air, receive an electrical discharge under atmospheric pressure or vacuum, inducing the formation of various reactive species, such as atomic oxygen, ozone, hydroxyls, reactive oxygen species (ROS) and reactive nitrogen species (RNS), charged particles, and UV radiation [3, 4].

NTP has demonstrated promising effects on the rheological, functional, and structural properties of starch [1, 5-7]. Starch is an extensively raw material used in the food industry, comprising vast applications, like gelling agent, thickener, stabilizer, and flavor encapsulator. Ideally, native starches would be suitable for all types of applications in food products; however, due to their non-reactive nature and insolubility in cold water, the food industry applies several chemical (e.g. oxidation, succinylation, esterification, etherification, and phosphorylation) [8], enzymatic [9], and physical techniques (e.g. high hydrostatic pressure, non-thermal plasma, ultrasound) [10] to enhance their technological properties. Cross-linking, depolymerization, and etching are the main modifications occurring in starch during plasma treatments that can result in granule surface modifications, reduced viscosity and crystallinity, and increased hydrophilicity [5, 11, 12]. In addition, polar hydrophilic groups introduced during plasma treatment can also lead to starch modification through the interaction between reactive plasma species and starch [13].

Most of the NTP systems used in studies to treat starch were operated in a non-continuous process [11, 14-18]. Although researchers have been investigating the feasibility to scale-up NTP technology [19-22], studies using NTP devices for continuous treatment are scarce in the literature. Moreover, studies to scale-up a plasma reactor for starch modification with higher yield [23] and with integration to existing production lines (e.g. transport, drying, and others) are also crucial for the food industry [24].

Microwave discharge is one of the several methods that can be used to generate the non-thermal plasma. The gas electrons absorb the microwave energy and through inelastic collisions reactive species are formed with high efficiency and with high electron density [23]. Recent studies have shown that the device used in this work modulated the functionality of wheat flour [25] and vital gluten [14,15] when operated in a non-continuous system. To advance the use of this NTP device, a continuous system was designed to expand its applications [26]. Thus, this study aimed to show that it is possible to modify corn, cassava, and potato starches using a continuous microwave non-thermal plasma system.

## 2. Materials and Methods

### *2.1. Raw materials*

Corn starch (Cargill, São Paulo, Brazil), cassava starch (Amafil, Parana, Brazil), and potato starch (Metachem, São Paulo, Brazil) were used in the experimental work.

### *2.2. Continuous Non-thermal plasma treatment*

The continuous microwave non-thermal plasma equipment (Institute of Food Technology, ITAL, Campinas, Brazil) has a non-thermal plasma generation unit for indirect treatment, which works in a continuous single-mode system under atmospheric pressure. It is composed of elements for microwave generation and transmission, with a frequency of 2.45 GHz x 1.9 kW. The unit has a gas supply system and a high-voltage ignitor (1.5 kV) used for plasma formation in a specific accessory. This accessory is connected through a conductive duct to a screw for treating the food matrix, from the continuous post-discharge flow of the NTP generated [26].

In this study, the input power supplier for generating plasma was 200 W and the argon gas flow rate was 10 L/min. Samples (150 g) were treated for 40 min in an environment of 45 ± 1 % RH at room temperature. Rotation of 10 rpm was used in the feed system and the auger. The NTP-treated samples were collected, vacuum-packed in plastic bags, and before starting the analyses, samples were homogenized in a planetary mixer for 5 minutes. Treatments were conducted in duplicates.

### *2.3. Microstructure analyses*

Granule morphology was observed using an optical microscopy (BX51/BX52, Olympus), coupled with a digital camera (Canon PowerShot A620/7.1 megapixels) under polarized light (100x magnification). Starch sample was placed on a glass slide with a drop of distilled water, and covered with a coverslip, which was then placed under an optical microscope.

Scanning electron microscopy (SEM) images of the starch samples were gathered using a TM4000 Plus (Hitachi, Tokyo, Japan) microscope. The samples were mounted on an aluminum stub and coated with a 15 nm golden layer. The SEM operating conditions were: electron beam current = 80 μA, constant acceleration voltage = 15 kV, and working distance = 5.95 mm. Captures were made at 500, 1000 and 3000x in corn and cassava starch, and 500, 1000 and 1500x in potato starch. Granule dimensions, circularity, and diameter were measured using ImageJ software [27].

