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JUVER ANDREY JIMENEZ MORENO

EXTRACTION AND HYDROLYSIS IN AN AUTOMATED IN-LINE REACTOR AS A TECHNOLOGY FOR THE VALUATION OF CAMBUCI (*Campomanesia phaea* Berg.*)* PEEL

EXTRAÇÃO E HIDRÓLISE EM REATOR AUTOMATIZADO IN-LINE COMO TECNOLOGIA PARA A VALORIZAÇÃO DA CASCA DE CAMBUCI (*Campomanesia phaea* Berg.*)*

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Dissertation presented to the Faculty of Food Engineering of the University of Campinas in partial fulfillment of the requirements for the degree of Master in Food Engineering.

Dissertação apresentada à Faculdade de Engenharia de Alimentos da Universidade Estadual de Campinas como parte dos requisitos exigidos para a obtenção do título de Mestre em Engenharia de Alimentos.

Orientadora: Profa. Dra. Tânia Forster Carneiro

Co-orientador: Prof. Dr. Mauricio Ariel Rostagno

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I dedicate this work to my family, who have been a constant source of inspiration and have always provided me with their unconditional support.

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RESUMO

O cambuci (*Campomanesia phaea* O. Berg) é um fruto nativo da Mata Atlântica brasileira, com diversos compostos bioativos que despertam interesse nas indústrias cosmética, farmacêutica e alimentar. O objetivo deste trabalho foi analisar os efeitos da extração e hidrólise utilizando água subcrítica na obtenção de açúcares e compostos bioativos provenientes da casca do Cambuci, por meio da aplicação de um sistema automatizado com análise in-line. Uma análise bibliométrica foi elaborada para avaliar pesquisas entre os anos de 2003 e 2022. Os resultados indicaram que os tópicos mais importantes se concentram em compostos fenólicos, capacidade antioxidante, flavonoides, compostos bioativos e desidratação osmótica. O processo de extração e hidrólise da casca de cambuci foi realizado usando um Delineamento Composto Central Rotacional (DCCR) com duas variáveis independentes (2^2), conduzidos no sistema automatizado in-line em diversas condições de extração e hidrólise, abrangendo temperaturas entre 40 e 160 ºC e faixa de pH de 4 a 7, com pressão de 15 MPa e uma vazão de 2 mL min^{-1}. Os resultados obtidos permitiram observar que a temperatura exerceu influência sobre a resposta dos compostos fenólicos totais e a capacidade antioxidante dos extratos e hidrolisados. Os valores máximos para TPC foram de 12.76 mg de ácido gálico equivalente (GAE) g^{-1} e 12.44 mg g^{-1}, utilizando temperaturas de 160 e 143°C, respectivamente. No caso da capacidade antioxidante, os valores máximos forma de 118.07 µmol de capacidade antioxidante equivalente ao Trolox (TEAC) g^{-1} (160°C) e 107.28 µmol TEAC g^{-1} (143°C). Por outro lado, a variação da escala de pH demonstrou efeito mínimo nas variáveis analisadas. Adicionalmente, por meio da análise in-line, foi possível determinar que o período de 42 minutos é adequado para a conclusão do processo de extração e hidrólise da casca do cambuci, o que permitiu estabelecer que a extração com água subcrítica é uma alternativa para a valorização e incorporação na economia circular deste subproduto.

Palavras-chave: *Biorrefinaria; Compostos bioativos; Economia circular; Extração; Frutas nativas; Hidrólise.*

ABSTRACT

Cambuci (*Campomanesia phaea* O. Berg) is a native fruit of the Brazilian Atlantic Forest, containing various bioactive compounds that have sparked interest in the cosmetic, pharmaceutical, and food industries. The objective of this work was to analyze the effects of extraction and hydrolysis using subcritical water on obtaining sugars and bioactive compounds from cambuci peel, utilizing an automated system with in-line analysis. A bibliometric analysis was conducted to evaluate research from 2003 to 2022. The results indicated that the most important topics focus on phenolic compounds, antioxidant capacity, flavonoids, bioactive compounds, and osmotic dehydration. The extraction and hydrolysis of cambuci peel were performed using a Central Composite Rotational Design (CCRD) with two independent variables (2^2), conducted in an automated in-line system under varying extraction and hydrolysis conditions, including temperatures ranging from 40 to 160 ºC, a pH range of 4 to 7, a pressure of 15 MPa, and a flow rate of 2 mL min^{-1}. The results showed that temperature significantly influenced the total phenolic compounds (TPC) and antioxidant capacity of the extracts and hydrolysates. The maximum TPC values were 12.76 mg gallic acid equivalent (GAE) g^{-1} and 12.44 mg g^{-1}, achieved at temperatures of 160°C and 143 °C, respectively. For antioxidant capacity, the highest values were 118.07 µmol of Trolox equivalent antioxidant capacity (TEAC) g^{-1} at 160 °C and 107.28 µmol TEAC g^{-1} at 143 °C. On the other hand, variations in pH had minimal effect on the analyzed variables. Additionally, in-line analysis indicated that a 42-minute period is sufficient for completing the extraction and hydrolysis process of cambuci peel, confirming that subcritical water extraction is a viable alternative for the valorization and incorporation of this by-product into the circular economy.

Keywords: *Biorefinery; Bioactive compounds; Circular economy; Extraction; Hydrolysis; Native fruits.*

CONTENT

CHAPTER 1 - General introduction, objectives, and dissertation structure

1.1. General introduction

The absence of proper protection and inadequate food handling throughout the supply chain leads to a significant increase in food losses and waste (FACCHINI et al., 2023). According to the Food and Agriculture Organization (FAO), losses in the food chain are directly linked to a reduction in food from harvest to consumption. This is attributed to various factors, including inefficient pest and disease controls, issues during the harvest, and suboptimal packaging, storage, and transportation conditions affecting food preservation (FAO, 2013). On the other hand, waste refers to safe food that goes unused for human consumption due to the decisions and actions of retailers, suppliers, and consumers.

Annually, food losses and waste surpass 1,300 million tons, constituting approximately 30% of the total global production (FAO, 2022). In essence, approximately one-third of the world's produced food goes unused, resulting in waste and generating negative impacts at economic, productive, and social levels. In Latin America, losses and waste amount to 127 million tons per year, which is enough to meet the nutritional needs of 300 million people. This food waste is directly linked to resource wastage, as factors such as water, the use of fertilizers and pesticides, energy, seeds, and labor are intricately connected to agriculture (DHIMAN; MUKHERJEE, 2022; MUTH et al., 2019). Therefore, it is imperative to explore alternatives that enable the reuse of food waste and prevent its disposal in landfills, thereby mitigating environmental issues (YOUSEFLOO; BABAZADEH, 2020). One technology that could contribute to waste reuse is biorefinery, as it enables the extraction of compounds with high added value (GIANICO et al., 2021).

Brazil boasts a wide array of native fruit plants within its territory; however, the majority are underestimated and underutilized (NASCIMENTO et al., 2022). Some native Brazilian fruits have significant potential for application in the cosmetic, pharmaceutical, and food industries due to their high biotechnologically relevant compounds (ARAÚJO et al., 2018). Furthermore, these fruits are rich in dietary fibers, unsaturated fatty acids, carotenoids, phenolic compounds, and exhibit a high antioxidant capacity (NASCIMENTO et al., 2022). The cambuci (*Campomanesia phaea* Berg.*)*, one of Brazil's native fruits, holds significant potential for applications in the cosmetic, pharmaceutical, and food industries, given its status as a rich source of phenolic compounds (SANCHES AZEVEDO et al., 2017), calcium, phosphorus, magnesium, potassium, sodium, and vitamin C (DONADO-PESTANA et al., 2018). Typically fleshy, with a sweet aroma and acidic taste, this fruit is primarily utilized in the production of jellies, jams, juices, ice creams, and liqueurs. Moreover, it boasts a pulp yield of approximately 80% (DONADO-PESTANA et al., 2018, 2021; TAVER et al., 2022). The cambuci tree is primarily found in the states of Minas Gerais and São Paulo (SANCHES AZEVEDO et al., 2017; VALLILO et al., 2005), and its fruits have a diameter ranging from 5 to 6 cm in the middle region and a thickness between 3 and 4.5 cm (VALLILO et al., 2005). Furthermore, its annual production can reach up to 300 tons, with 18.5% corresponding to the peel (DONADO-PESTANA et al., 2018).

Given that by-products, such as the peel, are generated during the industrial processing of cambuci, it is worthwhile to explore this residue to identify the bioactive compounds that can be extracted and to assess the prospects for their application. The extraction of these compounds requires the use of suitable and efficient solvents, with methanol, ethanol, acetone, and water being among the most employed (KUMAR; SRIVASTAV; SHARANAGAT, 2021). However, to achieve an extraction without loss of activity, ensuring high purity and maximum yield, it is crucial to consider the specific conditions of each method (BUNMUSIK et al., 2022). Over the last five decades, new extraction techniques have been developed with the goal of reducing the use of organic and synthetic chemicals, shortening extraction times, and enhancing the yield and quality of the extract (JHA; SIT, 2022). Among the new extraction technologies are ultrasound-assisted extraction (MIHAYLOVA; LANTE, 2019; SOMWONGIN et al., 2023), pulsed electric fields (BARBA et al., 2015; QUAGLIARIELLO et al., 2016), enzyme-assisted (GLIGOR et al., 2019), microwave-assisted (CHAN et al., 2011), pressurized liquid (CAO et al., 2021; CARABIAS-MARTÍNEZ et al., 2005) and supercritical fluid (VARDANEGA; OSORIO-TOBÓN; DUBA, 2022). On the other hand, chemical hydrolysis can be employed; however, this process generates solid waste, and there is a possibility of sugar degradation afterward. Another option is enzymatic hydrolysis, which avoids solid waste generation. However, it is a slow process with high energy consumption, and recovering and reusing the enzymes can be challenging (PRADO et al., 2016). Due to these advantages, hydrolysis in subcritical water has garnered more attention as a clean and rapid method. It eliminates the need for pretreatment, features a shorter reaction time, reduces corrosion, minimizes waste generation, and avoids the use of toxic solvents (PRADO et al., 2016; ZHAO et al., 2012).

By integrating an online detector into the extraction and hydrolysis cycle in subcritical water, real-time monitoring of the process becomes possible. This enables the determination of the optimal cessation point for extraction or hydrolysis, indicating the completion of the process. However, to establish the advantages of an automated in-line system for the valorization of various raw materials, it is essential to conduct multiple analyses with different samples. Hence, this work aims to explore the utilization of an automated in-line reactor for the valorization of byproducts, exemplified by cambuci peel.

1.2. Objectives

1.2.1. General objective

This project aimed to study the extraction and hydrolysis of cambuci peel (*Campomanesia phaea* Berg.) for the valorization of this agro-industrial sub-product, utilizing subcritical water in an automated in-line reactor.

1.2.2. Specific objectives

- ✓ To conduct a bibliometric review to identify and analyze trends in research on a native fruit such as cambuci, over the last 20 years;
- ✓ To optimize the operating conditions (temperature and pH of the mobile phase) for the extraction/hydrolysis of cambuci peel;
- ✓ To determine the optimal time for the extraction and hydrolysis process of cambuci peel through in-line analysis.

1.3. Dissertation structure

This document is organized into six chapters, with specific sections dedicated to bibliometric analysis and experimental results. These chapters correspond to articles submitted to indexed journals in the field of Food Engineering.

In **Chapter 1**, the introduction unfolds, presenting the main topic of the dissertation. This section also outlines the general and specific objectives and concludes with an overview of the dissertation's structure.

In **Chapter 2**, the results of the bibliometric analysis conducted using VOSviewer and Bibliometrix software are presented. The data, sourced from the Scopus© database, encompasses the evolution of publications, research areas, main keywords, and the most cited publications. Additionally, the chapter features a comprehensive bibliometric study covering authors, journals, institutions, and countries. Finally, it concludes with a concise review of the source, composition, industrial processing, and perspectives of cambuci.

The results of the bibliometric analysis have been published in the journal 'European Food Research and Technology'. The article, titled 'An overview of the ellagic acid and proanthocyanidins' polyphenols from cambuci (*Campomanesia Phaea Berg*): Myrtaceae's family' is authored by Juver Andrey Jiménez Moreno, Vanessa Cosme Ferreira, Larissa Castro Ampese, Leonardo de Freitas Marinho, Mauricio Ariel Rostagno, and Tânia Forster Carneiro.

In **Chapter 3**, the experimental results of the extraction and hydrolysis from the cambuci peel are presented. The article 'Obtaining Extracts and Hydrolysates from Cambuci Peel Through Subcritical Water: An In-line Detection Approach', whose authors are Juver Andrey Jiménez Moreno, Leonardo de Freitas Marinho, Letícia Sanches Contieri, Tiago Linhares Cruz Tabosa Barroso, Mauricio Ariel Rostagno e Tânia Forster Carneiro was published in the journal 'Food and Bioprocess Technology '.

In **Chapter 4**, the general discussions of the dissertation are presented.

In **Chapter 5**, the principal findings of the dissertation are delineated, accompanied by recommendations for prospective research within the field.

In **Chapter 6**, the references utilized in the development of this scholarly manuscript are delineated.

CHAPTER 2 – An overview of the ellagic acid and proanthocyanidins' polyphenols from cambuci (Campomanesia Phaea Berg): Myrtaceae's family.

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An overview of the ellagic acid and proanthocyanidins' polyphenols from cambuci (*Campomanesia Phaea Berg*): Myrtaceae's family

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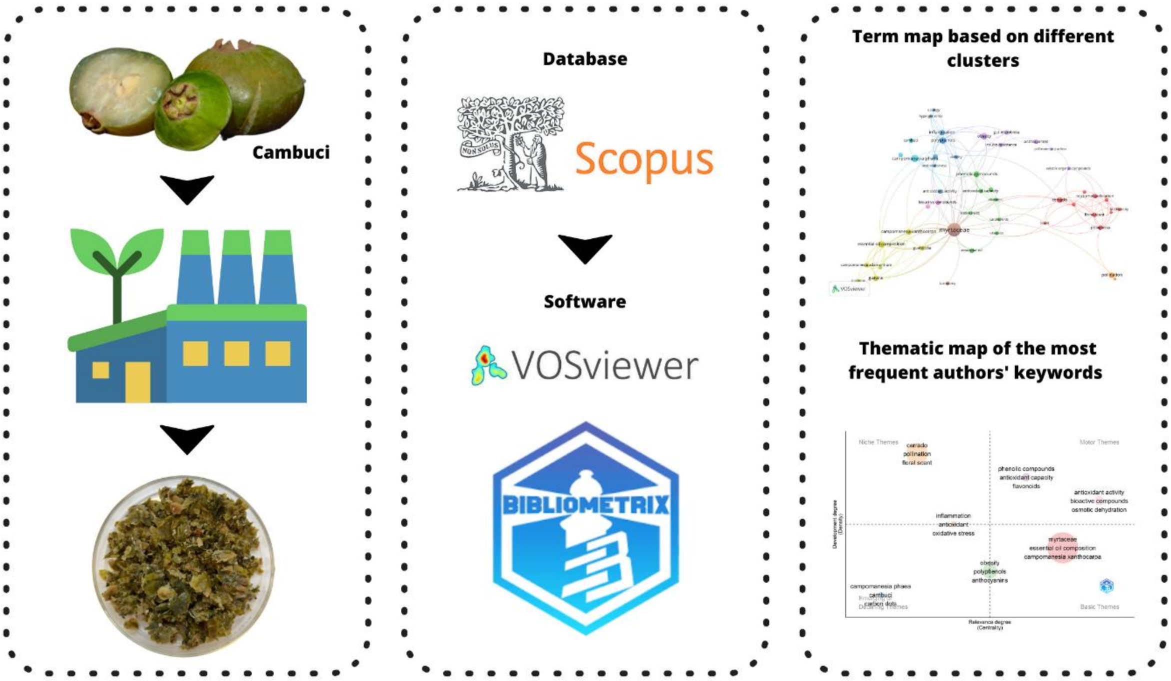
Graphical abstract



Abstract

Cambuci (*Campomanesia phaea Berg.)* is a native fruit to the Atlantic Forest biome of Brazil with several bioactive compounds, ascorbic acid, phenolic compounds, volatile compounds, tannins, and carotenoids and it is used mainly to produce jellies, ice cream, juices, and alcoholic beverages. This review and bibliometric analysis establish the progress, trends, and perspectives of research on this fruit. The results show an increase in publications is related to the emergence of this new research and demonstrate the potential growth of cambuci in different studies. Between 2003 and 2022, 153 articles and 22 reviews related to cambuci were published. The bibliometric analysis revealed that the main research area was *Agricultural and Biological Sciences*, and the main country producing research on the cambuci fruit was Brazil, with 118 articles published. Given that most publications are associated with Brazil, the foremost institutions dedicated to cambuci research are also Brazilian. The list of affiliations is spearheaded by the University of São Paulo and comprises eight other Brazilian organizations, including seven universities and one research institution (Embrapa). The author's keywords revealed that phenolic compounds, antioxidant capacity, flavonoids, antioxidant activity, bioactive compounds, and osmotic dehydration are motor themes in cambuci research. Additionally, ellagic acid is a bioactive phenolic compound in tannins, and proanthocyanidins are compounds of great relevance in cambuci and have great benefit for human health with antibacterial, antifungal, antiviral, anti-inflammatory, antidiabetic, gastroprotective, antidepressant activity. In conclusion, the review establishes that cambuci is an interesting source for its application in the food and pharmaceutical industries.

Keywords: *Native fruits; Natural products; Scientometrics; VOSviewer; Bioactive compounds; Food products.*

Introduction

Brazil is one of the most biodiverse countries in the world, with more than 40,000 different species, which equals 20% of the world's biodiversity [1]. For instance, fruit production in Brazil is 45 million tons per year, which makes Brazil the largest producer of fruit in the world. However, many fruits are unknown and explored despite having a high content of bioactive compounds [2]. Due to the large extension of the territory, it is possible to find many climates that generate many biomes that favor the appearance of different native fruits [3]. In addition, this allows the introduction and adaptation of various exotic fruits [4, 5]. The Myrtaceae family has excellent ecological and commercial importance, since it is one of the main families of fruit trees in the world, with 140 genera and approximately 5800 edible species, which are distributed in tropical and subtropical areas [6, 7]. The Myrtaceae family includes some fruits, such as *Psidium cattleianum* (araçá), *Myrciaria caulifora.* (jabuticaba), *Eugenia unifora* (pitanga), *Eugenia dysenterica DC.* (cagaita), *Campomanesia phaea Berg.* (cambuci), and *Myrciaria dubia McVaugh* (camucamu), among others [3, 5].

Cambucí (*Campomanesia phaea Berg.)* is a Brazilian Atlantic Rainforest biome fruit that has a high productive potential, since it has a wide variety of attributes that allow it to follow the trends of Brazilian consumers, such as wellness and health, sustainability, ethics, and high sensory quality [8]. The cambuci tree grows mainly in Serra do Mar, Minas Gerais, and São Paulo States of Brazil [9–11]. The fruits are fleshy, succulent, and edible and have a diameter between 5 and 6 cm in the middle region and a thickness between 3 and 4.5 cm [10]. The cambuci is juicy and has a sweet smell and a somewhat acid taste, which is why it is used in small proportions for the preparation of jams, jellies, ice cream, and even liqueurs, and has a pulp yield of about 80% [3, 8, 12]. Moreover, it is an essential source of phenolic compounds [9, 13]. Its annual harvest can reach 300 tons. However, it varies each year. Also, this is an approximation, because most cambuci fruits are harvested in periods of low demand [14].

Cambuci fruit has various bioactive compounds, ascorbic acid, phenolic compounds, volatile compounds, tannins, and carotenoids [8]. In Cambuci extracts, quercetin derivatives have been identified in concentrations ranging from 3.53 to 6.44 μg/mL, free ellagic acid in the range of 9.57 to 16.98 μg/mL, and total ellagic acid between 1312 and 2400 μg/mL [14]. On the other hand, Sanchez–Azevedo et al. [9] identified rutin in concentrations ranging from 0.048 to 0.105 g/kg (dry weight) for fruits from Rio Grande da Serra, Mogi das Cruzes, Paraibuna, and Paranapiacaba. Similarly, Epigallocatechin Gallate (EGCG) was found in concentrations between 0.099 and 0.138 g/kg. Phenolic compounds in cambuci are important in counteracting metabolic complications related to obesity, as they improve glucose tolerance and reduce fasting blood glucose and insulinemia [14]. In addition, the bioactive compounds in cambuci positively affect anti-inflammatory and antimicrobial action [15].

Compounds like ellagic acid have both medicinal and nutritional applications. However, in recent years, there has been a growing industrial interest in using this compound to synthesize bioengineering materials [16]. Furthermore, in line with the suggestions of Frayne et al. [17], nanocomposites utilizing ellagic acids (EA) have the potential for applications in environmental pollutant degradation, particularly in removing toxic aromatic compounds. On the other hand, proanthocyanidins could generate interest in the food industry, because they manage to efficiently reduce acrylamide, which could increase the shelf life of foods, as suggested by Sáyago–Ayerdi et al. [18].