*2.4. Physicochemical and technological properties*

Moisture, pH, and damaged starch were determined according to methods n. 44-15.06, n. 02.52-01, and n. 76-33.01 [28], respectively. Bulk density was determined according to [29]. Color parameters (L*: brightness, a*: redness-greeness, b*: yellowness-blueness) were measured using a MiniScan HunterLab CR-400 colorimeter, illuminant D 65, and $10^{o}$ observation angle.

*2.5. Particle size distribution*

Particle size distribution was determined using a laser ray diffraction system (LA-950-V2, Horiba Instruments, Hyoto, Japan). The sample dispersion was performed in 99.5% absolute ethanol and the mean diameter was determined according to De Brouckere’s diameter (D [4,3]). The distribution was characterized by the particle's diameter accumulating in the 10% (D10), 50% (D50), and 90% (D90) distribution. Span provided the polydispersity index of the particles, which was calculated according to the Equation 1:

$$\text{Span} = (D90 - D10) / D50 \quad (1)$$

*2.6. Pasting properties*

Rapid Visco Analyser (RVA 4500, Perten Instruments, Australia) was used to determine pasting properties of untreated and treated samples. The moisture content of all samples were adjusted to 14% and they were dispersed in 25 mL of distilled water. The starch suspensions were subjected to the following time-temperature profile at a constant stirring speed of 160 rpm: initial temperature of 25 $^{o}$C for 2 min, heating cycle until reaching 95 $^{o}$C at a constant rate of 14 $^{o}$C /min, maintaining this temperature constant for 5 min and, finally, cooling cycle until reaching 25 $^{o}$C, totaling 20 min.

*2.7. Gel strength*

Starches were gelatinized using the Rapid Visco Analyser (RVA 4500, Perten Instruments, Australia) following the method nº 76.21-01 (AACCI, 2010). The samples were poured into a specific holder and stored at 4 ºC for 18 h. After this period, the samples were left to reach room temperature for 1 h before reading the gel strength. The gel strength was obtained by a Texture Analyser (TA-XT2i, Halsmere, GBR) using a P/10 cylinder probe, set at 5 mm/s of pre-test speed, 1 mm/s of test speed, and 5 mm/s of post-test speed. The gel hardness was determined from the maximum peak force on the curve.

*2.8. Data analysis*

Results were expressed as the mean of three replicates. The physicochemical and technological properties were statistically analyzed by ANOVA and the results were compared by the least significant difference (t test, $p<0.05$) using the software Analysis of Variance for Balanced Data (SISVAR 5.6).

## 3. Results and discussion

*3.1. Microscopy properties*

Figure 1 shows the micrographs of starch samples observed by optical microscopy under polarized light and SEM before and after plasma treatment. It can be noticed that all starches had their Maltese cross unchanged after NPT treatment, which indicates the starches granule remained intact, even with the samples showing visible fissures and cavities on their surfaces. Plasma treatment can alter the starch granule surface morphology by the etching effect, which according to [30] would be more suitable to starch with large granule size than smaller size. However, in our study corn and cassava starches showed more visible surface cavities than potato starch, which was confirmed by the measurements of circularity and solitidy (Table 1).

Plasma etching degree is dependent on the plasma devices, process parameters, and starch sources used. For example, [31] observed cavities on the surface of corn starch with different amylose content after radio frequency plasma treatment but not for dielectric barrier discharge plasma treatment. No changes were observed on the Maltese cross for the red adzuki bean treated in a dielectric barrier discharge plasma device (40 V, 1-10 min) [32]; on granule shape for corn, rice, and potato starches treated in a radio frequency plasma (120 W, 60 min) [33]; or on the surface of corn and tapioca starches treated in radio frequency device (40-60 W, 10-20 min) [18]. In addition, [34, 35] already pointed to a correlation between polymer structure and process parameters to achieve the optimum etching conditions, indicating that microwave plasma reactor was more efficient to favor this mechanism for the materials studied to lower radio frequency.