The composition of the cambuci fruit is approximately 80.5% pulp, 18.5% peel, and 1% seeds [10]. The pulp generates interest in the cambuci's industrial processing, since it has a high yield (fibers), total soluble solids, and titratable acidity [19]. However, the peel and seeds have not been extensively studied. 25–30% of by-products are produced and discarded while processing fruits and vegetables, mainly seeds, peel, bark, and pomace [20]. The by-products are widely produced in the world. It has dietary fiber and many bioactive compounds (organic acids, minerals, phenolic compounds, and sugars). However, most are rejected and discarded [21, 22]. Nevertheless, due to overproduction and inadequate sustainable management of by-products, they end up in landfills, associated with various social, economic, and environmental problems [23]. For instance, the environment receives Greenhouse Gas emissions, because these organic materials are disposed of in landfills or burned, contaminating water, air, soil, and other foods [24].

Bagasse or seeds could be used in nutraceutical and pharmaceutical areas or can even be used in livestock feed products [25]. However, the conventional extraction techniques for different compounds require a large volume of solvents, which can be dangerous. On the other hand, they have low selectivity extraction yields and provide short extraction times. The extraction of bioactive compounds from byproducts must maximize the extraction yield, satisfy the demand of the industry, keep the bioactive compound free of impurities and toxic compounds, and avoid compound deterioration during the extraction process [26, 27].

Bibliometric analysis has become a tool to determine trends and gaps in research areas [28]. The use of this method has allowed for elucidating the trends and perspectives of research in Uvaia (*Eugenia pyriformis Cambess— Myrtaceae*) [29], jabuticaba (*Myrciaria caulifora*) [6], bacupari (*Garcinia brasilienses*) [30], recovery of food waste by anaerobic digestion [31], and anaerobic digestion technology [28]. This study aims to identify trends in research related to Cambuci and research gaps and opportunities through bibliometric analysis. The analysis focused on review articles and research published between 2003 and 2022.

Methodology

The bibliometric analysis was carried out using as a reference the methodology described by Sganzerla & da Silva [23] and Gabriel da Rosa et al. [6]. The search was performed through the Scopus© database with words and terms: cambuci OR campomanesia AND phaea OR "O. Berg Landrum" OR "(O. Berg) Landrum" OR cambucizeiro. Furthermore, the filter by document type was used, selecting "article" and "review". The files counted were exclusively articles and reviews published between 2003 and 2022. 175 documents were found 153 articles, and 22 reviews. The data were exported and added to the VosViewer © software, thus generating graphs and tables that facilitate the evaluation of the main characteristics of the set of documents found. The graphs show the clusters of the main keywords, the main authors, the most cited articles, and their respective countries.

The dataset obtained was also inserted into the bibliometric software Bibliometrix (R language), where graphics were obtained to present the most employed authors' keywords, a thematic map of the most frequent authors' keywords in the field of cambuci research, and the top authors. The bibliometric discussion was based on the 175 documents found and evaluated the evolution of publications related to cambuci between the years 2003 and 2022, which are the main research fields, the most cited documents, the principal authors, ten main affiliations, and the main countries. Finally, the main trends related to the topic were evaluated. Figure 1 presents the stages of bibliometric analysis.

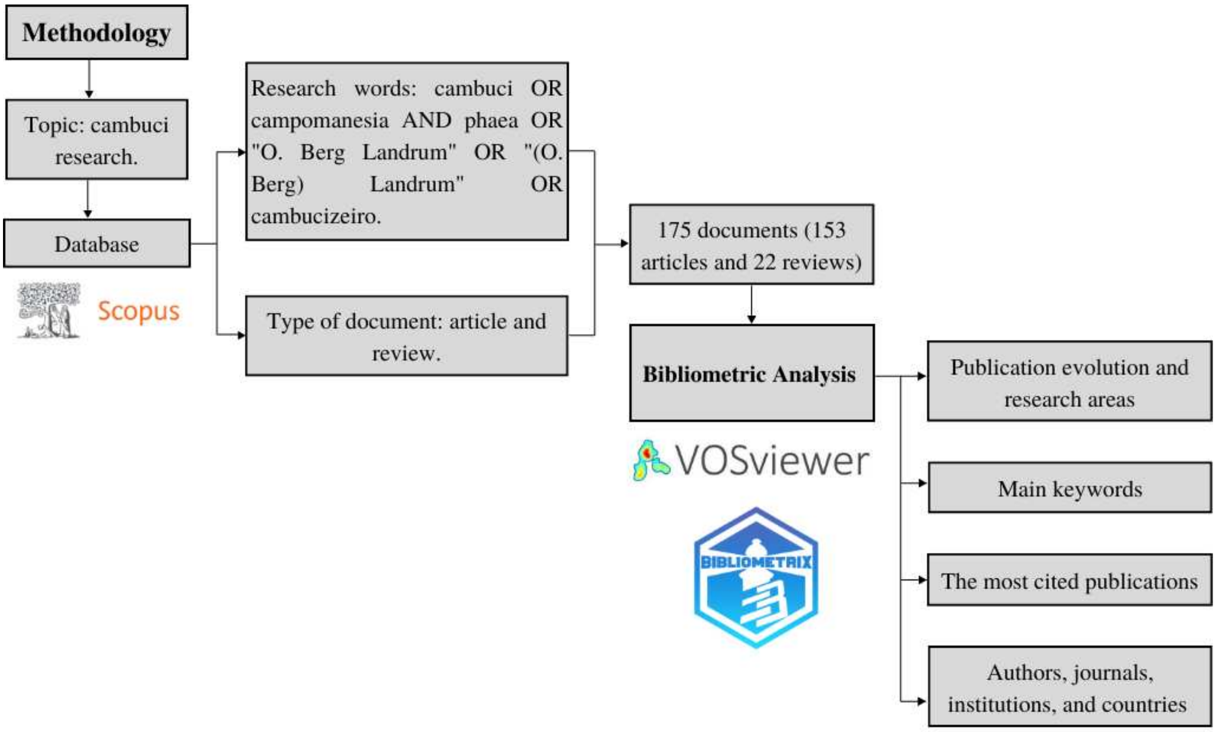


Fig. 1 Methodological steps for the bibliometric analysis

Research trends on Cambuci

Publication evolution and research areas

The progress of research of cambuci studies can be seen in Fig. 2. A total of 175 documents were found, of which 153 were articles and 22 reviews published between 2003 and 2022 in the Scopus database. In 2003, the first publication was founded. In 2004 and 2007, publications ceased, returning to occur in 2005, 2006, and 2008, with 1, 4, and 5 documents being published, respectively. The number of publications remained below 7 for 13 years until 2016, when a growing interest in cambuci began, demonstrated by the growth of publications between 2019 and 2021, the latter being the year that presented the highest number of publications, reaching the mark of 45 documents. The increase in publications is related to the emergence of new research areas. Until 2017, research areas alternated, reaching a maximum of 4 per year, which changed in 2019 with the publication of documents by new research areas. The emergence of these new research areas demonstrates the potential growth of cambuci in different studies and its multidisciplinary character.

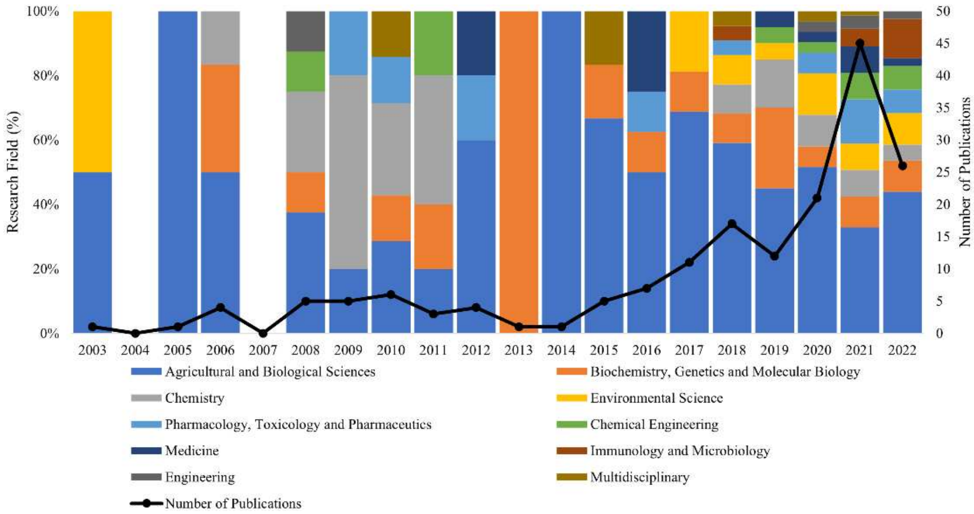


Fig. 2 Progress of the number of publications over the Years (2003–2022) in the field of cambuci research

Table 1 describes the ten main areas of publications in the research period. The main publication area is Agriculture and Biological Sciences, and a total of 115 articles were published, corresponding to 67.65% of all publications found in the Scopus database. The difference between the number of documents opened by the first and second areas is 50%, thus demonstrating the great interest in cambuci. The second main area is Biochemistry, Genetics, and Molecular Biology, with 30 documents, followed by Chemistry, Environmental Science and Pharmacology, and Toxicology and Pharmaceuticals, with 21 documents. The results showed that studies referring to cambuci were scarce until 2017. Over the years, this fruit has become the subject of several publications, especially in Agricultural and Biological Sciences, which was predominant among cambuci publications.

Research areas and keywords for potential impact

The study of the co-occurrence of the main keywords was performed to assess the hot spots and frontiers around cambuci studies, aiming to identify which research areas are of potential impact [32]. Figure 3a presents the grouping of the 48 main keywords addressed by the authors corresponding to the information of each study. It is possible to see clusters based on closely interrelated keywords, grouping similar topics identified by colors. The items are connected by lines of different widths, which are proportional to the link strength among them. Figure 3b presents the evolution of publications between 2012 and 2022 based on analyzing the authors' keywords.

Table 1. Ranking 10 top publication areas, affiliations, countries, and journals.

Ranking	Research areas	Number	%[a]
1st	Agricultural and Biological Sciences	115	67.65
2nd	Biochemistry, Genetics and Molecular Biology	30	17.65
3rd	Chemistry	25	14.71
4th	Environmental Science	21	12.35
5th	Pharmacology, Toxicology and Pharmaceutics	21	12.35
6th	Chemical Engineering	12	7.06
7th	Medicine	12	7.06
8th	Immunology and Microbiology	10	5.88
9th	Engineering	6	3.53
10th	Materials Science	5	2.94
Ranking	Affiliations (Institution)	Number	%
1st	Universidade de São Paulo	40	23.53
2nd	Universidade Federal de Minas Gerais	13	7.65
3rd	Universitat Salzburg	11	6.47
4th	Universidade Federal de Lavras	11	6.47
5th	Universidade Federal de Mato Grosso do Sul	9	5.29
6th	Universidade Estadual de Mato Grosso do Sul	9	5.29
7th	Universidade Federal de Santa Catarina	9	5.29
8th	Universidade Estadual de Campinas	6	3.53
9th	Universidade Federal do Rio de Janeiro	5	2.94
10th	Empresa Brasileira de Pesquisa Agropecuária - Embrapa	5	2.94
Ranking	Countries	Number	%
1st	Brazil	118	69.41
2nd	China	13	7.65
3rd	Austria	11	6.47
4th	United States	10	5.88
5th	India	7	4.12
6th	Italy	6	3.53
7th	Canada	4	2.35
8th	Spain	4	2.35

Ranking	Countries	Number	%
9th	Sweden	4	2.35
10th	Mozambique	3	1.76
Ranking	Journals	Number	%
1st	Food Research International	9	5.29
2nd	Journal of Essential Oil Research	9	5.29
3rd	Arthropod-plant Interactions	6	3.53
4th	Foods	4	2.35
5th	Journal of Ethnopharmacology	4	2.35
6th	Acta Botanica Brasilica	3	1.76
7th	Anais da Academia Brasileira de Ciências	3	1.76
8th	Ciência e Tecnologia de Alimentos	3	1.76
9th	Journal of Food Process Engineering	3	1.76
10th	Molecules	3	1.76

[a] Percentage of 175 documents (automatically calculated in Scopus). Search conducted on January 1st, 2023; * In Portuguese

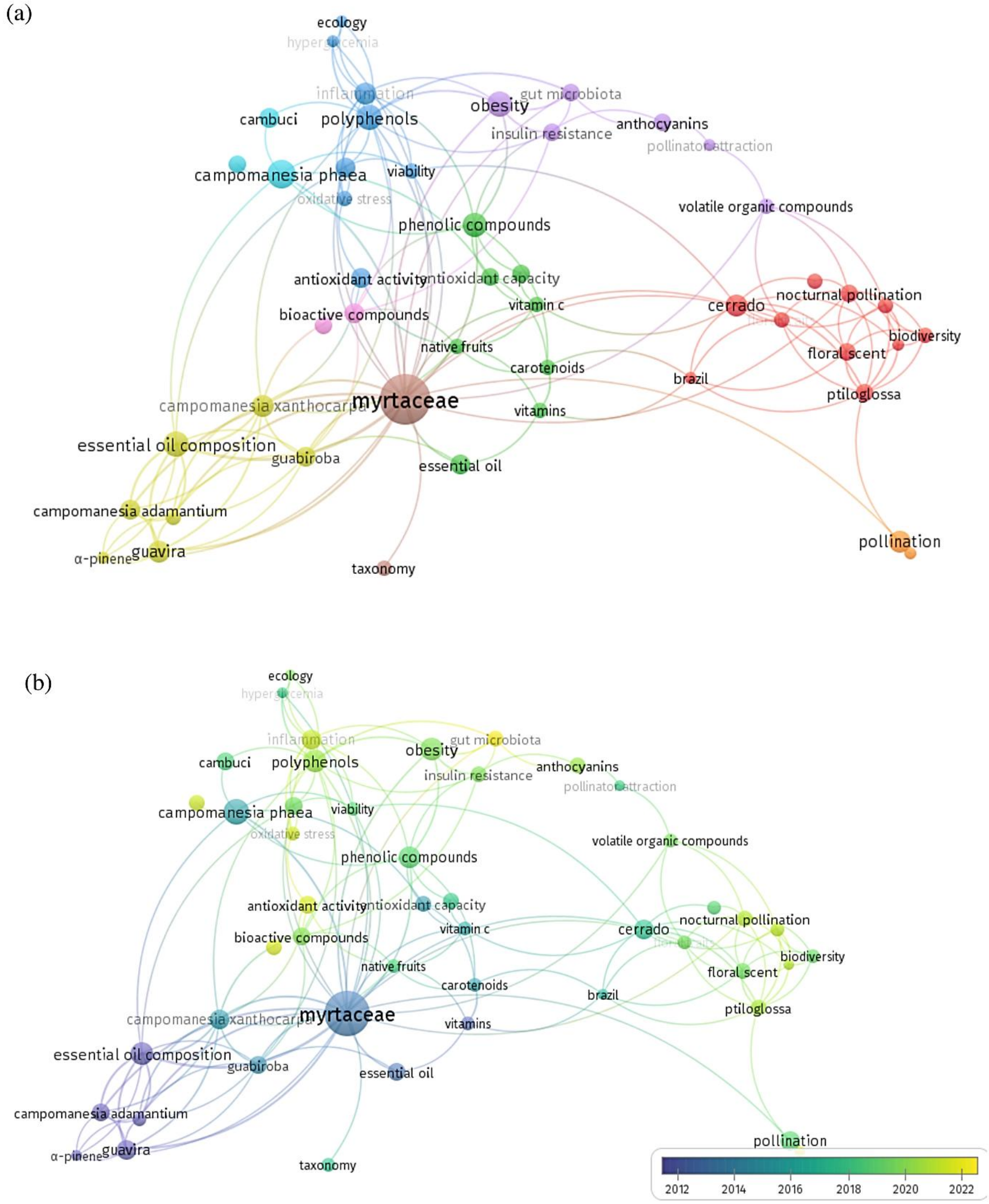


Fig. 3 The most used authors' keywords. (a) Term map based on different clusters; (b) Map of the evolution of the authors' keywords by year.

From the search implemented on Scopus, covering all publications related to cambuci, 175 studies were found and 641 author keywords. Ranking keywords by the total link strength, the top position is occupied by "*Myrtaceae*", followed by "*essential oil composition*", "*Campomanesia xanthocarpa*", "*guavira*", and "*Campomanesia adamantium*". This set of words is interesting, since Myrtaceae is a family comprehending 121 genera with many species, with considerable potential for obtaining carotenoids, volatile compounds, and phenolic compounds [7]. In addition to the family name, the keywords "*Campomanesia xanthocarpa*" and "*Campomanesia adamantium*" represent other species that are closely related to cambuci, since they belong to the same genera (*Campomanesia*). In the present study, 9 (nine) clusters were formed, and their items are presented in Table 2. In addition, Table 3 ranks the top 20 keywords according to the number of occurrences.

Table 2. Identification of the clusters based on the analysis of keywords.

Cluster	Number of items	Keywords on VOSviewer Network
1	10	Biodiversity, Brazil, cerrado, conservation, crepuscular bees, floral scent, floral traits, megalopta, nocturnal pollination, and ptiloglossa
2	8	Antioxidant capacity, carotenoids, essential oil, flavonoids, native fruits, phenolic compounds, vitamin C, and vitamins
3	8	Antioxidant, antioxidant activity, ecology, hyperglycemia, inflammation, oxidative stress, polyphenols, and viability
4	7	Campomanesia adamantium, campomanesia xanthocarpa, essential oil composition, guabiroba, guavira, ledol, and α-pinene
5	6	Anthocyanins, gut microbiota, insulin resistance, obesity, pollinator attraction, and volatile organic compounds
6	3	Cambuci, campomanesia phaea, and carbon dots.
7	4	Lepidoptera, and pollination
8	3	Myrtaceae, and taxonomy
9	2	Bioactive compounds and osmotic dehydration

Table 3. The top 20 keywords in the field of cambuci research (rank based on the occurrences).

Ranking	Keyword	Occurrences	Total link strength
1	Myrtaceae	31	48
2	Campomanesia phaea	10	9
3	essential oil composition	8	26
4	polyphenols	8	17
5	obesity	8	10
6	phenolic compounds	7	12
7	campomanesia xanthocarpa	6	19
8	cerrado	6	11
9	guavira	6	18
10	inflammation	6	9
11	pollination	6	3
12	antioxidant	5	6
13	antioxidant activity	5	4
14	bioactive compounds	5	9
15	cambuci	5	2
16	campomanesia adamantium	5	18
17	essential oil	5	4
18	guabiroba	5	11
19	anthocyanins	4	3
20	antioxidant capacity	4	6

It is possible to notice the family's name Myrtaceae is placed between the clusters presented in blue and yellow, which are related to three different species of fruits, respectively, *Campomanesia phaea*, *C. xanthocarpa*, and *C. adamantium*. Each of these clusters presents research topics around bioactive compounds of the correspondent specific fruit and are linked to words representing its potential applications. The clusters in violet and green do not present a species name. However, they present keywords related to diseases against which cambuci may be useful, such as "obesity" and "insulin resistance", and present attractive traits of the fruit, such as "antioxidant capacity" and "vitamin C". The red cluster presents words that comprehend some agricultural and biological aspects linked to the fruits given by the words: "biodiversity", "floral traits", and "nocturnal pollination", for example. On the other hand, the antioxidant activity, osmotic dehydration, pollination, and oxidative stress of cambuci are some topics that have begun to study as of 2020.

Figure 4 presents a thematic map, and the keywords are placed according to the development degree of the research field and its relevance. The thematic map contains four quadrants, namely motor themes (up-right), niche themes (up-left), emerging or declining themes (down-left), and basic themes (down-right). The thematic map presents the formation of 7 (seven) clusters distributed in the diagram, showing the development degree (or density) and the relevance degree (or centrality) of the research fields. According to Fig. 4, the keywords "*myritaceae*", "*essential oil composition*", and "*Campomanesia xanthocarpa*" are basic themes with a high relevance degree, which means that the research field is important but not well-developed yet, requiring more research and analysis, consisting of an interesting area for the development of new research. Also, the keywords "bioactive compounds", "antioxidant capacity", "flavonoids", "antioxidant activity", "phenolic compounds", and "osmotic dehydration" are motor themes, indicating research fields that are well-developed and considered great importance. It is relevant to highlight that among the terms pointed as motor themes, the cluster containing the later three keywords (i.e., "antioxidant activity", "bioactive compounds", and "osmotic dehydration") presents the highest level of relevance. This fact, together with the few publications around cambuci, may indicate the possibility of developing even more research themes already indicated as "motor themes".

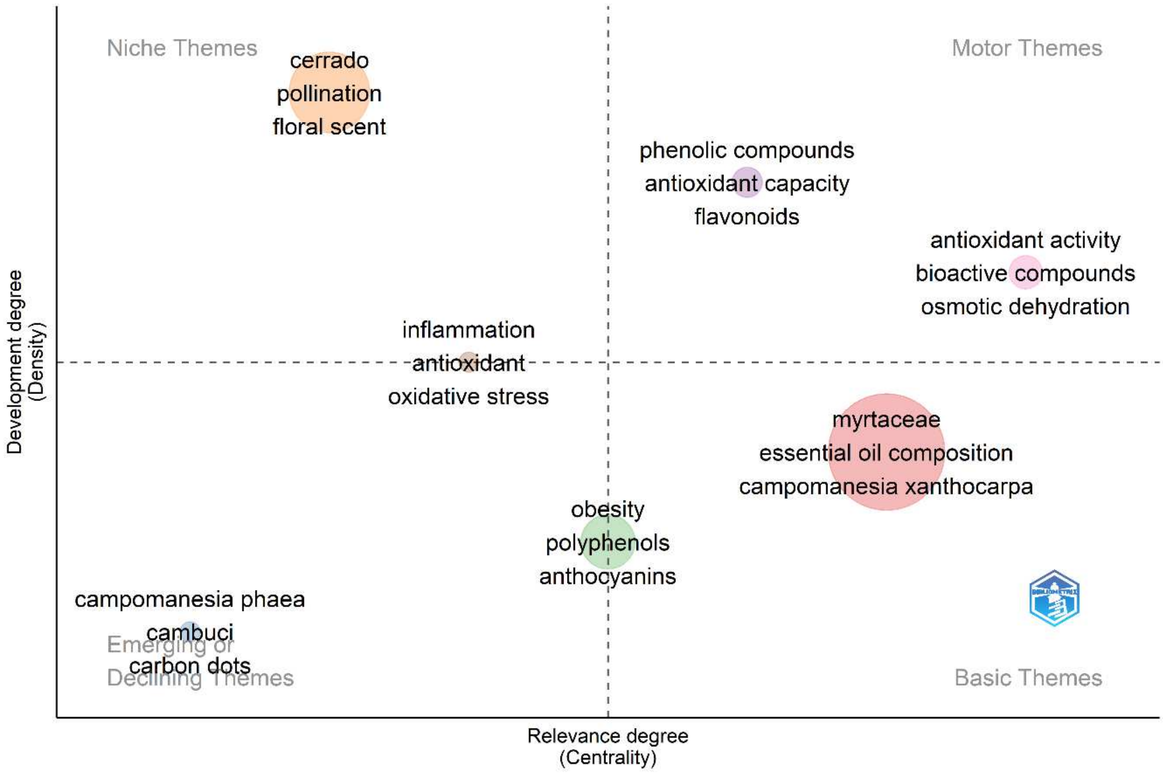


Fig. 4 Thematic map of the most frequent authors' keywords in the field of cambuci research.

The most relevant publications

Table 4 shows ten most cited articles in the research area related to cambuci; it creates a perspective of the determined branch of study. The article about bioactive compounds and antioxidant capacity of fruits written by Genovese et al. [29], and published in Food Science and Technology International was the most cited (129 citations). The study analyzed the antioxidant capacity and bioactive compounds of some exotic Brazilian fruits, such as jaracatia, cambuci frozen araçá, camu-camu, and cagaita pulps. Genovese et al. [29] determined that fruits and pulps were significant sources of bioactive compounds with antioxidant capacity, which, although cambuci did not stand out, presented considerable amounts of bioactive compounds.

Table 4. Top 10 most cited articles in the field of cambuci research.

Ranking	Journal	Publication Year	Total Citations	Average Citation per Year	Reference
1st	Food Science and Technology International	2008	129	8.06	[33]
2nd	Brazilian Journal of Botany	2006	122	6.78	[34]
3rd	Chemistry & Biodiversity	2011	118	9.08	[35]
4th	Food Chemistry	2018	63	10.50	[36]
5th	Food Science and Technology	2006	56	3.11	[37]
6th	Food Research International	2019	45	9.00	[38]
7th	Austral Entomology	2017	42	6.00	[39]
8th	Food Research International	2018	36	6.00	[40]
9th	Brazilian Journal of Pharmaceutical Sciences	2009	34	2.27	[41]
10th	Plant Biology	2017	33	4.71	[42]

Gressler et al. [30] published the second most cited research about Brazilian Myrtaceae in the Brazilian Journal of Botany (122 citations). The study aimed to synthesize all information regarding the reproductive ecology of Myrtaceae in Brazil, grouping data related to pollinators and seed dispersers of different species. The results showed that dispersal and pollination in Myrtaceae from Brazil were mainly carried out by bees and frugivorous vertebrates, respectively. The work about the chemical and biological properties of Mirthaceas developed by Stefanello et al. [31] and published in the Journal Chemistry & Biodiversity was the third most mentioned work (118 citations). It concluded that the neotropical Myrtaceae species has a lot of sources of bioactive compounds, because its products have relevant antioxidant and anti-inflammatory activities. The project published by Seraglio et al. [32] evaluated jambolan, guabiju, and jabuticaba about capacity, phenolic compounds, sugars, total monomeric anthocyanin, and minerals present during ripening, pointing out that, as well as research described by Stefanello et al. [31] about maturation and differing, since the previous study used neotropical Myrtaceae.

Vallilo et al. [33] developed the article published in the journal Food Science and Technology to analyze the nutritional constitution of the fruits of C. adamantium, which is part of the Myrtaceae family. The study showed that these fruits had relevant characteristics for application in the beverage industry (such as favoring) and food (in nature), such as minerals, ascorbic acid, and dietary fiber. The search for evaluation of antioxidant and antimicrobial activities of the essential oil of cambuci developed by [41] characterized the components of essential oils from leaves of Campomanesia adamantium (Cambess.). According to the results, during the reproductive phase, the composition was mainly monoterpenes, while in the vegetative phase, it was composed of sesquiterpenes. The results also showed that the reproductive phase of the essential oil had high antimicrobial activity against Candida albicans and Staphylococcus aureus, for instance.

Moura et al. [35] wrote a project about jaboticaba published in the journal Food Research International to evaluate two different Sabara jabuticaba extracts regarding their antiobesogenic characteristics and whether the tannin content present in the extract can influence the body weight gain and mitigation of hyperglycemia, hyperinsulinemia, total cholesterol, and hepatic triacylglycerol levels. Sabara jabuticaba is a species that belongs to the Myrtaceae family, as well as *Campomanesia phaea Berg*. This study proved that the tannin extracts considerably influenced these aspects and confirmed their anti-obesogenic characteristic. On the other hand, the article about the nocturnal pollination system was to portray a pioneering pollination system mediated between a plant and its nocturnal bee pollinators. And they conclude that the combination of the most substantial compounds is responsible for attracting nocturnal pollinating bees. In addition, olfactory suggestions are possibly linked to the attraction of daytime bees, since the main components of cambuci attract honeybees.

In a nutshell, the most relevant publications about cambuci (Campomanesia phaea Berg.) and other species belonging to the Myrtaceae family show that they could be a substantial source of bioactive compounds with great relevance to the different industries, and they have a high research and development potential.

Review of the number of articles, citations, and areas

Figure 5 presents the authors' production over time, indicating the number of articles each author has published per year and the citations each author received (color of circles) from 2008 to 2022. The most productive authors in the whole period are Dötterl, S. (11 publications, mainly about agricultural and biological sciences, and environmental science), Genovese, M. I. (11, agricultural and biological sciences, and biochemistry, genetics, and molecular biology), and Jacomino, A. P. (10, agricultural and biological sciences, and biochemistry, genetics, and molecular biology).

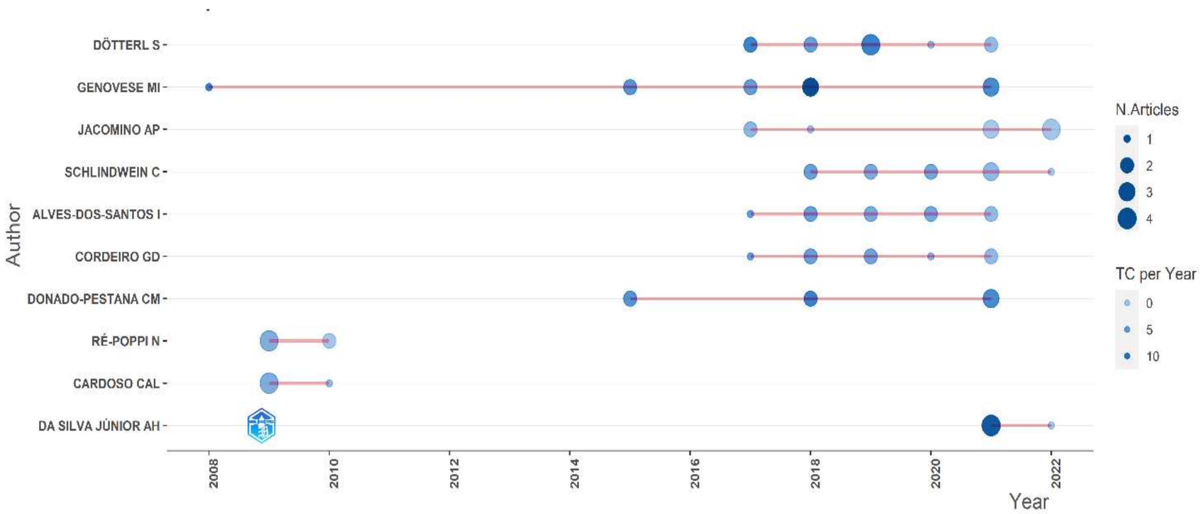


Fig. 5 Bibliometric analysis of the top authors in the field of cambuci research.

Dötterl, S. is afliated with the Paris-London University of Salzburg, Austria, and has been developing research around cambuci fruit in partnership with several Brazilian institutions, as well as some institutions from Italy and Germany. Genovese, M. I. is a Brazilian researcher, developing most of her studies only with national contribution and one study with the University of Laval from Canada. Jacomino, A. P. is affiliated with the University of São Paulo, Brazil, presenting most studies around cambuci with other Brazilian partners and one study with Michigan State University from the United States of America.

Table 1 indicates the publication areas on the number of publications. Brazil is the absolute leader in the number of publications, responsible for 118 documents related to biochemistry, chemistry, agriculture, biological sciences, genetics, and molecular biology. In the second position is China (13 documents), with genetics, biochemistry, agricultural, biological sciences, environmental sciences, and molecular biology research. The third place corresponds to Austria (11).

Since most publications are linked to Brazil, the most important organizations researching cambuci fruit are also Brazilian. The affiliation ranking is led by the University of São Paulo and features eight other Brazilian institutions, among which there are seven universities and one research organization (Embrapa). The second place in the affiliations' ranking is also a Brazilian institution. In third place is the University of Salzburg, Austria, with 11 papers, 10 of which are by Dötterl, S. Regarding the ranking of journals, the impact factor ranges from 2.409 (Arthropod-Plant Interactions) to 7.425 (Food Research International) with an average value of 4.211 (considering only the eight journals that present an impact factor score). These journals deal with food chemistry, microbiology, and safety, aspects related to essential oils, interactions of insects and other arthropods with plants, and the use of plants for pharmacological effects. Figure 6 shows the main keywords of the top 5 articles published regarding cambuci.

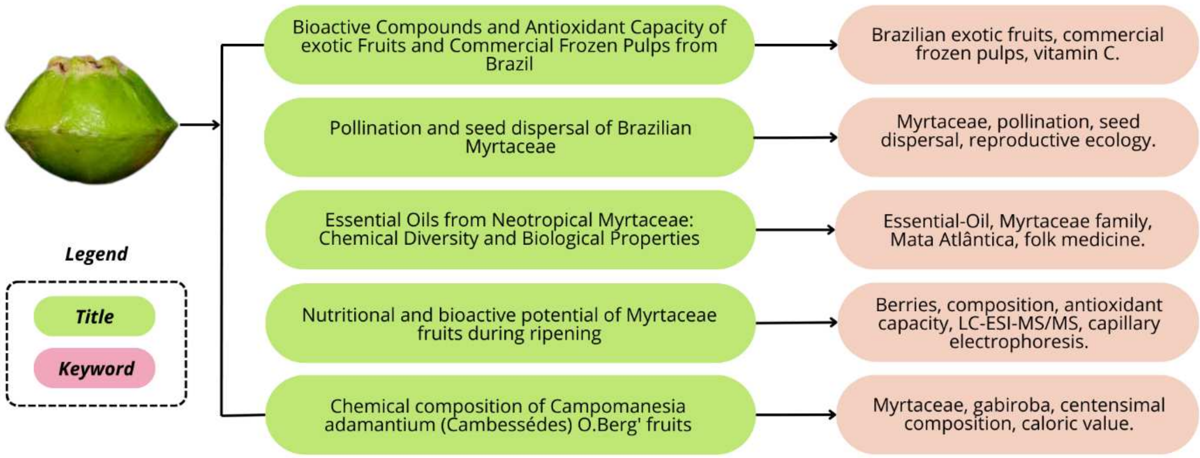


Fig. 6 Keywords of the most relevant articles on cambuci.

Review of cambuci fruit and waste

Source

Cambuci fruit (*Campomanesia phaea Berg.)* is also known as cambuchi, cambucy, cambuhi, cambucizeiro, camote, camuci, and camucim [43]. It is native to Brazil's Atlantic Forest biome [44, 45]. Mainly, this fruit is found in the State of São Paulo, Brazil. However, it has also been observed in Minas Gerais and Rio de Janeiro States [46]. Due to its short useful life, high moisture content (88% wet base), and consumption in places close to the harvest [10, 47]. Cambuci trees can be found in backyards, nature reserves, and small commercial orchards responsible for meeting the needs of the emerging market. On the other hand, this species can grow in acidic soils with high organic matter and aluminum content and low potassium and magnesium [43]. The trees have a height between 3 and 5 m, and the trunks have a diameter of 20 to 30 cm [10].

Cambuci is a green berry and ovoid-rhomboidal fruit with an acid-astringent taste [8], which measures approximately 6 cm in diameter in the central part and a thickness that varies between 3 and 4.5 cm [10]. Cambuci fruits have a high pulp yield and contain approximately 9 to 13 seeds, of which 30% are fertile. The seeds are white, fat, and orbicular [43, 48]. The flowering of the cambuci tree occurs in 4 and 5 months [49]. It occurs mainly between October and January, with the maximum intensity between November and December. Nonetheless, throughout the year, there is reduced extemporaneous flowering [43]. Ripe cambuci fruits are characterized by maintaining the green color of the peel, but they become more opaque and less intense. In addition, there is a reduction in the firmness of the fruits, and a natural detachment of the trees is evident. Traditionally, the harvest of these fruits consists of the manual collection of the fruits on the ground [43], which leads to loss of quality, contamination, and fruit dehydration [50].

Physicochemical characterization

Table 5 shows the composition of the cambuci pulp according to various authors. In general, cambuci has a high moisture content (greater than 80%) and insoluble fiber, which is related to the promotion of gastrointestinal health, antiobesity, as well as the reduction of suffering from diabetes and cardiovascular disease [51]. However, it is essential to note that its composition may also vary according to the area of origin. Sanches [48] analyzed the fruits from four regions: Rio Grande da Serra, Mogi das Cruzes, Paraibuna, and Paranapiacaba, obtaining a high concentration of lipids, ashes, protein, carbohydrates, and fiber. Moreover, the value of soluble solids can vary between 9.37 and 10.61, according to the investigations carried out by Tokairin [15] and Sanches [48]. On the other hand, the pH value varies from 2.4 to 2.91, as reported by Tokairin et al. [36] and Sanches [48].

Table 5. Physicochemical characterization of the cambuci pulp.

Composition	[52]	[9]	[43]	[19]
Moisture (%)	84.41	88.8	84.68	86.80
Ash (%)	2.30	2.60	2.64	2.20
Lipids (%)	1.44	4.60	3.16	2.89
Proteins (%)	3.00	3.00	8.86	1.17
Insoluble fiber (%)	-	-	33.12	30.68
Soluble fiber (%)	-	-	5.50	6.51
Carbohydrates (%)	11.62	8.44	-	-

Cambuci is a fruit with high phenolic compound levels and high antioxidant capacity, which can reduce the risk of developing metabolic diseases or obesity [8, 14]. For instance, Donado-Pestana et al. [14] reported values of 4.0 mg GAE/mL for total polyphenols in cambuci pulp [12] obtained 4.4 mg GAE/mL, [53] reported 3.4 mg GAE/mL and Genovese et al. [50] obtained 2.4 mg GAE/mL.

Moreover, Donado-Pestana et al. [12] established that the major polyphenols present in cambuci extracts are ellagic acid (4333 µg/100 mL), digalloyl-HHDP-glucose (Tellimagrandin II), p–coumaric acid hexoside, quercetin-O-(Ogalloyl)-pentoside (1862 µg/100 mL), quercetin rhamnose, and quercetin pentoside. Figure 7 shows the chemical structure of these compounds. Besides, Taver et al. [47] established that cambuci antioxidant capacity was between 9.23 and 12.2 µmol g^{-1}. Donado-Pestana et al. [14] established the antioxidant capacity of cambuci by different methods. They found that the antioxidant capacity by the oxygen radical absorbance capacity assay and by the ferric reducing antioxidant power assay, the antioxidant capacity was 13.87 µmol Trolox $mL^{-1.}$

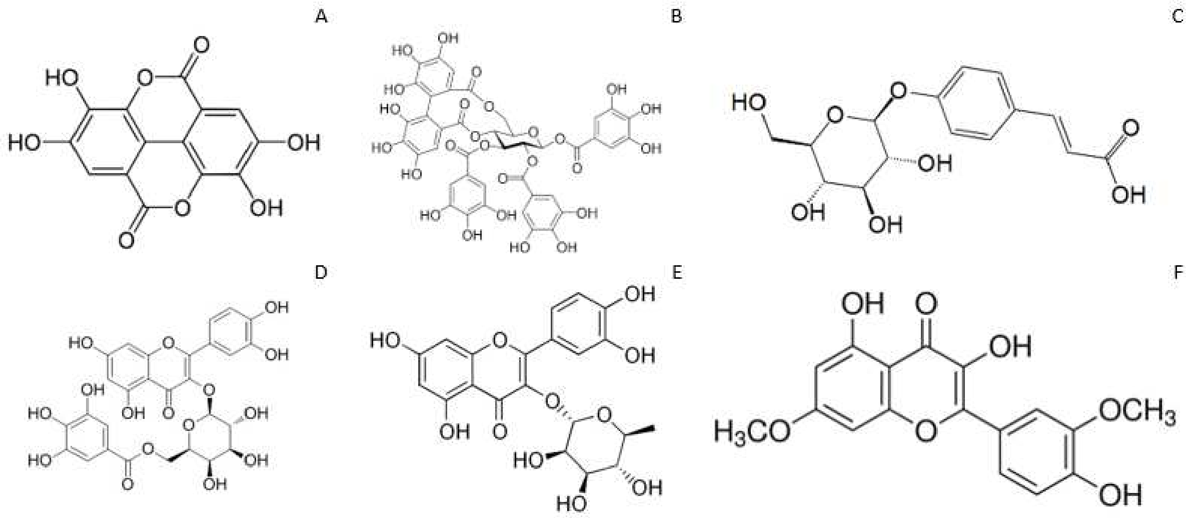


Fig. 7 Structures of the main polyphenols obtained from cambuci fruit. (A) ellagic acid, (B) Digalloyl-HHDP-glucose (Tellimagrandin II), (C) *p* - coumaric acid hexoside, (D) quercetin-O-(O-galloyl)-pentoside, (E) quercetin rhamnose, and (F) quercetin pentoside.

Ellagic acid is a bioactive phenolic compound in tannins [16, 54]. This compound is found naturally in fruits and vegetables (Table 6). It has an excellent benefit for human health due to its antibacterial, antifungal, antiviral, anti-inflammatory, antidiabetic, gastroprotective, and antidepressant activity [16, 55, 56]. Among the functions of ellagic acid is the inhibition of liver damage induced by alcohol, since it increases the levels of antioxidants, eliminates free radicals, and stabilizes cell membranes [57]. Ellagic acid is insoluble in water and slightly soluble in alcohol. It is also a thermostable compound, with a melting point of 350 °C and a molecular weight of 302.197 g/mol, which can be soluble in basic solvents [58].

Other compounds in cambuci are proanthocyanidins, phenolic compounds, responsible for the astringency and acid taste [52, 71]. The proanthocyanidins present in cambuci are tannins resulting from the polymerization of polyphenolic compounds called flavonoids. This compound can be found in various specimens of the plant kingdom, such as flowers, fruits, and bark, among others [72]. Its presence promotes bitterness, acidity, aroma, and astringency in the fruits, the latter characteristic of the cambuci [14, 52]. Proanthocyanidins have numerous bioactive activities, such as antioxidant capacity, which is more significant than the antioxidant activity presented by vitamin E and greater than vitamin C [73]. They also exhibit anti-inflammatory and antimicrobial activity [74]. When ingested, these tannins promote several health benefits due to their easy absorption, presenting a bioavailability of more than 90% [75].

Among the benefits, cardioprotective, neuroprotective, immunomodulatory, antidiabetic, and anticancer effects can be mentioned [76]. In addition, they have metabolic effects, since they decrease triglyceride levels, the number of foam cells, atherosclerosis, and lipogenesis [77].