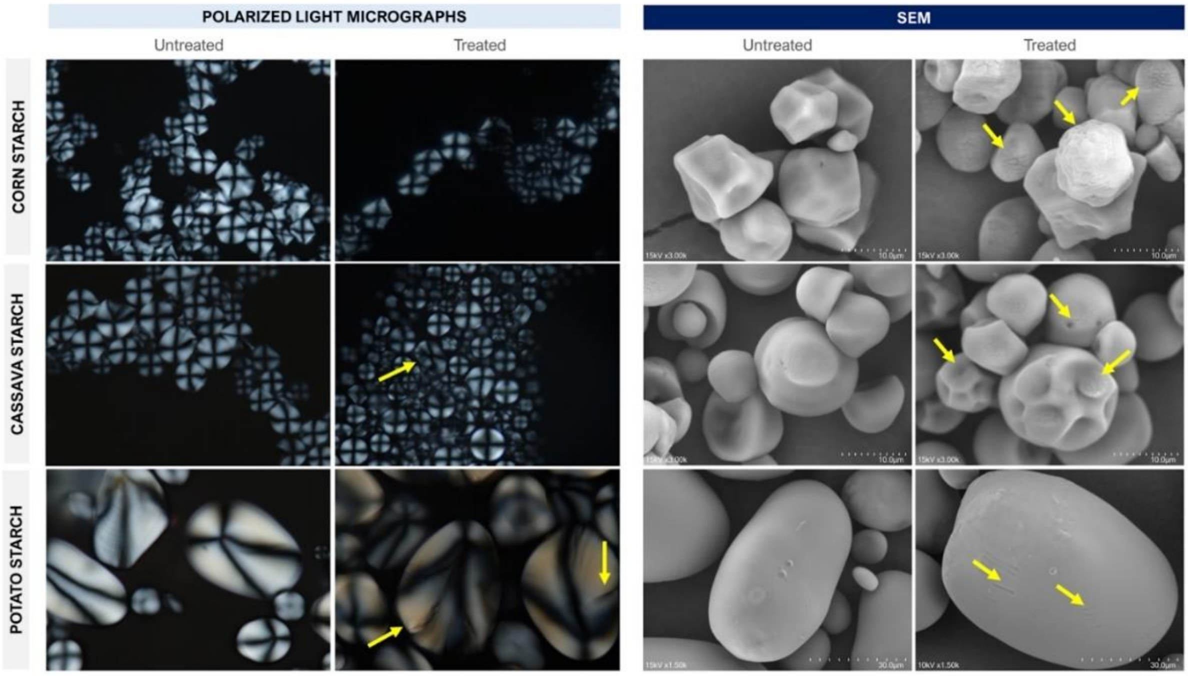


**Figure 1.** Polarized light micrographs (100x) and SEM (at 3000x) of corn, cassava, and potato starches, untreated and treated by non-thermal plasma. Arrows indicate fissures on the starch surface.

### *3.2. Physicochemical and technological properties*

The physicochemical and technological properties of untreated and treated starches are summarized in Table 1. As shown in Table 1, NTP treatment promoted a significant moisture reduction in the treated starches. The water molecules present in the starch are more suitable to degradation by secondary electrons formed, resulting in the breakdown of water molecules, and their subsequent remotion from the system by the carrier gas from the gas discharge during treatment. Similar results were found by [19] and [36] when different starch sources were treated.

The pH of treated cassava and potato starches were not affected after NTP treatment, only treated corn starch showed a significant pH decrease (Table 1). According to [37], the acidic groups, such as carboxyl, carbonyl, and peroxyl, generated by the partial oxidation of starch cause the decrease in pH after plasma treatment. This type of conversion of hydroxyl groups to acidic groups is similar to that observed in chemical modification of starches by oxidation method using chemicals such as hypochlorite, bromine, hydrogen peroxide, and others [11]. Similar decrease in pH after plasma treatment was also found in other studies [15, 18, 36].

Corn and potato starches showed significant increase in damaged starch content after NTP treatment (Table 1). Damaged starch is defined as the starch granule that has been physically damaged in the grain milling process or other processes, and can absorb 5x more water than undamaged starch [38]. Increase in damaged starch content was also observed under atmospheric air [7] and argon plasma treatment [39]. Surprisingly, cassava starch maintained similar content of damaged starch, indicating that the NTP treatment may not have induced homogeneous changes in the granule surface of the sample due to the nature of the plasma device. Initial studies using the same device showed lower percentage of damaged starch in wheat flour [25].

The bulk density of the samples showed no significant difference between untreated and treated samples (Table 1). According to [40], who treated arrowroot starch in a pin-to-plate cold plasma, the starch surface modification by the etching phenomenon can increase the frictional forces between particles, reducing their free movement and leading to a decrease in density. Although it was possible to observe fissures and cavities on the samples, the indirect NTP treatment used in this study reduces the direct contact of the starch with the plasma plume, which depending on the treatment conditions (e.g. power and time exposition), might be gentle enough to promote marked physical changes in the starch.

**Table 1.** Physicochemical and technological properties of untreated and treated starches on continuous non-thermal plasma system[1]

| Properties | Corn | | Cassava | | Potato | |
|---|---|---|---|---|---|---|
| | **Untreated** | **Treated** | **Untreated** | **Treated** | **Untreated** | **Treated** |
| Granule properties | | | | | | |
| Length (µm) | 16.55 ± 0.24 | 15.25 ± 0.81 | 14.23 ± 0.89 | 12.78 ± 0.74 | 126.98 ± 8.62 | 133.45 ± 7.52 |
| Width (µm) | 15.25 ± 0.16 | 14.58 ± 0.27 | 7.81 ± 0.86 | 9.93 ± 0.97 | 95.23 ± 5.84 | 89.52 ± 6.03 |
| Circularity | 0.58 ± 0.07* | 0.38 ± 0.06 | 0.88 ± 0.09* | 0.75 ± 0.05 | 0.82 ± 0.09* | 0.75 ± 0.04 |
| Solidity | 0.95 ±0.09* | 0.81 ± 0.02 | 0.96 ± 0.01* | 0.90 ± 0.02 | 1.00 ± 0.01* | 0.95 ± 0.02 |
| Moisture (%) | 12.17±0.12* | 10.62±0.04 | 12.23±0.06* | 10.46±0.07 | 12.93±0.90* | 10.88±0.50 |
| pH | 4.61±0.02* | 4.52±0.06 | 6.22±0.03 | 6.25±0.02 | 7.44±0.07 | 7.36±0.03 |
| Damaged starch (%) | 4.37±0.08 | 4.77±0.06* | 6.89±0.02 | 6.82±0.00 | 4.25±0.02 | 4.67±0.12* |
| Bulk density (g/mL) | 0.63±0.03 | 0.64±0.01 | 0.66±0.01 | 0.67±0.00 | 0.87±0.00 | 0.88±0.01 |
| Particle size distribution | | | | | | |
| D10% (µm) | 10.06±0.13 | 11.61±0.69* | 8.88±0.12* | 8.19±0.04 | 20.15±0.05 | 21.72±1.21* |
| D50% (µm) | 22.40±0.13 | 24.75±1.11* | 22.07±0.13* | 21.04±0.09 | 35.80±0.20 | 38.06±1.71* |
| D90% (µm) | 39.63±1.52 | 46.25±3.15* | 55.59±3.40* | 39.80±1.38 | 56.34±0.70 | 60.82±2.59* |
| D [4,3] (µm) | 26.12±2.49 | 27.42±1.45* | 31.42±2.51* | 24.03±0.54 | 36.77±0.34 | 39.52±1.73* |
| Span | 1.32±0.06 | 1.40±0.04* | 2.12±0.13* | 1.50±0.06 | 1.01±0.01 | 1.03±0.01* |
| Color | | | | | | |
| L* | 101.40±0.07* | 101.14±0.04 | 100.76±0.06 | 100.01±0.10 | 100.31±0.07* | 99.85±0.19 |
| a* | -0.38±0.01 | -0.42±0.01 | 0.02±0.02 | 0.14±0.06* | -0.13±0.03 | -0.12±0.03 |
| b* | 3.92±0.08 | 3.91±0.05 | 1.47±0.05 | 1.43±0.07 | 2.07±0.07 | 2.20±0.06 |
| Gel strength (N) | 1.96±0.16 | 2.26±0.04 | 0.30±0.02 | 0.36±0.01* | 1.01±0.06 | 1.16±0.04* |