Table 6. Ellagic acid concentration in pulp extracts.

Fruit	Concentration	Extraction method	Reference
Blueberry (*Vaccinium corymbosum L.*)	3 – 8.4 mg/g FW	Enzymatic hydrolysis combined with ultrasonic-assisted organic solvent extraction	[59]
	0.97 – 2.1 mg/g FW	Enzymatic hydrolysis extraction	[59]
Kakadu plum (*Terminalia ferdinandiana*)	1214 – 1726 mg/100g DW	100% methanol	[60]
Camu-camu (*myrciaria dubia*)	490 mg/100g DW	-	[61]
Cambuci (*Campomanesia phaea Berg.*)	240 mg/100g DW	-	[62]
	480 mg/100g DW	70% methanol for 1 minute, and centrifugated for 10 minutes (20°C).	[52]
Feijoa (*Acca sellowiana (O. Berg) Burret*)	9.2 mg/g DW	70% ethanol, material to solvent ratio of 1:30, at 50 °C for 30 min.	[63]
Strawberry (*Fragaria × ananassa)*	0.95 – 1.77 mg/g FW	80% ethanol, extract centrifuged at 6000 rpm and 4 °C for 10 min.	[64]
Raspberry (*Rubus chingii Hu*)	9.46 – 30.70 mg/100g FW	55 % ethanol with ultrasonic treatment for 30 min.	[65]
Jabuticaba (*Myrciaria jaboticaba (Vell.) O.Berg*)	3.88 mg/100g DW	35 °C for 50 h, extraction at 45 °C with water and propanone (52:48 v/v) using a 1:20 w/v ratio.	[66]
Guava (*Psidium guajava* L.)	5.72 – 30.60 mg/100 g DW	100% methanol	[67]
Buriti (*Mauritia flexuosa* L.)	0.13 mg/100 g DW	Ethanol and a supramolecular solvent system (SUPRAS) formed by octanoic acid aggregates	[68]
Psidium cattleianum	2213 – 3818 µg/g	100% ethanol. Solvent ratio of 1:10 (w/v) and magnetic stirring, for 4 h, at 25°C.	[69]
Araçá (*Psidium cattleianum* Sabine)	29.1 µg/mL	Samples (250 mg) were mixed with methanol (1:40, w/v) and stirred for 5 min.	[70]

DW: dry weight; FW: fresh weight

When proanthocyanidins are in an acidic medium in the presence of heat, it promotes the formation of anthocyanidins, a property that can be further explored [75]. Several residues are used as a source of proanthocyanidins, mainly grape seeds [78, 79]. Given its numerous benefits and properties, this compound shows great promise. The concentration of proanthocyanidins present in Cambuci varies according to the method of analysis used and the harvest period. Studies show that the proanthocyanidin content in cambuci ranges from 11.5 to 52.2 mg CAT/100g [8]. Furthermore, fruits of the cambuci trees harvested in different localities also presented variations in the concentration of this compound. However, the concentration is significant [14]. Table 7 shows proanthocyanidin concentration in some fruits. The higher concentration of proanthocyanidins in cambuci is like that presented by the green grape. It is higher than most other fruits, demonstrating that Cambuci is a promising raw material for obtaining proanthocyanidins.

Table 7. Proanthocyanidin concentration in some fruits.

Fruit	Concentration	Units	Reference
Cambuci	11,5 – 52,2	Mg CAT /100g FW	[8]
Kiwi fruit	13,3	Mg / 100 g FW	[80]
Avocado	17,8		
Strawberry Honeoye	49,2		
Strawberry Jonsok	34,2		
Apple Lobo	43,3		
Cherry	26,8		
Peach	37,7		
Nectarine	21,6		
Pear	20,7		
Cloudberry	31,9		
Grape red	32,6		
Grape green	54,0		

CAT: catechin equivalent; FW: fresh weight

Industrial processing of cambuci and perspectives

The annual production average of cambuci fruits is approximately 300 tons per year in Brazil [9]. Figure 8 shows the agro-industrial processing of cambuci, which allows visualizing the research opportunities on the by-products of the fruit. The cambuci fruit is mainly used to prepare juices, jellies, and ice creams [3, 9]. However, it can also be consumed in nature [8]. The cambuci is receiving more attention from consumers due to its attributes for industrialization, such as high fiber and soluble solids, pulp yield, titratable acidity, and especially the high amount of pectin. The high concentration of pectin has increased gelling capacity, a significant characteristic of certain proteins in industrialized products, such as candies, jellies, and gelatine [46, 81]. In addition, cambuci has phenolic and bioactive compounds in its structure that have anti-inflammatory, antioxidant, and antimicrobial characteristics [53, 82–85].

In Brazil, the primary consumers of this fruit are haute cuisine restaurants, juices, and a wide variety of recipes [43]. The versatility of cambuci fruit allows it to be prepared with sweet or savory products that can be served in meals from breakfast to dinner [50]. In addition, its peel is sold at fairs, emphasizing its vitamin C content [50] and its potential for treating diabetes [3]. *Campomanesia phaea* (cambuci) extracts showed remarkable results when evaluated concerning the ability to eliminate reactive oxygen, aging, inflammation, and neurodegenerative diseases [82]. Research has shown that phenolic compounds in cambuci have antidiabetic properties, since it contains ellagitannin derivatives in their composition, which is responsible for significantly lowering postprandial glucose levels [3, 86]. Furthermore, these polyphenols are relevant in attenuating obesity-related metabolic disorders, such as lowering insulinemia and preventing dyslipidemia by raising HDL cholesterol and lowering LDL cholesterol [12, 14].

Silva Júnior et al. [83] proved that the use of cambuci juice as a precursor of carbon dots (a modern class of materials that has several advantages, such as low cytotoxicity) for the detection of zinc ions (an essential micronutrient in biological pathways) is considered feasible, and that is one of the reasons for using cambuci juice on research, because conventional methods have several disadvantages, such as high-cost reagents. On the other hand, cambuci is extremely rich in water, which makes it very perishable, thus limiting its production and commercialization. Thus, developed studies have proven that drying processes aligned with pre-treatments (using sorbitol and ethanol combined with freezing) is an effective alternative to increase the stability of cambuci due to the higher amount of fiber, pulp yield, total titratable acidity, and pectin [47, 87, 88]. Spricigo et al. [86] developed the work that showed that there are different types of amino acids in cambuci, such as glutamic acid, that collaborate with neural development, promoting the improvement of cognitive functionalities, such as memory.

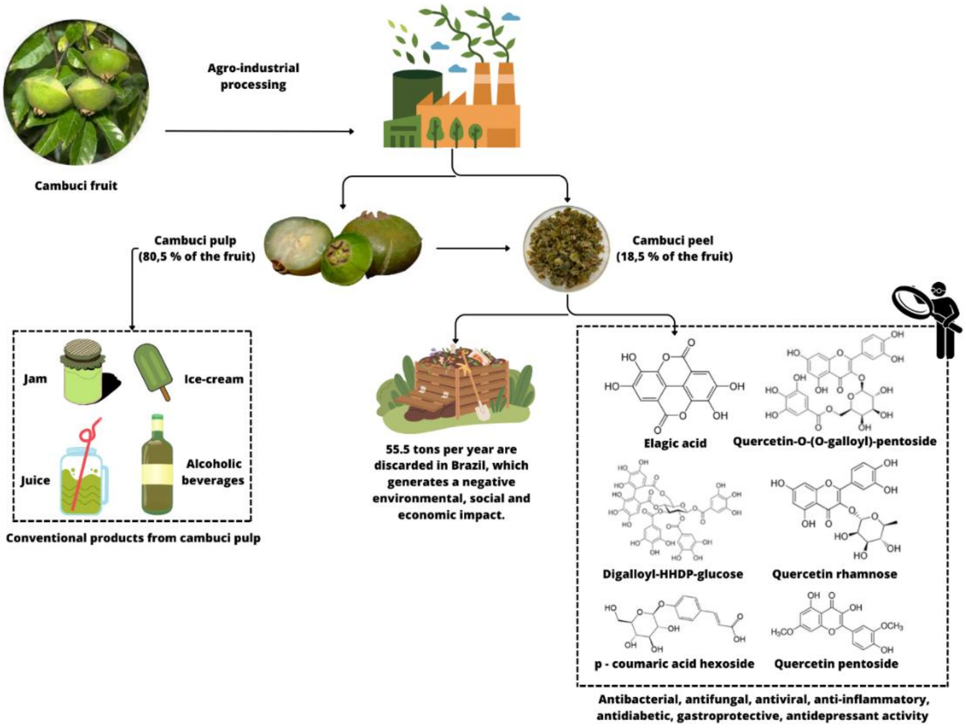


Fig. 8 Agro-industrial processing of cambuci fruit.

Conclusions

The bibliometric analysis gave us the research trends and perspectives on a native Brazilian fruit (cambuci) between 2003 and 2022. The analysis indicated that 153 articles and 22 reviews were published, and the predominant research area was agriculture and biological sciences. In addition, it was possible to establish that issues, such as phenolic compounds, antioxidant capacity, flavonoids, antioxidant activity, bioactive compounds, and osmotic dehydration, are driving issues considered of significant importance, and it is suggested that future research be related to these issues to develop knowledge of this native fruit. Besides, it was possible to determine that Brazil is the country that has developed the most research on cambuci (118 documents) with topics related to genetics, agriculture, chemistry, biological sciences, biochemistry, and molecular biology. Cambuci is a fruit with great potential for the food and pharmacy industry, since it has compounds, such as ellagic acid and proanthocyanidins, which have anti-inflammatory, antioxidant, and antimicrobial effect that significantly favors healthy subjects. In conclusion, this study showed the progress of research, trends, and updates on the valuation of cambuci, and suggests continuing to develop research on this native Brazilian fruit that has a high industrial potential.

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Author contributions

JAJM: methodology, data curation, formal analysis, and writing–original draft preparation. LCA: formal analysis, and writing—reviewing and editing. VCF: formal analysis, and writing— reviewing and editing. LFM: formal analysis, and writing—reviewing and editing. MAR: supervision. TFC: conceptualization, methodology, writing—reviewing and editing, and supervision.

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Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Declarations

Conflict of interest

The authors have no relevant financial or non-financial interests to disclose. Compliance with ethics requirements This article does not contain any studies with human or animal subject.

Compliance with ethics requirements

This article does not contain any studies with human or animal subject.

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CHAPTER 3 – Obtaining Extracts and Hydrolysates from Cambuci Peel Through Subcritical Water: An In-line Detection Approach.

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Obtaining extracts and hydrolysates from cambuci peel through subcritical water: an in-line detection approach

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Abstract

The cambuci is a fruit that has been attracting interest due to its rich composition of phenolic compounds. However, the fruit's peel is an understudied agro-industrial by-product. This study aimed to assess the effects of temperature and pH in an innovative system. This system integrates an in-line UV-VIS analysis detector into the process of extracting and hydrolyzing bioactive compounds from cambuci peel, enhancing process monitoring. The system operated with 2 grams of cambuci peel in the reactor for 60 minutes, using a water flow rate of 2 mL min^{-1}, 15 MPa, temperatures ranging from 40-160 °C, and pH levels from 4-7. The results indicated that increasing the temperature led to a more favorable response in total phenolic compounds (TPC) and the antioxidant capacity of the samples, while the selected pH range had a minimal impact. Furthermore, the obtained material revealed the presence of citric acid, glucose, arabinose, and fructose, with fructose showing the highest concentration. The in-line coupling system proved to be highly effective in real-time monitoring and determining the completion of the extraction or hydrolysis process, as it allowed for the observation of decreasing concentrations of the compounds of interest. Thus, this study contributes to the understanding and optimization of the extraction of bioactive compounds from cambuci peel, providing valuable insights for future applications in the food and natural products industry.

Keywords: *Boactive compounds, Biomass, Biorefinery, Circular economy, Hydrolysis.*

Introduction

Brazil is one of the world’s most biodiverse nations, boasting an astounding array of over 40,000 distinct plant species, constituting an impressive 20% of the Earth’s plant life (Oliveira et al., 2012). Nonetheless, numerous native and exotic species remain relatively untapped but can serve as a revenue source for the local community. Furthermore, it presents an opportunity to enter niche markets where consumers seek compounds known for their health benefits (Seraglio et al., 2018). The *Myrtaceae* family comprises approximately 145 genera and 5970 species (Barbieri et al., 2017), ranking as one of the prominent families within the Brazilian Atlantic Forest (Stefanello et al., 2011). Nonetheless, several fruits within this family are still classified as wild plants or are traded at a limited scale (Donado-Pestana et al., 2015). Among them are the camu-camu (*M. dubia* McVaugh), the guabiju, the jambolan (*S. cumini* (L.) Skeels), and the cambuci (*Campomanesia phaea* O. Berg) (DonadoPestana et al., 2015; Seraglio et al., 2018).

Cambuci is found mainly in Minas Gerais State, São Paulo (SP) State, and Rio de Janeiro (RJ) State of Brazil. The fruit has a diameter of 5–6 cm, high humidity, acidity, and fiber content (Donado-Pestana et al., 2018). In addition, it is made up of approximately 80.5% pulp, 18.5% peel, and 1% seeds (Tokairin et al., 2018). The annual production of this fruit can reach 300 tons (Donado-Pestana et al., 2015). Certain studies have indicated that cambuci is rich in polyphenolic compounds, including ellagitannins and proanthocyanidins (Paes et al., 2021; Sanches Azevedo et al., 2017), and a high antioxidant capacity in vitro (Sanches Azevedo et al., 2017). The peel becomes the main by-product throughout the processing and represents an approximate waste of 55.5 tons per year. Information on the use of cambuci peel is limited because this fruit lacks a large-scale industry. However, the company Asmussen (Natividade da Serra, SP, Brazil) has reported that some of the by-products are destined for composting. On the other hand, Souza Ronchi (2021) mentions that cambuci peel is used to make tea, representing a potential market opportunity. This by-product remains largely unexplored but could be a source of polyphenolic compounds and sugars of interest to the food industry.

Recently, there has been growing interest in a more sustainable economy, with the concept of a circular economy gaining significance. Its primary goal is the reduction and reuse of byproducts (Grimaldi et al., 2022). Some research has been carried out on the use of by-products in different areas, including animal feed (Murugesan et al., 2021; Rahmani et al., 2022), material processing Grimaldi et al., 2022), industrial chemical products (Arslan et al., 2016), and energy (Chen et al., 2023), which allows establishing that by-products should continue to be studied, especially by their applications in the industry. Besides, over the last 5 decades, environmentally friendly extraction techniques have been developed to minimize the use of organic and synthetic chemicals, shorten extraction times, and enhance the yield and quality of extracts (Jha & Sit, 2022).

The extraction of bioactive compounds from these by-products often involves the use of solvents such as methanol, ethanol, water, acetone, and, in some cases, acidified water (Bunmusik et al., 2022; Chanioti & Tzia, 2018; Cvjetko Bubalo et al., 2016; Kumar et al., 2021). For a sustainable bioactive compound extraction process, the use of “green” extraction methods is essential (Osamede Airouyuwa et al., 2022). These methods are considered safe and sustainable once proven to eliminate toxic solvents, reduce energy consumption, and reduce environmental impact (Barba et al., 2015; Osamede Airouyuwa et al., 2022).

Hydrothermal pretreatment (HTP) under high-pressure conditions becomes a sustainable alternative for the extraction and hydrolysis process. This is because water in a sub/ supercritical state has low viscosity and high diffusivity, making it easier to penetrate the structure of the lignocellulosic matrix. Furthermore, the low dielectric constant of supercritical water enhances the solubility of organic compounds (Maciel-Silva et al., 2019). The manipulation of temperature and pressure conditions allows for adjustments to the physical properties of water, including density, ionic product, and dielectric constant (Cocero et al., 2018).

Coupling an in-line detector to the extraction and hydrolysis process in subcritical water is a combination of technologies that allows real-time analysis of the extracted components. However, a detailed analysis of samples in an innovative subcritical water extraction system with in-line detection is necessary to confirm the benefits of this combination. The hydrothermal treatment of cambuci peel is a promising option for exploring compounds with potential value in the cosmetic, pharmaceutical, and food industries. Moreover, employing water as an extraction solvent facilitates an eco-friendly and cost-effective process, given its short extraction times and high efficiency (Zhang et al., 2020). Additionally, it enables the comprehensive utilization of the entire product, thereby minimizing waste generation and mitigating potential negative environmental impacts.

To achieve this, it is crucial to characterize the extracts and hydrolysates obtained from the system, enabling future applications to be envisioned. The study aims to characterize the extracts and hydrolysates of the cambuci peel obtained through subcritical water in an in-line detection system, varying the temperature and pH.

Materials and Methods

Raw Material

The cambuci peel, provided by the Asmussen Company (Natividade da Serra, SP, Brazil), was dried at 60 °C for 24 h, crushed into 1 mm particles after drying, and stored at−18 °C for physicochemical characterization and obtaining the extracts. The analysis included total solids, volatile solids, moisture, ashes, total protein, and total nitrogen AOAC (2012), and the elementary analysis was conducted (CHNS-O element analyzer, Thermo Fisher Scientific Inc., The Netherlands).

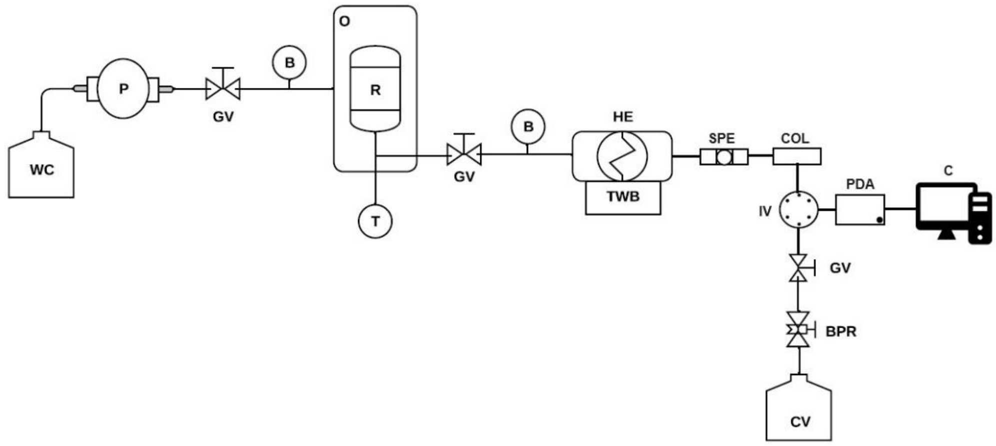
System of Coupling a Detector Analysis In-line on Subcritical Water Process

The extraction and hydrolysis in subcritical water were carried out using a semi-continuous flow reactor with a total volume of 100 mL (**Fig. 1**). The system came equipped with a high-pressure liquid pump featuring a double piston (Model 36-USA), which fed and pressurized the system. A micrometric valve was employed to control the system pressure and was measured with manometers (0–50 MPa). The extraction and hydrolysis temperature were monitored with two thermocouples (type K) inside and outside the reactor. The hydrolysates were cooled using a coil submerged in a thermostatic bath (Marconi, model MA-184, São Paulo, SP, Brazil), which used water as a coolant at 10 °C.

The reactor operating conditions and process variables (temperature and pH) were determined based on information obtained in previous studies (Barroso et al., 2022a, b; Ferreira et al., 2023; Lachos-Perez et al., 2020; Maciel-Silva et al., 2019; Sganzerla et al., 2022). For the hydrothermal pretreatment, the system was operated for 60 min with a feed of 2 g of dry cambuci peel at a constant pressure and flow of 15 MPa and 2 mL min^{-1}. Samples were collected at 6-min intervals to ascertain the kinetics of extraction and hydrolysis. Then, the extracts and hydrolysates were centrifuged at 3000 rpm for 10 min and stored at −18 °C for future analysis. The hydrothermal process of the cambuci peel was performed using the surface response methodology (RSM). It involved the utilization of central composite rotatable design

(CCDR), for two independent variables (temperature and pH). The best extraction/hydrolysis condition will be selected for monitoring in an in-line system (PLE-PDA). It utilizes pressurized liquid extraction (PLE) with detection in-line employing a photodiode array (PDA). The temperatures of 40 and 57 °C will be considered extraction temperatures, and temperatures of 100, 143, and 160 °C will be considered hydrolysis temperatures (**Table 1**).

(a)



(b)

Fig. 1 Experimental unit for extraction and hydrolysis of cambuci peel. (a) schematic diagram; (b) 3D representation. Label: WC, Water container; P, Pump; GV, Gate valve; HE, Heat exchanger; B, Barometer; O, Oven; R, Reactor; T, Thermostat; TWB, Thermostatic water bath; SPE, Solid Phase Extraction; COL, Column; IV, Interface valve; PDA; Detector; C, Computer; BPR, Micrometric valve; CV, Collect vessel

Table 1 Model's experimental parameters and test responses

Test		Non-codified variables Temperature (°C)	Non-codified variables pH	Codified variables Temperature (°C)	Codified variables pH	DPPH (μmol TEAC g^{-1})	FRAP (μmol TEAC g^{-1})	Reducing sugar (mg g^{-1})	TPC (mg g^{-1})
T1	Extraction	57	4.4	–1	–1	58.30	96.15	104.65	10.55
T2	Hydrolysis	143	4.4	+1	–1	56.36	456.57	105.86	45.91
T3	Extraction	57	6.6	–1	+1	57.21	85.70	87.48	10.35
T4	Hydrolysis	143	6.6	+1	+1	52.14	302.29	132.54	38.57
T5	Extraction	40	5.5	–1.41	0	55.80	66.34	85.63	6.72
T6	Hydrolysis	160	5.5	+1.41	0	47.22	501.91	101.59	46.40
T7	Hydrolysis	100	4.0	0	–1.41	61.41	149.55	120.75	17.79
T8	Hydrolysis	100	7.0	0	+1.41	56.69	156.18	53.15	15.89
T9	Hydrolysis	100	5.5	0	0	58.91	165.74	116.01	16.75
T10	Hydrolysis	100	5.5	0	0	58.20	165.35	115.99	15.77
T11	Hydrolysis	100	5.5	0	0	57.45	163.71	114.98	16.24

Extract and Hydrolysate Characterization

Color

The colorimetric coordinates $L*$ (lightness), $a*$ (red/green value), and $b*$ (blue/yellow value) of the CIELab system were obtained by measuring transmittance values every 5 nm between 340 and 830 nm in a UV-Vis spectrophotometer (Model UV-M51, Bell Photonics) (Gilchrist & Nobbs, 2017; Ohta & Robertson, 2006). The hue angle (h°) was calculated using Formula 1.

$$h° = \tan^{-1}\left(\frac{b^*}{a^*}\right) \tag{1}$$

pH

The pH was measured using a digital pH meter (model THS-3E- USA) at 25 °C. The pH meter was previously calibrated with buffer solutions.

Antioxidant activity through free radical DPPH and FRAP assay

The antioxidant capacity of the cambuci peel extracts and hydrolysates was assessed by measuring their ability to inhibit the 2,2-diphenyl-1-picrylhydrazyl (DPPH) free radical (DA SILVEIRA et al., 2018). In this assay, 150 µL of the sample was mixed with a 5.85 mL DPPH solution and incubated in the dark for 30 min. After, the absorbance at 515 nm was determined using a UV-Vis spectrophotometer (Model UV-M51, Bell Photonics). A Trolox standard curve (50-1000 µM) was used for quantification ($y = -0.0006x + 0.6762$, $R^2 = 0.9922$). The analysis was carried out in triplicate ($n = 3$). The results were expressed as µmol of Trolox equivalent antioxidant capacity (TEAC) g^{-1} dry cambuci peel (µmol g^{-1}).

The Ferric Reducing Antioxidant Power (FRAP) assay, modified from the procedure outlined by Benzie & Strain, (1996), comprised the combination of 200 µL of the sample with 200 µL of a 3 mM ferric chloride solution and 3.6 mL of a 2,4,6-Tris(2-Pyridyl)-S-Triazine (TPTZ) solution. This mixture underwent incubation at 37 °C for a duration of 30 minutes, followed by 10 minutes of rest in a lightless setting. Subsequently, the absorbance at 620 nm was measured in a UV-Vis spectrophotometer (Model UV-M51, Bell Photonics). A Trolox calibration curve (100-1000 µM) was used for quantification ($y = 0.0018x + 0.0319$, $R^2 = 0.9976$). The analysis was conducted in triplicate ($n = 3$). The results were expressed as µmol of Trolox equivalent antioxidant capacity (TEAC) g^{-1} dry cambuci peel (µmol g^{-1}).

Total Phenolic Compounds

TPC was measured using the Folin–Ciocalteu method (Singleton et al., 1999) with modifications. Added 60 µL of samples to a mixture containing 300 µL of Folin–Ciocalteu reagent and 3 mL of distilled water. The solution was homogenized, and after 3 min in the dark environment, 0.9 mL of sodium carbonate (15%) and 1.74 mL of distilled water were added. After 2 h, the absorbance at 765 nm was determined using a UV-Vis spectrophotometer (Model UV-M51, Bell Photonics). The values were determined based on a calibration curve (0–1000 µM) using a gallic acid solution ($y = 0.0005x – 0.0127$, $R^2 = 0.9935$). The results were reported in mg gallic acid equivalent (GAE) g^{-1} dry cambuci peel.

Reducing Sugars

The concentration of reducing sugars (RS) was assessed through a Somogyi–Nelson Method, described by Norton (1944). The absorbance of the reaction was determined at 540 nm using a UV-Vis spectrophotometer (Model UV-M51, Bell Photonics). A calibration curve of glucose was used for quantification (50–500 mg L^{-1}) ($y = 5000.36x -41.108$, $R^2 = 0.9862$). The analysis was performed in triplicate ($n = 3$), and the results were reported as mg glucose g^{-1} dry peel of cambuci.

Sugars, Citric Acid, and Inhibitors

Sugars, citric acid, and inhibitors were quantified utilizing HPLC coupled with a refractive index detector (RID), following the procedure described by Barroso et al. (2022a, b). Concentrations of glucose, fructose, arabinose, citric acid, and 5-HMF were computed based on calibration curves for each corresponding standard. The results were reported as mg g^{-1} of dried cambuci peel.

UPLC-PDA-MS Analysis

After establishing optimal conditions, the cambuci peel extracts were diluted fivefold in water, filtered in a nylon filter of 0.22 µm, and further injected in an ultra-performance liquid chromatography coupled to a photodiode array and mass spectrometry (UPLC-PDA-MS) (Acquity, Waters Co., Milford, MA, USA). The separation system was performed in an Acquity UPLC BEH C18 50×2.1 mm, 1.7 µm analytical column, and the mobile phases consisted of water (A) and acetonitrile (B), both acidified with 0.1% acetic acid (v/v). The gradient elution was 2% B (0–1 min), 2–15% B (1–2 min), 15–35% B (2–8 min), 35–100% B (8–10 min), and 100% B (10–12 min). The chromatography conditions were a flow rate of 0.5 mL/min, a column oven at 40 °C, and an injection volume of 3 μL. UV spectra were recorded from 210 to 400 nm at λmax of 254 nm. MS analysis was performed in positive and negative ionization mode (100–1000 Da) with cone voltage of 15 V and 30 V, respectively, capillary voltage of 0.8 kV, and probe at 600 °C. The software Empower 3 was applied for data processing (Waters Alliance, Milford, MA, USA). The term “fraction” denotes the specific time intervals during extraction. For instance, fraction 1 corresponds to the initial 6 min, fraction 2 to 12 min, and so forth, with fraction 10 representing the entire 60-min extraction period.

Statistical Analysis

The results were subjected to statistical analysis using one-way analysis of variance (ANOVA), and significant differences at the 5% level were further assessed through Tukey’s test. Statistical analysis was conducted using Statistica® software (version 10.0, StatSoft Inc., OK, USA). The experimental results for antioxidant activity, phenolic compounds, reducing sugars, and organic acids were analyzed by response surface methodology (RSM).

Results and Discussion

Evaluation of Raw Material

The composition of dried cambuci peel is presented in Table 2. The value obtained for the moisture of the cambuci peel was 7.36±0.11%. The protein results (4.93±0.04%) are higher than those reported by Sanches Azevedo et al. (2017) for the cambuci pulp (2.09±0.01%) and even higher than those reported by Damiani et al. (2011) for araça peel (1.39±0.76%), a fruit that also belongs to the *Myrtaceae* family. On the other hand, the ash content of the cambuci peel is higher than those reported by Schiassi et al. (2018) for the araça and cagaita pulp (belonging to the *Myrtaceae* family). However, the ashes obtained from the cambuci peel (1.23 ± 0.11%) turn out to be approximately 50% of that obtained in the pulp of this fruit, as reported by Sanches Azevedo et al. (2017) and Sanches (2013). Elementary analysis shows that the cambuci peel is composed mainly of oxygen (48.70%) and carbon (44.38%) and to a lesser extent, hydrogen (5.93%) and nitrogen (1.00%), values close to those reported by Rojas-González et al. (2019) for by-products such as passion fruit peel, soursop peel, and pineapple peel.

Table 2 Composition of dried cambuci peel

Parameters	Cambuci peel
Total solids (%)	92.64 ± 0,11
Volatile solids (%)	91.41 ± 0,01
Moisture (%)	7.36 ± 0,11
Total protein (%)	4.93 ± 0,04
Total nitrogen (%)	0.79 ± 0,01
Elementary Analysis	
Ashes (%)	1.23 ± 0,11
Nitrogen (%)	1.00
Carbon (%)	44.38
Hydrogen (%)	5.93
Sulfur (%)	0
Oxygen (%)	48.70

The results are presented as the mean ± standard deviation. The analysis was performed in triplicate (n=3).

Color

Figure 2 presents the color of extracts and hydrolysates for 11 tests. The extract obtained at 40 °C (T5) had a lower color intensity during all the collection times. Likewise, at 57 °C (T1 and T3), the extracts show less color intensity in the first 12 min, increasing after up to minute 48. Later, intensity decreases, possibly due to extraction process completion (Ferreira et al., 2023). At 100 °C (T7-T11), between 12 and 24 min, slight darkening appears in extracts. On the other hand, the tests at 143 and 160 °C (T2, T4, and T6) show significant color change, becoming darker and browner. This can be attributed to Maillard reaction products and sugar decomposition products, also called caramelization products, which are directly related to the high temperatures of the process (Agcam, 2022). Similar results have been observed in extracts and hydrolysates of jaboticaba peel (Barroso et al., 2022a, b), pitaya peel (Ferreira et al., 2023), and açai seeds (Maciel-Silva et al., 2019).

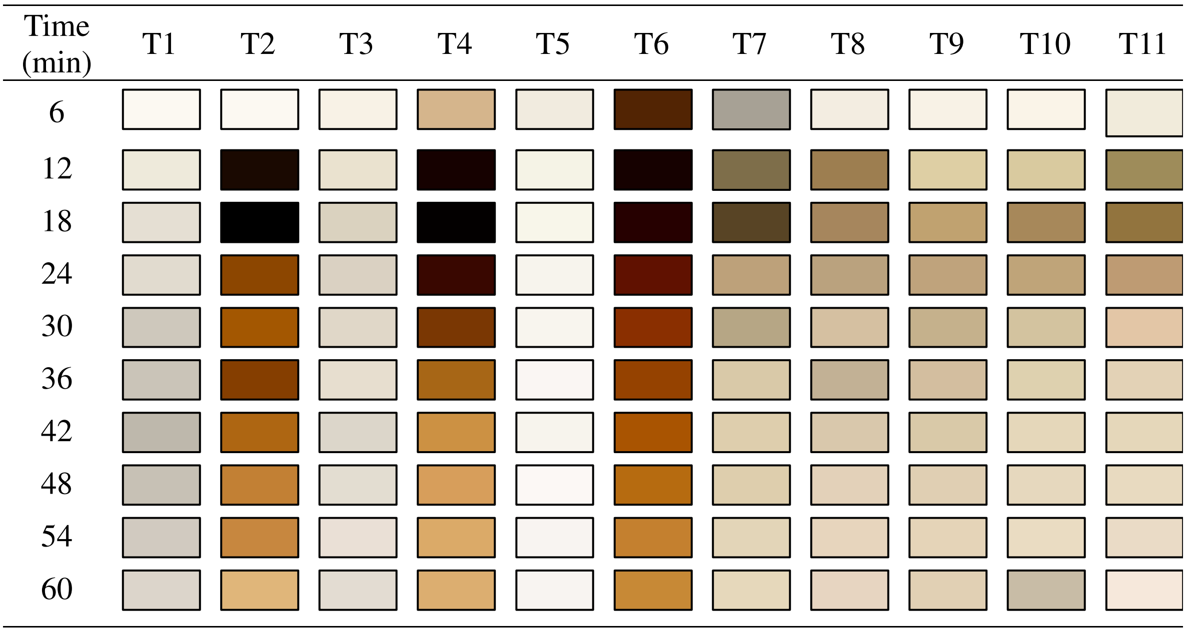


Fig. 2 Visual depiction of the color of the extracts and hydrolysates acquired from the cambuci peel

Table 3 presents the CIELab color appearance parameters (h°, b*, L*, and a*) of the extracts and hydrolysates post-hydrothermal pretreatment. For the extracts that were obtained at low temperatures (40 and 57 °C), the luminosity values (L*) ranged between 74.68 and 98.04, which indicates that the coloration of the extract is closer to white (Yee et al., 2023). For the tests carried out at 100 °C, the values of L* oscillated between 30.27 and 95.86, which allows for determining a greater variation in the luminosity of the extracts with increasing temperature. Likewise, significant differences were found between the collection times of hydrolysates at 100 °C. The hydrolysates at temperatures of 143 and 160 °C presented an oscillation between 0.08 and 97.42 for this parameter, with significant differences for all collection times. However, it was possible to establish that the values closest to 0.08 were found at 12 and 18 min and gradually increased to 60 min.

For the parameter a* (greenness (−) to redness (+)), extraction temperatures yielded the lowest values (40 and 57 °C), varying between− 0.48 and 1.18. Furthermore, extraction at 40 °C showed no significant differences in any sampling times by the Tukey test. For hydrolysis at 143 °C and pH 4.4, the maximum value was obtained at 36 min (a*=29.01). In the case of hydrolysis at 143 °C and pH 6.6 and hydrolysis at 160 °C, the highest values of a* parameter were 27.82 and 36.56, respectively, and were found at 30 min. As expected, the highest values for the a* parameter were found in the samples subjected to higher temperatures, which could induce oxidation of the phenolics present in the extracts and generate a reddish color (He et al., 2019; Yao et al., 2023). The highest a* values for hydrolysates at 100 °C were achieved between 18 and 24 min during the hydrothermal process.

Analyzing the b* parameter, it is evident that the extraction temperatures displayed lower values, ranging from 1.98 to 10.87. Regarding hydrolysis temperature, the peak value (b*=58.28) occurred at 160 °C, precisely at 42 min into the process. Based on the above, it can be concluded that higher temperatures promote the extraction of water-soluble phenolic compounds associated with reddish and yellowish colors (Ali et al., 2019).

For the extracts and hydrolysates obtained in the hydro - thermal process, the h° angles varied between 17.26 and 88.58. Regarding extraction temperatures, the extracts have hue angles exceeding 79.55°, suggesting a tendency towards yellow color. In contrast, hydrolyzed at 100 °C, display hue angles range from 68.97 to 88.34, indicating a color between red and yellow. For hydrolysates at temperatures of 143 °C and 160 °C, the h° values indicate a brown coloration, aligning with the coloration seen in **Fig. 2**.

Table 6 Color parameters of the samples obtained from the cambuci peel through hydrothermal processing

Parameters	**T1**	**T2**	**T3**	**T4**	**T5**	**T6**	**T7**	**T8**	**T9**	**T10**	**T11**
*L**											
6 min	97.45 ± 0.05 ^{a}	97.42 ± 0.04 ^{a}	95.17 ± 0.09 ^{a}	75.8 ± 0.38 ^{f}	93.12 ± 0.10 ^{d}	20.11 ± 0.39 ^{f}	66.62 ± 0.09 ^{a}	93.60 ± 0.11 ^{a}	95.38 ± 0.11 ^{a}	95.86 ± 0.09 ^{a}	93.10 ± 0.11 ^{e}
12 min	92.72 ± 0.10 ^{b}	3.38 ± 0.11 ^{g}	90.07 ± 0.09 ^{c}	2.11 ± 0.07 ^{h}	95.17 ± 0.08 ^{c}	1.98 ± 0.07 ^{g}	47.09 ± 0.30 ^{b}	55.17 ± 0.38 ^{h}	83.40 ± 0.31 ^{g}	81.71 ± 0.31 ^{c}	59.60 ± 0.40 ^{c}
18 min	89.13 ± 0.11 ^{c}	0.08 ± 0.00 ^{h}	84.84 ± 0.15 $^{d, e}$	0.29 ± 0.00 ^{h}	96.46 ± 0.06 ^{g}	3.53 ± 0.12 ^{g}	30.27 ± 0.28 ^{c}	57.86 ± 0.38 ^{g}	68.23 ± 0.39 ^{f}	58.90 ± 0.38 ^{g}	51.61 ± 0.42 ^{d}
24 min	87.57 ± 0.09 ^{d}	37.67 ± 0.56 ^{a}	84.35 ± 0.16 ^{e}	10.05 ± 0.30 ^{e}	95.70 ± 0.05 ^{b}	20.38 ± 0.41 ^{f}	68.10 ± 0.34 ^{g}	67.71 ± 0.31 ^{f}	68.30 ± 0.34 ^{f}	68.51 ± 0.36 ^{f}	67.00 ± 0.37 ^{b}
30 min	80.67 ± 0.10 ^{g}	45.82 ± 0.62 ^{f}	86.30 ± 0.13 ^{b}	31.67 ± 0.51 ^{d}	96.51 ± 0.05 ^{g}	33.01 ± 0.54 ^{d}	68.50 ± 0.25 ^{g}	79.02 ± 0.25 ^{d}	73.00 ± 0.30 ^{d}	79.49 ± 0.28 ^{d}	82.00 ± 0.27 ^{a}
36 min	79.26 ± 0.09 ^{h}	35.34 ± 0.54 ^{a}	88.38 ± 0.12 ^{f}	49.43 ± 0.60 ^{c}	97.25 ± 0.02 ^{e}	38.83 ± 0.58 ^{c}	81.55 ± 0.25 ^{f}	73.10 ± 0.22 ^{e}	78.50 ± 0.26 ^{c}	83.70 ± 0.24 ^{b}	85.35 ± 0.23 ^{h}
42 min	74.68 ± 0.11 ^{j}	50.76 ± 0.63 ^{e}	85.41 ± 0.13 ^{d}	65.66 ± 0.61 ^{b}	96.36 ± 0.05 ^{g}	45.86 ± 0.63 ^{b}	82.85 ± 0.25 $^{e, f}$	80.90 ± 0.22 ^{c}	81.48 ± 0.24 ^{b}	86.77 ± 0.22 ^{h}	86.40 ± 0.21 $^{g, h}$
48 min	77.89 ± 0.10 ^{i}	59.17 ± 0.63 ^{d}	88.19 ± 0.12 ^{f}	69.68 ± 0.58 ^{a}	98.04 ± 0.02 ^{a}	53.18 ± 0.65 ^{a}	83.19 ± 0.24 ^{e}	84.88 ± 0.19 ^{b}	83.65 ± 0.23 ^{g}	87.45 ± 0.20 ^{h}	87.52 ± 0.19 $^{f, g}$
54 min	82.04 ± 0.09 ^{f}	62.06 ± 0.60 ^{c}	89.82 ± 0.10 ^{c}	73.19 ± 0.55 ^{g}	96.97 ± 0.03 $^{e, f}$	59.54 ± 0.64 ^{e}	85.64 ± 0.23 ^{d}	86.42 ± 0.19 ^{i}	85.54 ± 0.22 ^{e}	87.92 ± 0.21 ^{h}	88.01 ± 0.19 ^{f}
60 min	85.40 ± 0.08 ^{e}	76.47 ± 0.51 ^{b}	87.79 ± 0.10 ^{f}	74.04 ± 0.52 $^{f, g}$	96.85 ± 0.03 ^{f}	62.36 ± 0.63 ^{e}	86.99 ± 0.22 ^{d}	86.33 ± 0.18 ^{i}	84.29 ± 0.23 $^{e, g}$	76.83 ± 0.19 ^{e}	92.64 ± 0.12 ^{e}
a*											
6 min	-0.31 ± 0.11 ^{a}	-0.27 ± 0.10 ^{d}	-0.13 ± 0.19 ^{a}	5.78 ± 0.78 ^{e}	-0.23 ± 0.20 ^{a}	20.71 ± 0.13 ^{e}	0.27 ± 0.20 ^{b}	-0.03 ± 0.22 ^{a}	-0.02 ± 0.25 ^{a}	-0.09 ± 0.21 ^{d}	0.01 ± 0.24 ^{c}
12 min	-0.39 ± 0.20 ^{a}	6.23 ± 0.07 ^{b}	0.74 ± 0.32 ^{a}	7.05 ± 0.31 ^{e}	-0.36 ± 0.16 ^{a}	8.31 ± 0.47 ^{b}	3.22 ± 0.62 $^{a, c}$	6.70 ± 0.69 ^{b}	2.12 ± 0.67 $^{a, d}$	1.93 ± 0.66 $^{c, d}$	1.58 ± 0.13 $^{c, d}$
18 min	0.09 ± 0.20 $^{a, b}$	0.22 ± 0.02 ^{d}	0.84 ± 0.31 ^{a}	1.14 ± 0.08 ^{a}	-0.48 ± 0.12 ^{a}	18.37 ± 1.29 ^{e}	5.82 ± 0.47 ^{a}	7.32 ± 0.64 ^{b}	5.94 ± 0.74 ^{b}	6.32 ± 0.69 ^{a}	6.30 ± 0.74 ^{b}
24 min	0.06 ± 0.17 $^{a, b}$	28.72 ± 0.32 ^{e}	0.53 ± 0.26 ^{a}	24.41 ± 0.48 $^{b, c}$	-0.18 ± 0.10 ^{a}	35.89 ± 0.76 ^{d}	4.96 ± 0.64 $^{a, c}$	5.10 ± 0.51 $^{b, c}$	5.07 ± 0.62 $^{b, c}$	4.99 ± 0.68 $^{a, b}$	9.06 ± 0.73 ^{a}
30 min	0.79 ± 0.19 $^{b, c}$	28.34 ± 0.53 ^{e}	0.56 ± 0.23 ^{a}	27.82 ± 0.16 ^{b}	-0.19 ± 0.09 ^{a}	36.56 ± 0.22 ^{d}	2.84 ± 0.47 $^{b, c}$	3.97 ± 0.40 ^{c}	4.61 ± 0.51 $^{b, c, d}$	2.90 ± 0.55 $^{b, c}$	6.05 ± 0.69 ^{b}
36 min	0.89 ± 0.18 $^{b, c}$	29.01 ± 0.22 ^{e}	0.81 ± 0.26 ^{a}	23.25 ± 0.72 ^{c}	-0.32 ± 0.05 ^{a}	33.58 ± 0.12 $^{c, d}$	2.80 ± 0.46 $^{b, c}$	3.82 ± 0.32 ^{c}	3.81 ± 0.43 $^{b, c, d}$	2.14 ± 0.46 $^{c, d}$	3.39 ± 0.49 ^{d}
42 min	1.32 ± 0.19 ^{c}	27.27 ± 0.61 ^{e}	0.7 ± 0.22 ^{a}	17.86 ± 0.99 ^{d}	-0.07 ± 0.09 ^{a}	31.00 ± 0.39 ^{c}	2.86 ± 0.44 $^{b, c}$	4.04 ± 0.26 ^{c}	3.58 ± 0.37 $^{b, c, d}$	2.01 ± 0.40 $^{c, d}$	2.72 ± 0.39 ^{d}
48 min	1.18 ± 0.19 ^{c}	21.91 ± 0.83 ^{c}	0.48 ± 0.20 ^{a}	14.81 ± 1.00 $^{d, f}$	-0.17 ± 0.05 ^{a}	26.62 ± 0.69 ^{a}	2.70 ± 0.42 $^{b, c}$	3.60 ± 0.19 ^{c}	3.19 ± 0.33 $^{c, d}$	1.86 ± 0.38 $^{c, d}$	2.88 ± 0.32 ^{d}
54 min	1.07 ± 0.16 ^{c}	20.41 ± 0.83 ^{c}	0.82 ± 0.21 ^{a}	13.26 ± 0.94 ^{f}	0.04 ± 0.06 ^{a}	21.98 ± 0.87 ^{e}	2.59 ± 0.40 $^{b, c}$	3.94 ± 0.13 ^{c}	3.13 ± 0.30 $^{c, d}$	1.98 ± 0.38 $^{c, d}$	2.83 ± 0.24 ^{d}
60 min	0.76 ± 0.15 $^{b, c}$	10.16 ± 1.00 ^{a}	0.52 ± 0.17 ^{a}	12.21 ± 0.90 ^{f}	-0.05 ± 0.06 ^{a}	18.54 ± 0.98 ^{e}	2.47 ± 0.38 $^{b, c}$	4.21 ± 0.08 ^{c}	3.54 ± 0.29 $^{b, c, d}$	1.71 ± 0.35 $^{c, d}$	3.11 ± 0.20 ^{d}