[1]Mean ± standard deviation

*Significant by t-test ($p<0.05$) between untreated and treated sample

NPT treatment showed a slight effect on color parameters for the treated samples. After treatment, corn and potato starches exhibited a slightly reduction in brightness (L*), and cassava starch showed higher redness (a*) compared to their controls (Table 1). [19] also observed a similar behavior when treating cassava starch in low pressure argon semi-continuous plasma. The nature of the pigments formed on the starch surface remains unknown [41], but no negative impacts were reported in the literature. The color of starch plays an important role in many food applications, where whiteness is a desirable attribute; therefore, this represents an advantage of using non-thermal plasma.

### *3.3. Particle size distribution*

Particle size of starch granules varies according to their botanical origin, having a range for corn, cassava, and potato between 5 and 30 μm, 4 and 35 μm, and 5 and 100 μm, respectively [42]. In addition, they may differ according to cultivar, production site, and processing.

In the present study, starch size was determined in alcoholic suspensions. The mean diameter (D4,3) of the native starches was between 26.12 and 39.52 μm, similar to that found by [43], who obtained values ranging from 10.1 to 33.6 μm. However, the treated samples showed significant difference in all parameters compared to their respective control (Table 1). Treated corn and potato starches increased the values for the particle size parameters, whilst treated cassava starch had an opposite behavior. The larger particles can be attributed to starch hydration and swelling capacity after treatment. The reduction in D4.3 indicates that treated cassava starch can disperse and solubilize easily during gelatinization, leading to more dissociated starch molecules, which can also contribute to paste clarity.

### *3.4. Pasting properties*

Table 2 shows the RVA pasting parameters of untreated and treated samples in heating, holding, and cooling cycles. All starches had their pasting profile modified after NTP treatment, with the corn starch more significantly affected.

According to the findings (Table 2 and Figure S1 – Supplementary material), the modification caused by NTP treatment resulted probably in both cross-linking and depolymerization reaction, with predominance of one of these mechanisms in the different starch sources. Depolymerized starch can present low viscosity, as it becomes more prone to absorb water due to the cleavage of glycosidic linkages during NTP treatment. The results obtained for treated corn starch suggests that depolymerization was dominant, impacting in all pasting parameters. [44] and [45] also observe a decrease in viscosity for treated corn starch in different cold plasma devices. Treated potato starch also seems to be impacted by the depolymerization reaction that had led to a decrease in hold peak, which represents the minimum viscosity of the starch at constant temperature (95 °C). The decrease in hold peak can also be attributed to the fissures observed in Figure 1.

On the other hand, cross-linked starch can present low swelling capacity and raw peak viscosity depending on the cross-linking degree, as it becomes more resistant to gelatinization and heat when highly cross-linked, but at certain low cross-linking degree, the viscosity can increase [46]. The significant increase in raw peak (maximum viscosity at 95 °C with maximum swelling capacity without granule disruption), and hold peak (minimum viscosity of the starch at constant temperature 95 °C) for treated cassava starch suggests that cross-linking was dominant during plasma treatment. [40] reported similar behavior for arrowroot starch, as a consequence of enhanced swelling power capacity and starch molecular weight by cross-linking reaction.

Besides the physical treatment processing parameters and starch source, the pasting properties of starches are influenced by the amylose/amylopectin ratio, presence of lipids, and granule size distribution [47]. The observed decreases in pasting viscosities for treated corn starch can be also related to amylose-lipid complex formation, which according to [48] can restrict the starch granule disruption and consequently reduce the amylose leaching into the medium. This complex is more likely to occur in cereal starches, since they have more lipids than tuber starches [49], a fact that did not occur with cassava and potato starches. Therefore, if this reaction was dominant in the process, the swollen starch granules could be strengthened by the covalent cross-linked bonds between the starch chains, and consequently, the loss of viscosity under high temperature and shear conditions would be minimized, resulting in a reduction in values, as found for corn starch.