Parameters	**T1**	**T2**	**T3**	**T4**	**T5**	**T6**	**T7**	**T8**	**T9**	**T10**	**T11**
b*											
6 min	3.92 ± 0.03 ^{c}	3.39 ± 0.03 ^{d}	6.33 ± 0.02 ^{g}	27.15 ± 0.13 ^{e}	7.25 ± 0.04 ^{a}	31.72 ± 0.49 ^{b}	6.69 ± 0.01 ^{j}	7.58 ± 0.02 ^{h}	7.68 ± 0.01 ^{j}	7.01 ± 0.02 ^{j}	8.25 ± 0.03 ^{i}
12 min	8.07 ± 0.08 ^{d}	5.22 ± 0.16 ^{c}	10.86 ± 0.00 ^{i}	3.40 ± 0.12 ^{g}	6.20 ± 0.06 ^{b}	3.15 ± 0.11 ^{d}	22.70 ± 0.02 ^{b}	29.26 ± 0.04 ^{a}	23.17 ± 0.02 ^{c}	23.41 ± 0.05 ^{c}	30.32 ± 0.02 ^{b}
18 min	8.02 ± 0.06 ^{d}	-0.07 ± 0.00 ^{e}	10.87 ± 0.00 ^{i}	0.28 ± 0.01 ^{h}	4.61 ± 0.05 ^{c}	5.70 ± 0.21 ^{c}	21.56 ± 0.04 ^{c}	28.34 ± 0.06 ^{b}	29.74 ± 0.02 ^{a}	28.92 ± 0.03 ^{a}	33.26 ± 0.05 ^{a}
24 min	6.64 ± 0.04 ^{g}	53.62 ± 0.34 ^{g}	9.19 ± 0.01 ^{a}	16.88 ± 0.49 ^{f}	3.93 ± 0.03 ^{d}	34.36 ± 0.67 ^{a}	25.74 ± 0.03 ^{a}	22.01 ± 0.04 ^{c}	25.23 ± 0.03 ^{b}	26.72 ± 0.02 ^{b}	28.07 ± 0.04 ^{c}
30 min	7.15 ± 0.00 ^{e}	55.59 ± 0.31 ^{f}	8.23 ± 0.01 ^{c}	47.75 ± 0.47 ^{i}	3.38 ± 0.02 ^{e}	53.11 ± 0.67 ^{f}	18.09 ± 0.01 ^{d}	18.08 ± 0.02 ^{d}	21.74 ± 0.03 ^{d}	20.60 ± 0.01 ^{d}	19.64 ± 0.00 ^{d}
36 min	6.65 ± 0.01 ^{g}	51.01 ± 0.42 ^{h}	8.92 ± 0.01 ^{b}	51.65 ± 0.25 ^{a}	2.14 ± 0.03 $^{f, g}$	57.44 ± 0.43 ^{e}	17.80 ± 0.00 ^{e}	16.00 ± 0.00 ^{i}	18.92 ± 0.01 ^{e}	17.17 ± 0.01 ^{e}	16.38 ± 0.02 ^{e}
42 min	7.13 ± 0.03 $^{e, f}$	54.43 ± 0.35 $^{f, g}$	7.67 ± 0.00 ^{d}	48.43 ± 0.25 ^{i}	3.32 ± 0.02 ^{e}	58.28 ± 0.27 ^{e}	17.38 ± 0.01 ^{f}	15.75 ± 0.02 ^{i}	17.35 ± 0.01 ^{f}	15.28 ± 0.01 ^{f}	14.90 ± 0.01 ^{f}
48 min	6.94 ± 0.03 ^{f}	49.70 ± 0.34 ^{h}	6.91 ± 0.00 ^{f}	44.46 ± 0.22 ^{b}	1.98 ± 0.02 ^{g}	57.14 ± 0.25 ^{e}	17.21 ± 0.01 ^{g}	14.10 ± 0.06 ^{e}	16.03 ± 0.02 ^{h}	14.47 ± 0.02 ^{h}	13.68 ± 0.04 ^{g}
54 min	5.83 ± 0.03 ^{a}	47.54 ± 0.32 ^{a}	7.17 ± 0.02 ^{e}	41.44 ± 0.19 ^{c}	2.28 ± 0.00 ^{f}	54.14 ± 0.21 ^{f}	16.51 ± 0.01 ^{h}	13.77 ± 0.07 ^{f}	15.67 ± 0.03 ^{i}	14.72 ± 0.01 ^{g}	13.08 ± 0.04 ^{h}
60 min	5.11 ± 0.02 ^{b}	36.10 ± 0.25 ^{b}	5.84 ± 0.01 ^{h}	39.25 ± 0.17 ^{d}	2.14 ± 0.01 $^{f, g}$	52.88 ± 0.15 ^{f}	15.76 ± 0.01 ^{i}	13.26 ± 0.07 ^{g}	16.39 ± 0.04 ^{g}	12.85 ± 0.00 ^{i}	8.09 ± 0.03 ^{i}
h°											
6 min	85.47 ± 1.63 $^{a, b}$	85.52 ± 1.63 ^{a}	88.28 ± 1.18 ^{a}	77.98 ± 1.63 ^{e}	88.21 ± 1.55 ^{a}	56.86 ± 0.56 $^{h, i}$	87.73 ± 1.67 ^{a}	88.34 ± 0.23 ^{a}	88.17 ± 0.11 ^{a}	88.28 ± 0.74 ^{a}	88.33 ± 0.06 ^{a}
12 min	87.22 ± 1.44 $^{a, b}$	39.94 ± 0.50 ^{c}	86.13 ± 1.65 ^{a}	25.74 ± 0.21 ^{c}	86.71 ± 1.46 ^{a}	20.78 ± 0.41 ^{b}	81.93 ± 1.53 $^{a, b}$	77.12 ± 1.31 ^{b}	84.79 ± 1.63 $^{a, b}$	85.31 ± 1.58 $^{b, c}$	87.03 ± 0.24 ^{a}
18 min	88.58 ± 0.63 $_{a}$	17.78 ± 1.51 ^{d}	85.61 ± 1.60 ^{a}	13.83 ± 0.46 ^{d}	84.11 ± 1.48 ^{a}	17.26 ± 0.56 ^{c}	74.89 ± 1.19 ^{b}	75.52 ± 1.24 ^{b}	78.72 ± 1.37 $^{b, c}$	77.68 ± 1.32 ^{b}	79.28 ± 1.25 ^{d}
24 min	88.58 ± 0.47 ^{a}	61.83 ± 0.41 $^{e, f}$	86.70 ± 1.61 ^{a}	34.66 ± 0.25 ^{b}	87.44 ± 1.41 ^{a}	43.75 ± 0.05 ^{a}	79.11 ± 1.39 ^{b}	76.97 ± 1.30 ^{b}	78.64 ± 1.37 $^{b, c}$	79.44 ± 1.40 ^{b}	72.13 ± 1.36 $^{b, c}$
30 min	83.74 ± 1.46 $^{a, b}$	62.99 ± 0.56 $^{e, f}$	86.11 ± 1.59 ^{a}	59.77 ± 0.39 ^{a}	86.77 ± 1.55 ^{a}	55.46 ± 0.18 ^{i}	81.10 ± 1.44 $^{a, b}$	77.62 ± 1.22 ^{b}	78.04 ± 1.31 ^{c}	81.99 ± 1.50 $^{b, c}$	72.91 ± 1.82 $^{b, c, d}$
36 min	82.42 ± 1.49 $^{a, b}$	60.38 ± 0.39 ^{e}	84.85 ± 1.63 ^{a}	65.77 ± 0.76 ^{f}	81.61 ± 1.29 ^{a}	59.69 ± 0.28 $^{g, h}$	81.08 ± 1.43 $^{a, b}$	76.57 ± 1.09 ^{b}	78.63 ± 1.24 $^{b, c}$	82.90 ± 1.51 $^{b, c}$	78.33 ± 1.62 $^{c, d}$
42 min	79.55 ± 1.48 ^{b}	63.39 ± 0.66 $^{e, f}$	84.79 ± 1.63 ^{a}	69.76 ± 1.13 $^{f, g}$	88.44 ± 1.22 ^{a}	61.99 ± 0.41 $^{f, g}$	80.68 ± 1.43 $^{a, b}$	75.61 ± 0.87 ^{b}	78.34 ± 1.17 $^{b, c}$	82.53 ± 1.45 $^{b, c}$	79.66 ± 1.44 ^{d}
48 min	80.38 ± 1.53 ^{b}	66.21 ± 0.95 ^{f}	86.03 ± 1.65 ^{a}	71.58 ± 1.24 ^{g}	85.07 ± 1.48 ^{a}	65.02 ± 0.66 $^{e, f}$	81.11 ± 1.38 $^{a, b}$	75.70 ± 0.65 ^{b}	78.77 ± 1.10 $^{b, c}$	82.70 ± 1.45 $^{b, c}$	78.12 ± 1.25 $^{c, d}$
54 min	79.65 ± 1.53 ^{b}	66.77 ± 0.99 ^{f}	83.48 ± 1.67 ^{a}	72.27 ± 1.26 ^{g}	88.49 ± 1.00 ^{a}	67.91 ± 0.87 $^{d, e}$	81.09 ± 1.35 $^{a, b}$	74.03 ± 0.43 ^{b}	78.71 ± 1.03 $^{b, c}$	82.36 ± 1.43 $^{b, c}$	77.80 ± 0.97 $^{c, d}$
60 min	81.60 ± 1.62 ^{a,b}	74.28 ± 1.58 ^{b}	84.91 ± 1.67 ^{a}	72.72 ± 1.27 $^{e, g}$	88.39 ± 1.34 ^{a}	70.69 ± 1.00 ^{d}	81.11 ± 1.32 $^{a, b}$	72.41 ± 0.21 ^{b}	77.82 ± 0.94 ^{c}	82.45 ± 1.51 $^{b, c}$	68.97 ± 1.15 ^{b}

pH

According to the kinetic curves of the pH (Fig. 3), it is possible to establish that throughout the process of extraction and hydrolysis of 11 tests, pH tended to increase, a trend that was also reported by Castro et al. (2023) for grape pomace. However, within the first 6 min of extraction, a decrease in pH is evident, attributed to the consumption of amino groups and the release of organic acids in the initial stage of the process (Li et al., 2021). Its subsequent increase is associated with the neutralization of organic acids and the formation of products from Maillard reactions (Ferreira et al., 2023). In addition, it was observed that the pH was influenced by temperature, presenting a variation between 2.98 (T1) and 7.87 (T6). Extraction tests (T1, T3, and T5) presented the lowest pH results during the 60 min of the process, reaching a maximum pH value of 4.1. On the other hand, hydrolysis temperatures favored the increase in the pH of the obtained hydrolysates (T6). Similar results were obtained for jabuticaba peel extracts/hydrolysates (Barroso et al., 2022a, b), soybean residues (Vedovatto et al., 2021), and brewers' spent grains (Sganzerla et al., 2022). This behavior could be related to the decrease in amino acids because of the interaction between amino groups with reducing sugars during the Maillard reaction (Jeong et al., 2021), to the neutralization of organic acids (Ferreira et al., 2023), and an increase of $[OH^{-}]$ ions by self-ionization due to high temperatures and pressure (Ahmed & Chun, 2018; Park et al., 2015).

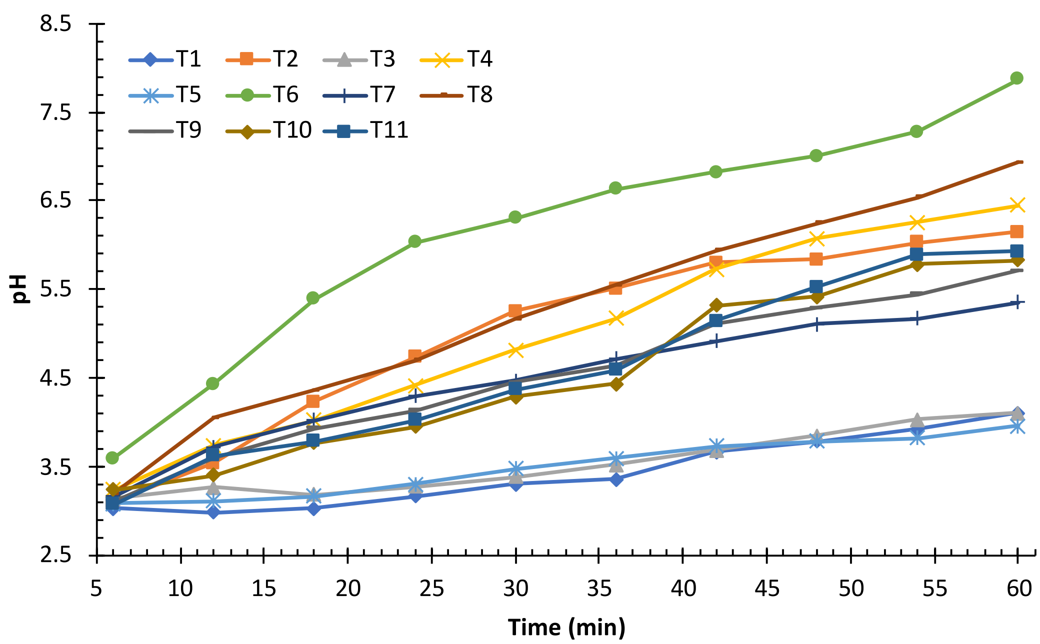


Fig. 3 pH of the samples extracted from the cambuci peel using hydrothermal processing

Antioxidant Activity

Figure 4 illustrates the antioxidant capacity of the cambuci peel extracts/hydrolysates as assessed using the DPPH and FRAP tests. The DPPH method revealed that the antioxidant capacity of tests conducted at 100 °C exhibited a linear trend from 6 to 60 min, with values ranging between 5.60 and 6.21 μmol TEAC g^{-1}. Moreover, hydrolysates obtained at higher temperatures (T2, T4, and T6) yielded lower antioxidant capacity values, ranging from 3.18 to 6.11 μmol TEAC g^{-1}. An explanation for this could be the degradation of tannins because of high temperatures (Pinto et al., 2021).

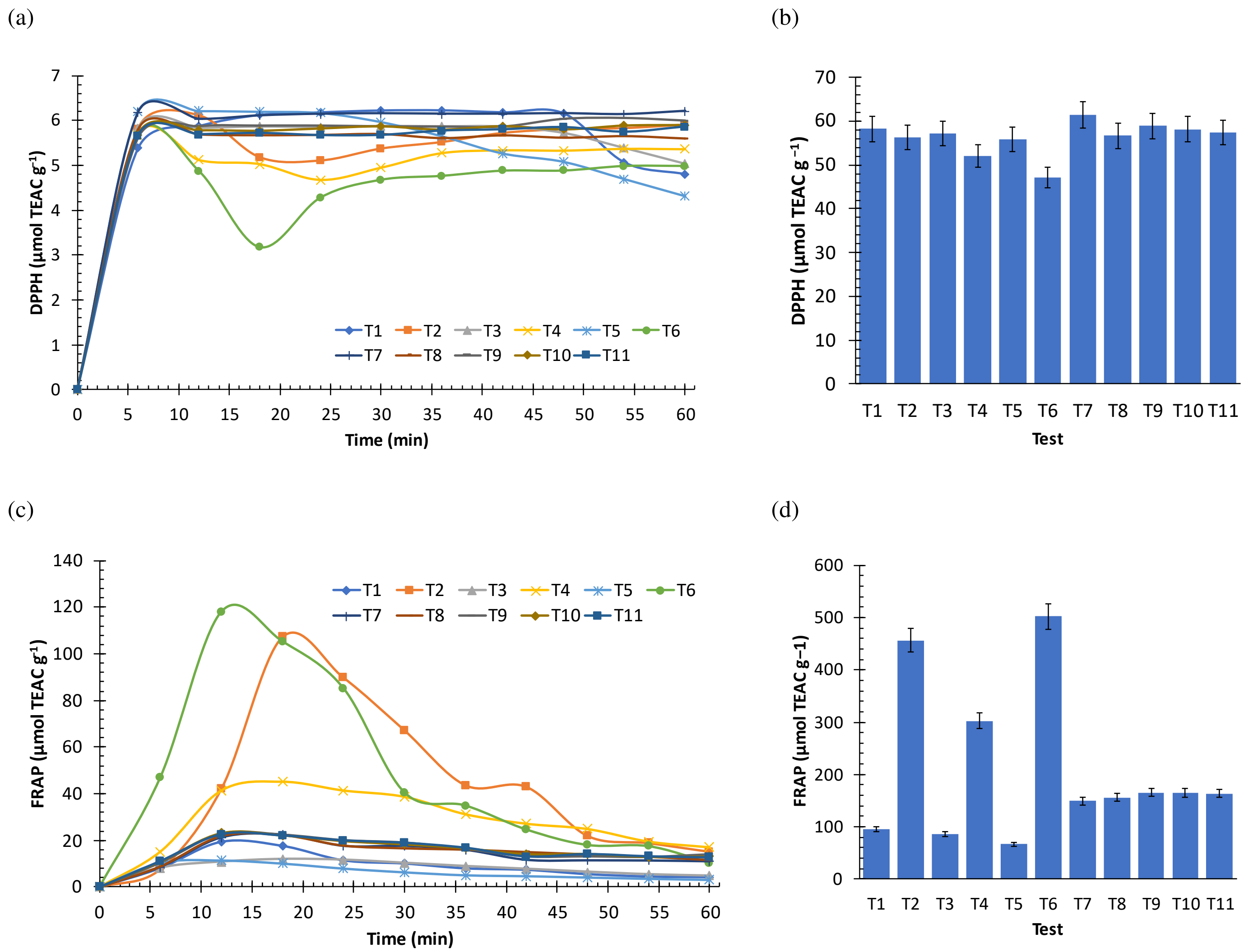


Fig. 4 Antioxidant activity of the samples extracted from the cambuci peel using hydrothermal processing. (a) DPPH; (b) accumulated DPPH; (c) FRAP; (d) accumulated FRAP

For the T1 and T3 extraction tests, carried out at 57 °C, a decrease in antioxidant capacity was obtained after 36 and 42 min of extraction, respectively. The test carried out at 40 °C presented a marked decrease after 24 min of the process. Similar results were obtained by Bodoira et al. (2019) for Pistachio (*Pistacia vera* L.) nuts extract. The accumulated values of antioxidant capacity by the DPPH method (Fig. 4b) allowed to establish that T7 (100 °C and pH 4) had a greater antioxidant capacity (61.41 μmol TEAC g^{-1}).

The FRAP assay revealed that high temperatures favor antioxidant capacity (Fig. 4c). The maximum value of all the kinetics was obtained at 160 °C and pH 5.5 (118.07 μmol TEAC g^{-1}), followed by 107.28 μmol TEAC g^{-1} and 45.26 μmol TEAC g^{-1} (T2 and T4, respectively), obtained at the 18 min of the process. Similar behavior was observed by Gan and Baroutian (2022) from New Zealand Wakame (Undaria pinnatifida) seaweed. Furthermore, the results of antioxidant capacity by this method show a behavior similar to that of phenolic compounds, which indicates a possible correlation between these parameters. Also, it was evident that the extraction temperatures of 40 and 57 °C exhibited the lowest levels of antioxidant capacity throughout the 60-min extraction period, the values oscillated between 4.24 and 19.34 μmol TEAC g^{-1} for T1, between 4.84 and 11.83 μmol TEAC g^{-1} for T3, and between 3.06 and 11.37 μmol TEAC g^{-1} for T5. The cumulative values of antioxidant capacity for this assay (Fig. 4d) revealed that the most favorable condition for antioxidant capacity was T6 (160 °C and pH 5.5), and the most unfavorable condition was T5 (40 °C and pH 5.5).

The antioxidant capacity for the DPPH and FRAP assays was assessed using a central composite rotational design (CCRD) with two independent variables: temperature and pH (Table 1). In the DPPH method, the significant coefficients were associated with temperature (both linear and quadratic terms) and pH (linear term). This method yielded a coefficient of determination (R^2) value of 0.9581. For the FRAP method, the significant coefficients were linear and quadratic temperature, presenting an R^2 of 0.9797, which suggests that the models effectively match the selected parameters. The models for the DPPH and FRAP assays are shown below:

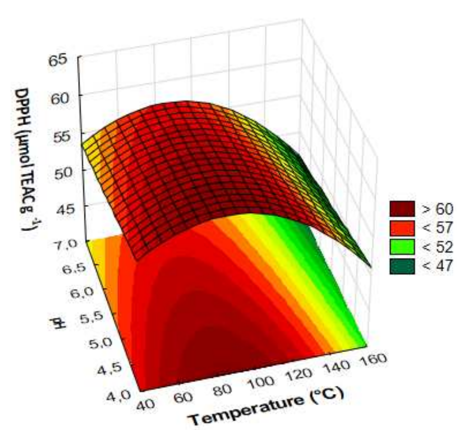
$$\text{DPPH} = 53.5924 + 0.3118\cdot(\text{Temperature}) - 0.0018\cdot(\text{Temperature}^2) - 1.384\cdot(\text{pH}),\ (R^2\ \text{Adj} = 0.9162)$$

$$\text{FRAP} = 169.8880 - 3.6345\cdot(\text{Temperature}) + 0.0356\cdot(\text{Temperature}^2),\ (R^2\ \text{Adj} = 0.9593)$$

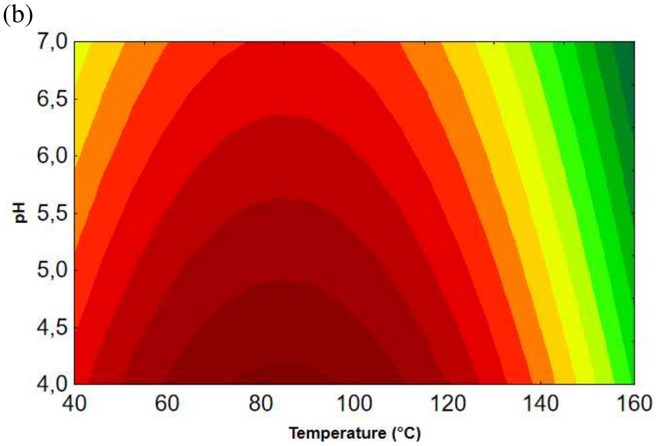
The optimization results for the antioxidant capacity by the DPPH assay were Temp = 79.6 °C, pH = 6.4, and DPPH =58.15 µmol TEAC g^{-1}. For the FRAP essay, the optimization parameters were Temp = 160 °C and FRAP = 500.24 µmol TEAC g^{-1}. By performing the hydrolysis again at 160 °C, it was possible to determine that the antioxidant capacity accumulated by the FRAP method was 483.72 µmol of TEAC g^{-1}, which differs by 3.3% from the value predicted by the model obtained. The behavior of the antioxidant capacity as determined by the FRAP test closely resembled the results reported by Ferreira et al. (2023), who studied the extracts and hydrolysates of pitaya residues using a high-pressure hydrothermal process. However, for the antioxidant activity by the DPPH assay, there was a variation in behavior, which could be attributed to the fact that DPPH has a reduced solubility in aqueous media, which means that this radical has a low availability to interact with antioxidant compounds (Angonese et al., 2021). Furthermore, the pigmentation of some compounds present in the samples could also interfere with absorption due to the spectra overlapping (Yeo & Shahidi, 2019).

Figure 5 illustrates the behavior of antioxidant capacity (DPPH, FRAP, and TPC). Noticeably, the lowest values correspond to the green areas, while the highest values are found in the red or brown areas. This provides an insight into the range of values that these variables can assume for different combinations of temperature and pH. In the case of DPPH, it is evident that both pH and temperature impact the analyzed response (Fig. 5a, b). This suggests that at temperatures around 80 °C and low pH, better antioxidant capacity yields could be generated when employing this method. On the other hand, regarding the FRAP method, it can be concluded that pH does not affect the response variable; instead, it is solely influenced by the temperature of the process.

(a)



(b)



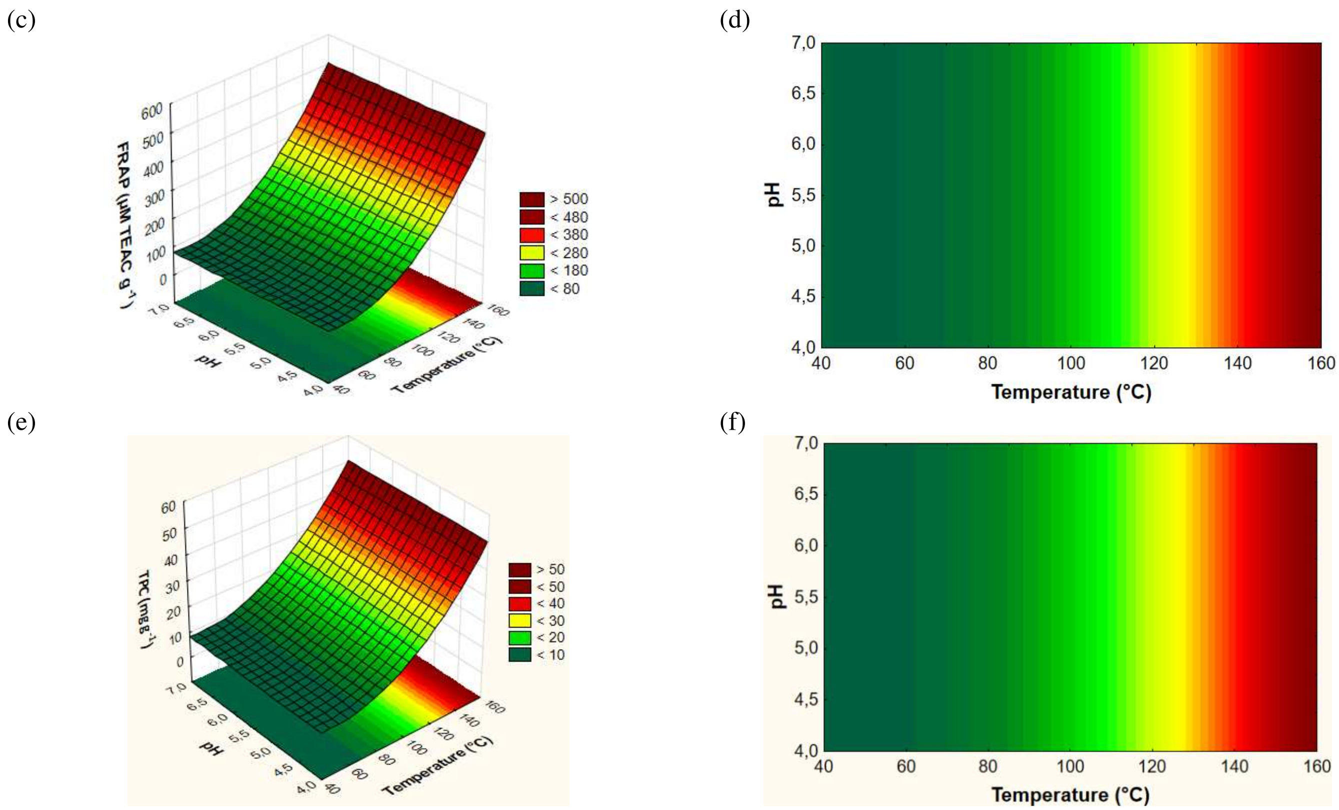


Fig. 5 The influence of temperature and pH on the antioxidant capacity of cambuci peel hydrolysates. (a) DPPH response surface; (b) DPPH contour curve; (c) FRAP response surface; (d) FRAP contour curve; (e) TPC response surface; (f) TPC contour curve

Total Phenolic Compounds

Phenolic compounds were monitored in extracts and hydrolysates of cambuci peel, and the results are presented in Fig. 6. It was observed that the greatest concentration of phenolic compounds was obtained mainly between 12 and 18 min of extraction/hydrolysis. Likewise, it was possible to establish that the hydrolysates at 143 and 160 °C (T2, T4, and T6) presented a higher concentration of phenolic compounds, with maximum values of 12.44 mg g^{-1} (T2), 9.50 mg GAE g^{-1} (T4), and 12.76 mg GAE g^{-1} (T6). Moreover, it was established that extraction temperatures (40 and 57 °C) did not favor the concentration of TPC in the extracts since these tests obtained the lowest concentrations, varying between 0.35 and 1.97 mg GAE g^{-1}. The accumulated concentration of the extracts and hydrolysates of the cambuci peel presented in Fig. 6b makes it easier to visualize the behavior described above. The maximum accumulated concentration of TPC was obtained at 160 °C (46.40 mg GAE g^{-1}), and the lowest accumulated concentration was at 40 °C (6.72 mg GAE g^{-1}).

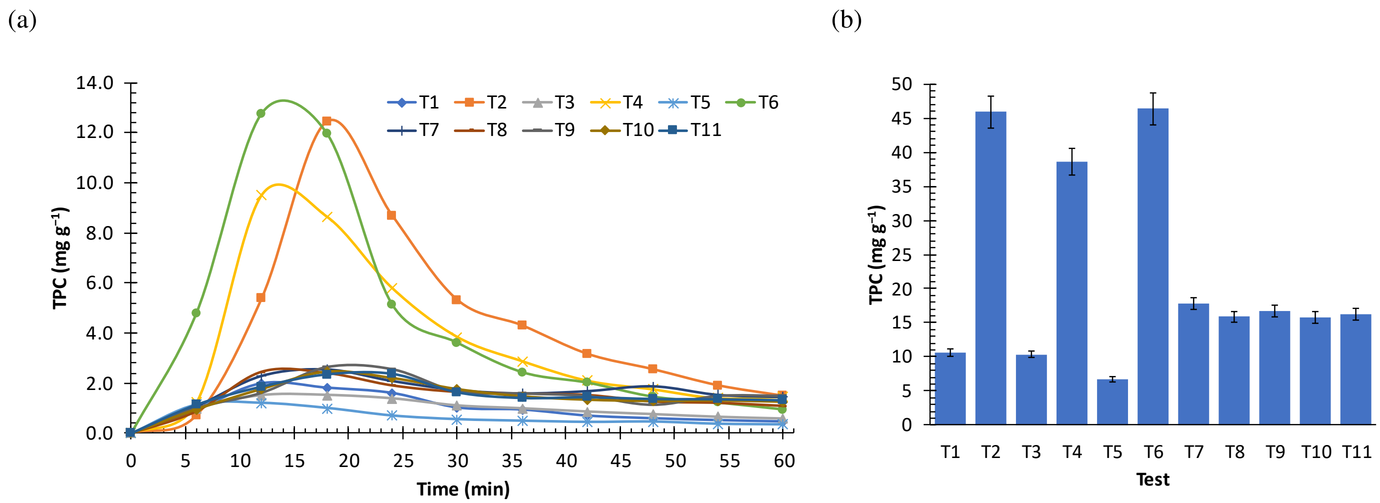


Fig. 6 Kinetic and accumulated performance of total phenolic compounds (TPC) from the cambuci peel through hydrothermal processing. (a) TPC; (b) accumulated TPC

TPC was also evaluated with a CCRD, with temperature and pH as independent variables (Fig. 5e, f). For TPC, the significant coefficients were linear and quadratic temperature, with an R^2 = 0.9776, thus indicating a high correlation between the data and the model presented below.

$$TPC = 15.3136 - 0.3057 \cdot (\text{Temperature}) + 0.0033 \cdot (\text{Temperature}^2), \; (R^2 \text{ Adj} = 0.9553)$$

For TPC, the optimization parameters were Temp=160 °C and TPC=50.88 mg g^{-1}. After performing the hydrolysis again at 160 °C, it was determined that the accumulated TPC was 48.3 mg g^{-1}, which differs by 5.1% from the value predicted by the model obtained. The rise in temperature and the total phenolic compound concentration can be explained by the fact that increased temperature reduces the surface tension of water. This reduction allows water to penetrate the solid matrix more easily and promotes the dilution of the compounds (Herbst et al., 2021). Results with this trend were obtained for potato peel (Singh & Saldaña, 2011), carrot leaves (Song et al., 2018), acerola pomace (Mesquita et al., 2022), and pitaya peel (Ferreira et al., 2023).

Reducing Sugars

The kinetic profile of reducing sugars is presented in Fig. 7. For the tests, the highest yield was obtained during the first 12 min of the extraction/hydrolysis process. In all cases, the kinetics decrease after reaching the highest yield, achieving stabilization after 30 min. The extraction tests presented a maximum reducing sugar yield between 34.20 and 39.35 mg g^{-1}. Meanwhile, the hydrolysis tests carried out at temperatures of 100 and 160 °C presented the highest yields of reducing sugars between 27.33 and 50.42 mg g^{-1}. In the case of the tests carried out at 143 °C (T2 and T4), the reducing sugars obtained were 33.56 and 39.33 mg g^{-1}, respectively. After the first 6 min of extraction/hydrolysis, T6 (160 °C and pH 5.5) was the test with the highest yield of reducing sugars, which could be attributed to an increase in the ionization constant of water at high temperature and pressure (Ahmed & Chun, 2018; Park et al., 2015). These results favor the increase of H+ and OH− ions and facilitate the hydrolysis of cellulose and hemicellulose to monosaccharides (Pattnaik et al., 2021).

From the kinetics of reducing sugars, it is possible to determine that until 30 min (process stabilization), a higher recovery of 86.69% (m/m) is achieved for all extraction and hydrolysis tests. This indicates that the extraction and hydrolysis process could be limited to 30 min. The accumulated values of reducing sugars at the end of the 60 min of extraction/hydrolysis are presented in Fig. 7b. T4 obtained the highest accumulated production of reducing sugars (132.54 mg g^{-1}), followed by treatment T7 with 120.75 mg g^{-1}.

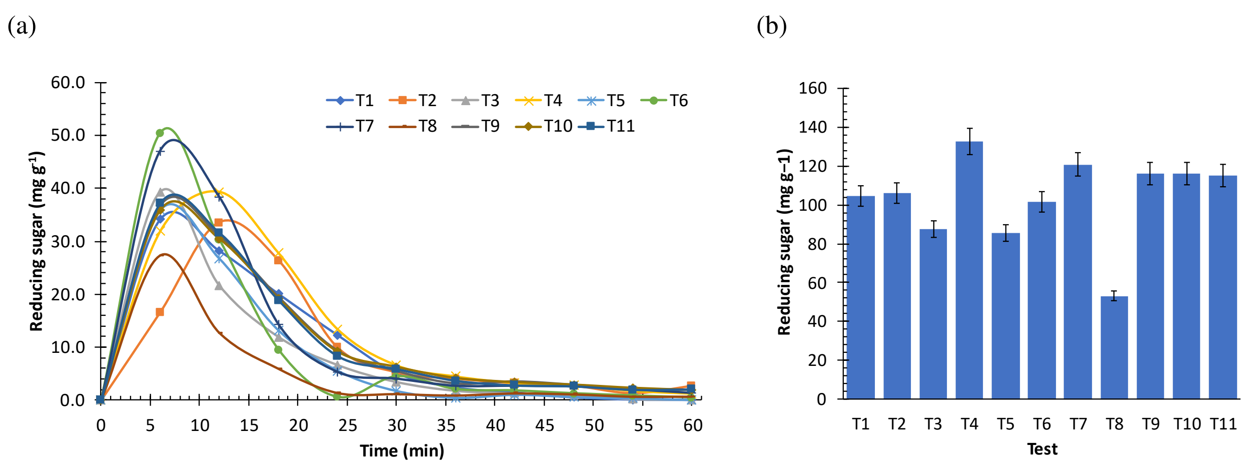
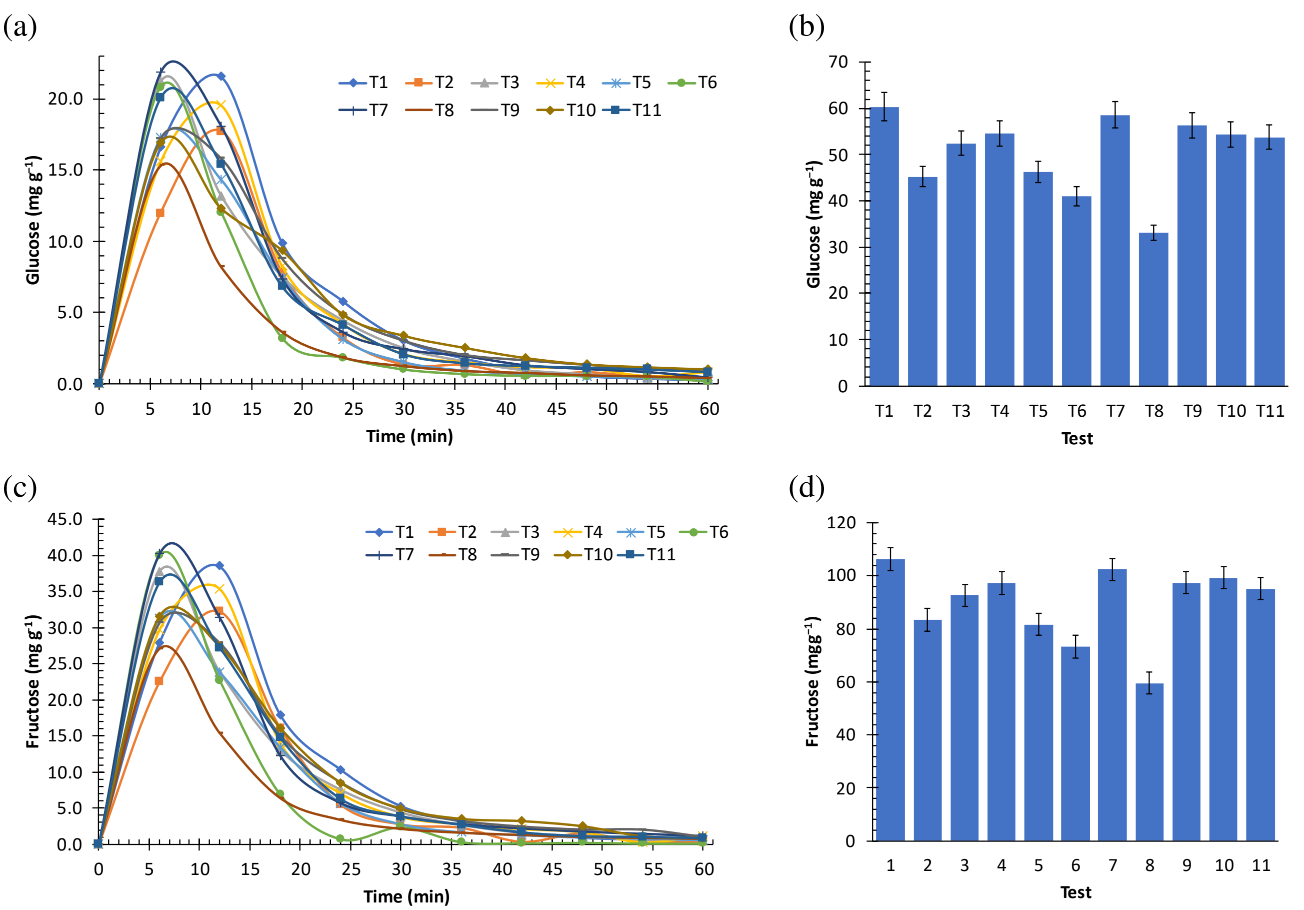


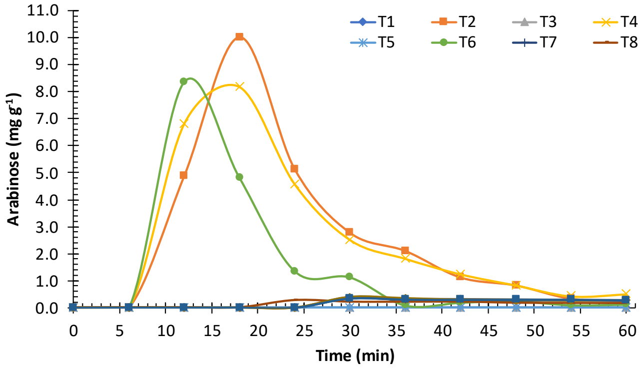
Fig. 7 Kinetic and accumulated performance of the production of reducing sugars obtained from the cambuci peel hydrothermal processing. (a) reducing sugars; (b) accumulated reducing sugars

Sugars, Citric Acid, and Inhibitors

The kinetics and accumulation of sugars such as glucose, fructose, and arabinose obtained in the extraction/hydrolysis process are presented in Fig. 8. The analysis revealed that the sugars that presented the best yields in the cambuci peel were glucose and fructose. The highest glucose and fructose results were obtained in T7 with 21.85 mg g^{-1} and 40.20 mg g^{-1}, respectively, 6 min after starting the process. Furthermore, it is possible to establish that between 6 and 12 min of the extraction/hydrolysis process, there are increased yields of these two monosaccharides for all the tests in the study. At the end of the extraction/hydrolysis, the treatments that obtained the highest accumulated glucose yields were T1 (57 °C and pH 4.4) (106.35 mg g^{-1}) and T7 (100 °C and pH 4.0) (102.4 mg g^{-1}), values higher than those reported for peach palm by-products (glucose 65.5 mg g^{-1}—fructose 53.5 mg g^{-1}) (Giombelli et al., 2020) and orange peel (glucose 50.8 mg g^{-1}—fructose 34.2 mg g^{-1}) (Lachos-Perez et al., 2020).