Thus, it can be assumed that NTP treatment improved the starches stability and reduced the tendency of retrogradation. Moreover, NTP demonstrates the possibility to adjust the process according to the modification desired.

**Table 2.** Pasting properties of untreated and treated starch samples on continuous non-thermal plasma system.

| **Parameters** | **Corn** | | **Cassava** | | **Potato** | |
|---|---|---|---|---|---|---|
| | **Untreated** | **Treated** | **Untreated** | **Treated** | **Untreated** | **Treated** |
| **Cold Peak (cP)** | 71.50±2.50* | 71.25±1.25 | 73.00±0.00* | 70.75±1.75 | 70.00±1.00 | 73.00±3.50* |
| **Raw Peak (cP)** | 2817.50±15.50* | 2560.92±235.42 | 4448.00±2.00 | 4475.75±40.75* | 12100.50±4.50 | 11993.00±5.00 |
| **Hold Peak (cP)** | 1720.50±67.50* | 1618.67±94.67 | 1544.50±3.50 | 1589.25±11.25* | 1888.50±1.50* | 1649.75±6.25 |
| **Breakdown (cP)** | 1097.00±52.00* | 942.25±140.75 | 2903.50±5.50 | 2886.50±29.50 | 10212.00±3.00 | 10343.25±11.25 |
| **Final Visc (cP)** | 6298.50±35.50* | 5490.08±756.58 | 4187.50±25.50 | 4089.25±1.75 | 5984.00±145.00 | 5964.75±20.75 |
| **Setback (cP)** | 4578.00±103.00* | 3871.42±661.92 | 2643.00±29.00* | 2500.00±13.00 | 4995.50±146.50 | 4315.00±27.00 |
| **Peak Time (min)** | 7.70±0.03 | 7.76±0.11* | 6.40±0.00 | 6.44±0.02 | 5.63±0.03 | 5.73±0.07* |

[1]Mean ± standard deviation

*Significant by t-test ($p<0.05$) between untreated and treated sample

*3.5. Gel strength*

The results of gel strength of treated and untreated starches are presented in Table 1. Treated cassava and potato starches showed higher gel strength, from 0.30 N to 0.36 N and from 1.01 N to 1.16 N after treatment, respectively. The amylopectin and amylose molecules are rearranged forming a more crystalline and ordered structure. At this point a gel is formed during cooling, but the gel strength will be determined by amylose-amylopectin ratio, amylose-lipid complex, granule shape, and ionic strength [50]. This means native starches containing high amylose content have the capacity to form stiff gels, whereas native starches containing high amylopectin content form soft gels.

In modified starches, cross-linking reaction induced by NPT treatment can increase the gel stiffness. [5] also observed an increase in gel strength for cassava starch after plasma treatment due to cross-linking. Although treated potato starch seemed to be more affected by depolymerazation reaction, the fissures on the granule surface can allow the penetration of plasma ions inside the granule structure and resulted in cross-linking between amylose chains [44], which could explain the increase in gel strength. In addition, potato starch has high phosphorus content compared to the other starches, which can favor cross-linking reactions.

## 4. Conclusion

The indirect microwave argon non-thermal plasma (NTP) treatment showed favorable results to modify starch physicochemical properties without using chemical reagents. NTP treatment significantly modified granule surface, increased the damaged starch contents of treated corn and potato starches and statistically reduced the moisture of all samples. The gel strength of cassava and potato starches was also improved. The results suggest that NTP modified the treated starches through etching phenomenon, cross-linking and depolymerization reactions, which can also be observed in a non-continuous treaments. Therefore, argon plasma treatment was an innovative alternative to conventional starch modification and a pespective for future scale-up. From an industrial point of view, this continuous NTP system can be also for several other types of food powders.

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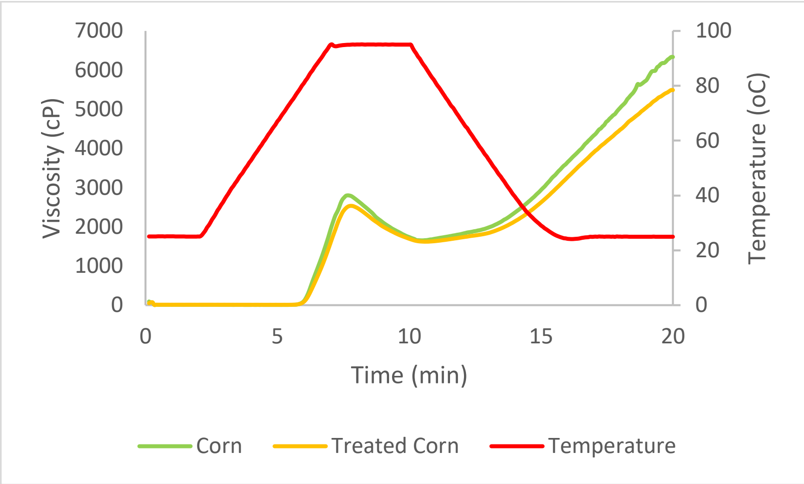
## Supplementary material

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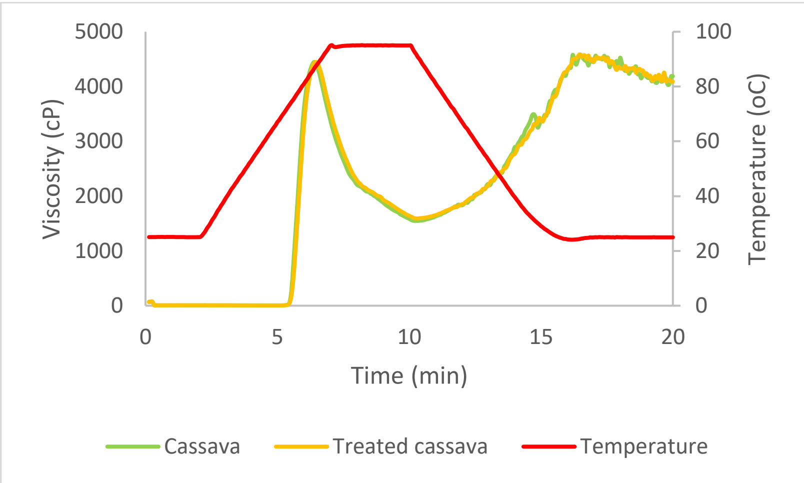


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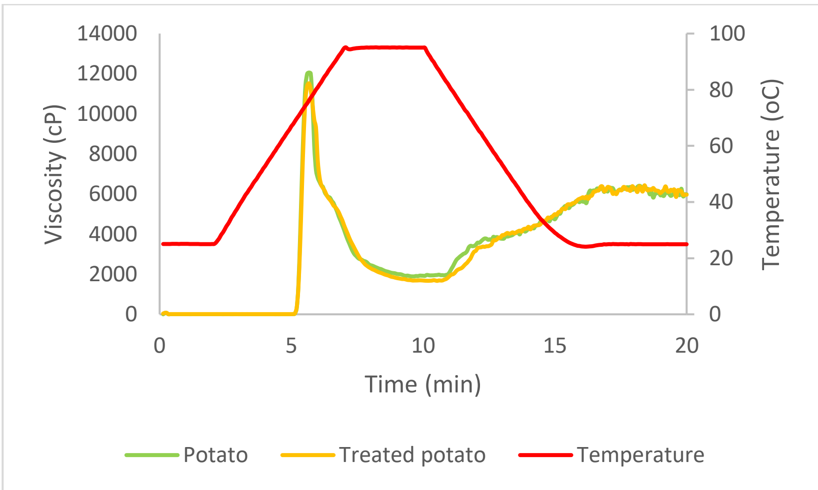


(c)

**Figure S1 –** Viscosity profile of corn (a), cassava (b), and potato (c) starches before and after non-thermal plasma treatment.

# CHAPTER VIII

## GENEREAL DISCUSSION

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This chapter presents a brief discussion of the most important results obtained in this research, reagarding the extrusion and non-thermal plasma treatment

## DISCUSSION

The aim of this study was to evaluate and characterize the modifications resulting from the extrusion cooking and non-thermal plasma processes in starch and fiber source raw materials, as well as to present and expand the application of a new dietary fiber source, the young bamboo culm flour.

The first three chapters summarized the main aspects of starch, since its sources, chemical composition, properties characterization, and physical process of modification. These chapters provided support for the theoretical basis of the study, as well as definition of the physical process to modify the raw materials. As observed, the extrusion cooking is already a well established procedure with known effects in starch, but the variety of parameters and combination of different raw materials still allow researchers to innovate. And in the case of the non-thermal plasma technology, it was possible to observe the increase in interest in this technique due to the eco-friendly appeal, but the lack of data regarding scale-up. Therefore, both technologies could be used to explore possibilities and provide scientific data for the industry.

The fourth chapter summarized the main aspects of bamboo as a raw material for food and feed products. According to this review, the main applications of bamboo fiber in the food area were focused on improving technological properties of dairy, meat, and starchy products, such as water holding capacity, or nutritional properties, such as fat replacement. However, it was not mentioned in the literature any application of bamboo fiber in extruded products, giving us an opportunity to use and study this raw material in a conventional thermal process, having the data presented in chapter five.

Considering that the young bamboo culm is a new source of fiber for food application, adjustment in the flour processing is required, however, the harvest of young bamboo culm up to one year old allowed the use of the same mills used for cereals grains. Bamboo species, age, and month of harvest interfere directly in its chemical composition, but in general it is possible to obtain above 70% of dietary fibers, as observed in other studies (FELISBERTO et al., 2018; FELISBERTO et al., 2017). The fiber composition of YBCF comprises xylans and cellobiose, which have the potential to act as prebiotics. Bifidogenic growth was detected in the raw material and also after thermal treatment, showing that the rice flour may have helped to preserve YBCF functionality. To confirm YBCF functionality, short chain fatty acids (SCFA) can be determined.

Another interesting characteristic of YBCF was the effect of its particles that increased the torque and specific mechanical energy during the extrusion cooking, leading to high starch conversion of the rice flour, as evidenced by the pasting properties of the samples. Previous study conducted by Robin et al. (2011) showed that there is a minimum amount of fiber and particle size to increase the local mechanical stress and consequently the starch conversion, which in our study could be observed with 10% fiber content and a particle size above 500 µm.

The last chapter, chapter six, showed the potential of a continuous non-thermal plasma system to modify different sources of starch. The modifications occurred through the cross-linking and depolymerization, allowing the reduction of moisture, formation of fissures and cavities on the granule surface that can promote fast enzyme action and hydration. These modifications were observed in the literature, however, most of the studies were conducted in small batches sizes. Therefore, the equipment allowed similar modifications with the advantage of scale-up.

# CAPÍTULO IX

## CONCLUSION

The previous chapters have been summarized in order to highlight the technical information considered of most importance

## CONCLUSION

The young bamboo culm has proven to be a source of dietary fibers with a potential for a wide range of functional applications in different food matrices. In extruded products, it contributed with prebiotic activity in addition to the insoluble fiber intake. The content above 80% of dietary fiber and the abundance of bamboo in the Brazil, makes the young bamboo culm an essential raw material for the development of local economy, which has already been receiving incentive from the Brazilian Government, through the Law 12.484 that established the National Policy of Incentive to the Sustainable Management and Cultivation of Bamboo.

The partial replacement of rice flour with young bamboo culm flour (YBCF) led to a reduction in expansion and an increase in hardness and density. However, such aspects were expected considering the nature of bamboo fibers. At the same time, the characteristics of the YBCF particles favored a higher starch conversion during the extrusion cooking due to the increase in particle-particle friction, which led to an increase of specific mechanical energy. Furthermore, studies on thermal stability of YBCF and sensory are required to obtain a functional and tasty ready-to-eat product.

As an emergent non-thermal technology, the continuous plasma system developed by the Institute of Food Technology (ITAL) showed potential to be scaled-up. The combination of different techniques to create the new system allowed the modification of starches through cross-linking and depolymerization reactions, improving, for example, the gel strength of cassava and potato starches. The continuous system has also shown potential not only for starch modification but also for any powdery ingredient, therefore, expanding its application in different fields.

# CHAPTER X

## REFERENCES

This chapter presents all references used for the general introduction and discussion of the project

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# CHAPTER XI

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