(e)



(f)

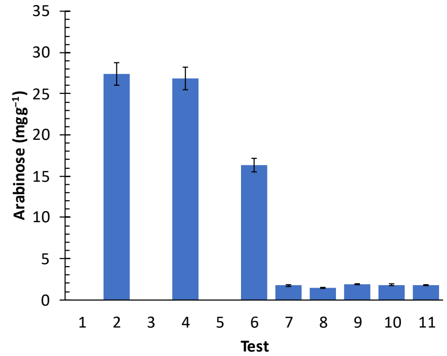


Fig. 8 Kinetic and accumulated performance of monosaccharides obtained from the cambuci peel through hydrothermal processing. (a) glucose; (b) accumulated glucose; (c) fructose; (d) accumulated fructose; (e) arabinose; (f) accumulated arabinose

Arabinose was only present in hydrolysis tests. In the case of the tests carried out at 100 °C, the maximum yield of this monosaccharide was evident 30 min into the process. In comparison, the hydrolysates at 143 °C and 160 °C presented better yields at 18 and 12 min, respectively. The accumulated arabinose maximums were obtained in T2 (27.40 mg g^{-1}) and T4 (26.86 mg g^{-1}). The observation indicates that the monosaccharide is formed due to the hydrolysis process of polysaccharides since elevated temperatures facilitate the breakdown of lignocellulosic structures (Barroso et al., 2022a, b; Castro et al., 2023). The citric acid obtained in the extracts and hydrolysates of the cambuci peel presented its maximum values between 6 and 12 min of the process (Fig. 9), being T2 (23.50 mg g^{-1}), T4 (24.58 mg g^{-1}), and T6 (23.03 mg g^{-1}), the tests with better yields.

The 5-hydroxymethylfurfural (5-HMF) is a fermentation inhibitor that is related to the breakdown of glucose (T. C. G. Oliveira et al., 2020) and the increase in temperature (Martins-Vieira et al., 2023). This was confirmed as the inhibitor 5-HMF was only detected in hydrolysates T4 (143 °C and pH 6.6) and T6 (160 °C and pH 5.5), with accumulated concentrations of 0.87 mg g^{-1} and 0.59 mg g^{-1}, respectively. In both tests, the inhibitor was identified after 12 min of extraction. The presence of this compound in cambuci peel hydrolysates is due to the breakdown of biomass, which utilizes carbohydrates like fructose, glucose, hemicellulose, and cellulose in the formation of 5-HMF (Özkaynak Kanmaz, 2018; Pińkowska et al., 2020).

(a) (b)

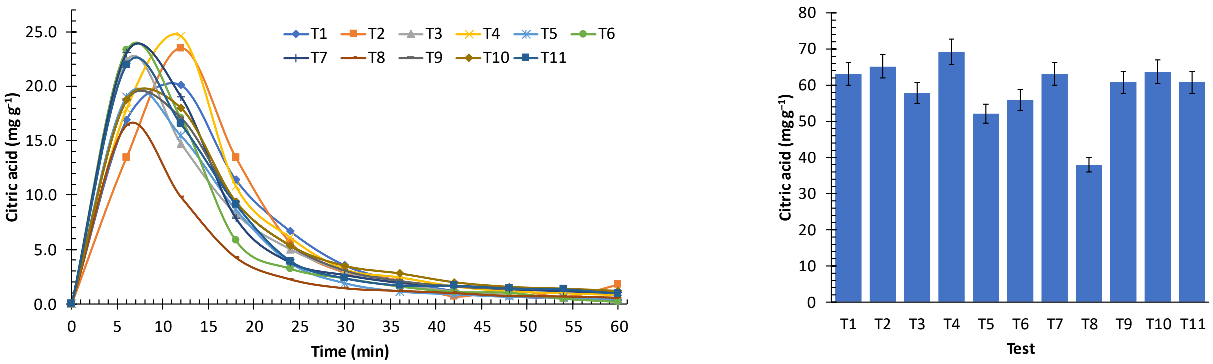


Fig. 9 Citric acid obtained from the cambuci peel through hydrothermal processing. (a) citric acid; (b) accumulated citric acid

UPLC-PDA-MS Analysis

The extracts were analyzed by UPLC-PDA-MS to tentatively identify potential candidates in each extract. Considering previous reports in the literature about the metabolites already identified in the cambuci fruit (Donado-Pestana et al., 2021), it was possible to point out the potential identifications related to the compounds presented in Table 4. The results indicate that ellagic acid and digalloyl-hexoside were consistently present in the cambuci peel throughout the entire extraction period of 60 min. Notably, quercetin pentoside was observed only within 6 min of the extraction process. The quantification of ellagic acid was carried out due to its presence at all extraction times. Figure 10 quantifies the acid in the cambuci peel extracts in the best extraction condition (160 °C). There, it is observed that at 18 min, a higher ellagic acid content is obtained, values higher than those reported for mango pulp residue extract (200 µg/mL) (Murugan et al., 2020) and Nigella sativa L. straw (25.54 µg/ mL) (Farouk et al., 2023).

Table 4 Tentative identification of metabolites present in extracts the extracts by UPLC-PDA-MS

Retention Time (min)	Name	*m/z*	Lamba (λ)	Fraction	Ref.
1.1	*p* - coumaric acid hexoside	325.17 [M-H]-	256, 291	1,4,5,7,9,10	(Barros et al., 2012)
1.2	Digalloyl-hexoside	483.14 [M-H]-	277	1,2,3,4,5,6,7,8,9,10	(García-Villalba et al., 2015)
3.3	Ellagic acid	301.06 [M-H]-	253, 356	1,2,3,4,5,6,7,8,9,10	(Aguilar-Zárate et al., 2017)
3.7	Quercetin pentoside	433.22 [M-H]-	266, 364	1	(Carocho et al., 2014)

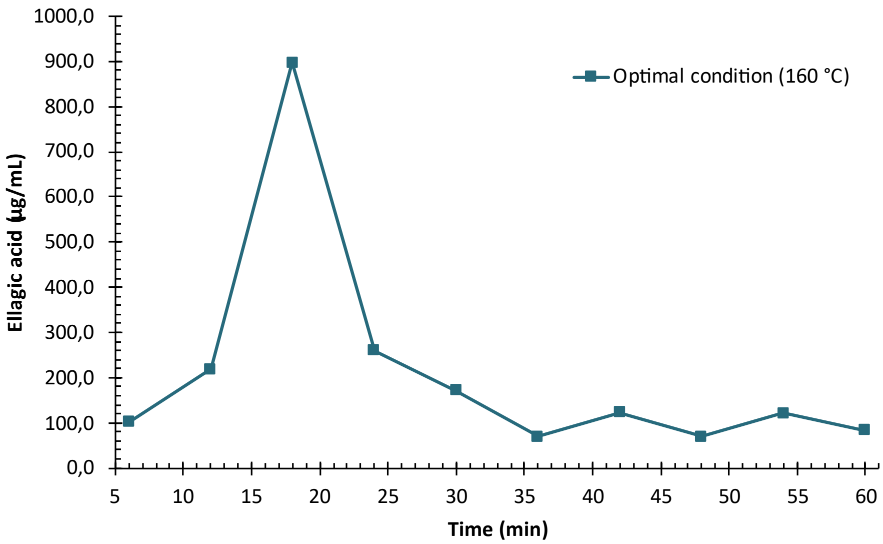


Fig. 10 Ellagic acid concentration for different extraction times in the optimal condition

In-line Process

The behavior of the in-line process is presented in Fig. 11. The monitoring of the process was carried out at three different wavelengths. The wavelengths were selected from the literature for compounds present in cambuci. For instance, ellagic acid was identified at 253 nm (Akter et al., 2021), galloyl-hexoside was identified at 273 nm (Aguilar-Zárate et al., 2017), and quercetin-*O*-(*O*-galloyl)-pentoside was identified at 356 nm (Saldanha et al., 2013). After 42 min, the 253 nm and 273 nm curves show near-zero absorbance, which indicates that the compounds at these wavelengths are in very low concentrations and are not easily identifiable. The 356 nm curve, starting at 36 min, begins to decrease rapidly, which indicates a decrease in the compounds identifiable at that wavelength. This information allows us to approximate when the extraction or hydrolysis process is completed in an in-line process, as the compounds of interest show a rapid decrease in concentration, sometimes reaching values that are too low for identification. For cambuci peel, this time is approximately 42 min. This method of in-line analysis enables a reduction in resource usage, minimizes the need for human intervention, and enables real-time monitoring of the entire process (Da Silva et al., 2023). Furthermore, since the online system is automated, data collection is facilitated, which allows adequate monitoring of the process in real time (Maciel-Silva et al., 2022).

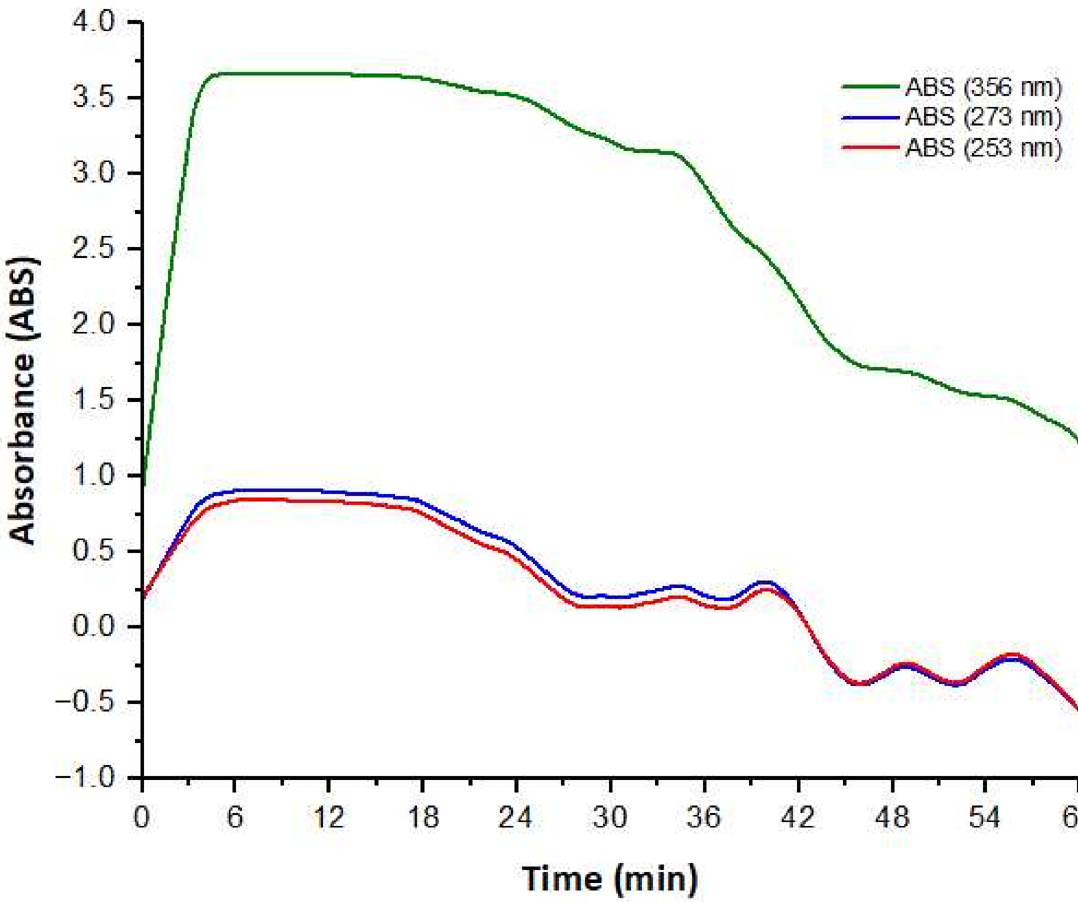


Fig. 11 In-line process monitoring at different wavelengths

Conclusions

The hydrothermal process on semi-continuous flow is a viable alternative for the valorization of cambuci peel because it is a safe and sustainable process for the recovery of bioproducts. In the extraction and hydrolysis of the studied by-product, temperature was the predominant parameter in the analyses conducted. The antioxidant activity by the FRAP method presented a maximum yield at 160 °C (501.91 µmol of TEAC g^{-1}), as did the total phenolic compounds (46.40 mg g^{-1} of dried cambuci peel). The maximum yield of total reducing sugars (132.54 mg g^{-1}) was observed at 143 °C. Likewise, high temperatures promoted the formation of inhibitors such as 5-HMF after 12 min of the process, which is related to Maillard reaction products and fructose and glucose degradation. On the other hand, the sugars identified in the cambuci peel were glucose, arabinose, and fructose, the latter being the sugar with the highest yields (102.44 mg g^{-1}). The compounds identified in the cambuci peel were *p* - coumaric acid hexoside, digalloyl-hexoside, ellagic acid, and quercetin pentoside. The hydrothermal process was carried out for 60 min. However, in-line analysis revealed that 42 min is sufficient to recover more than 80% of the bioactive compounds. This finding suggests potential savings in time and money, which may spark industry interest. The use of this technology is promising for the valorization of a by-product such as cambuci peel, which has not yet been widely studied and could contribute to the reduction of waste and would consequently favor the circular economy, a topic of great interest at present.

Finally, additional analyses, such as infrared (IR) spectroscopy for the identification of functional groups, mass spectrometry for identifying and quantifying compounds, or nuclear magnetic resonance (NMR), along with assessments for toxicity, microbiological activity, and stability of phenolic compounds, could be conducted. These analyses would provide detailed structural information about the compounds in this raw material and explore its potential applications across various industries.

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Author Contributions

J.A.J.M: Conceptualization, Methodology, Investigation, Validation, Writing - Original draft, Writing - review &; editing. L.F.M: Investigation, Validation, Writing - review &; editing. L.S.C: Validation, Writing - review &; editing. T.L.C.T.B: Validation, Writing - review &; editing. M.A.R: Supervision, Resources, Writing - review &; editing. T.F.C: Conceptualization, Methodology, Supervision, Resources, Project administration, Funding acquisition, Writing - review &; editing.

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Data Availability

No datasets were generated or analyzed during the current study. Declarations Competing Interests The authors declare no competing interests.

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CHAPTER 4 – General discussion

This work emphasizes the significance of valorizing by-products from cambuci (*Campomanesia phaea* Berg.), which contributes to the reduction of waste generated in the food industry. Moreover, a native fruit from the Brazilian Atlantic Forest is currently under exploration, with limited prior study. Nevertheless, it shows promising potential for different industries. Additionally, innovative and environmentally friendly technologies, such as extraction and hydrolysis with subcritical water, are being employed.

Firstly, the bibliometric analysis has revealed that cambuci (*Campomanesia phaea* Berg.) is a fruit with limited research attention. Nonetheless, the conducted studies depict this fruit as a promising source of vitamin C, showcasing its potential for the treatment of diseases like diabetes. The analysis focused on research studies conducted with cambuci between 2003 and 2022. The search was carried out using the Scopus© database with the keywords: cambuci OR campomanesia AND phaea OR "O. Berg Landrum" OR "(O. Berg) Landrum" OR cambucizeiro. Additionally, a filter for document type was applied, selecting "article" and "review." Over these years, a total of 175 publications (153 articles and 22 reviews) were identified, encompassing 609 author keywords.

Most publications are affiliated with Brazil, aligning with the organizations actively researching this native fruit of the Brazilian Atlantic Forest. The University of São Paulo emerges as the foremost institution globally in cambuci research. Furthermore, the analysis identified phenolic compounds, antioxidant capacity, flavonoids, and bioactive compounds as pivotal topics of significant importance for future research.

The aim of the bibliometric analysis is to provide guidance for future research on current issues and to continue contributing to the knowledge of native Brazilian fruits, which hold immense potential for utilization across various industries.

Secondly, the utilization of agro-industry waste holds significant potential for various industries, including pharmaceuticals, cosmetics, and food, owing to the diverse range of compounds that can be derived from these byproducts. Additionally, this approach facilitates a reduction in the environmental impact that occurs when such waste lacks proper treatment at the end of the chain. Promoting the adoption of environmentally friendly technologies, such as extraction with subcritical water, is crucial for minimizing operation times and reducing the use of toxic agents that can have a profound environmental impact.

Studying native Brazilian fruits raises awareness of their potential, which are often underutilized due to the unknown properties and benefits they could offer consumers. Similarly, researching their byproducts could contribute to the development of an industry that maximizes the utilization of the fruit while minimizing environmental impact.

The utilization of cambuci peel as a raw material, along with the extraction and hydrolysis using subcritical water in an automated in-line reactor, enabled us to determine that, at 160°C and after 42 minutes of the process, extracts exhibited high antioxidant capacity and contained elevated concentrations of citric acid, glucose, arabinose, and fructose. Furthermore, it was established that temperature plays a fundamental role in the extraction and hydrolysis processes of cambuci peel.

Due to the limited number of studies on cambuci, further research is necessary to explore this fruit comprehensively, as it yields promising results for diverse industries. This approach encourages the cultivation and industrialization of cambuci, contributing to Brazilian science by expanding knowledge about a native product that has not traditionally been explored.

CHAPTER 5 – General conclusions

This study confirmed that the cambuci peel remains largely unexplored, yet it holds significant potential to attract interest from various industries. The key conclusions from Chapters 2 and 3 are presented below.

- ✓ The bibliometric analysis conducted using data from the Scopus database spanning the years 2003 to 2022 facilitated the identification of the most pertinent research topics related to cambuci.
- ✓ The motor topics include phenolic compounds, antioxidant capacity, flavonoids, bioactive compounds, and osmotic dehydration. It is crucial to conduct research that develops and establishes connections between these topics to comprehensively understand the benefits of a native fruit like cambuci,
- ✓ The analysis highlights Brazil as a leading country in cambuci research, with most publications (69.41%) originating from Brazilian institutions.
- ✓ Cambuci exhibits potential for various industries, as several studies have determined its high content of bioactive components, which are beneficial for human health.
- ✓ The treatment with subcritical water emerges as an alternative for the valorization of agro-industrial waste, such as cambuci peel.
- ✓ Higher temperatures promoted an increase in the antioxidant capacity and total phenolic compounds in both the extracts and hydrolysates. On the other hand, the pH of the mobile phase water had minimal effects on the response of the described variables. Therefore, it is recommended to work with water at a neutral pH to avoid the use of any chemical agents that may alter it.
- ✓ The highest temperatures (143 and 160 °C) enhanced the response of total phenolic compounds, reaching maximum values of 12.44 mg g^{-1} and 9.50 mg GAE g^{-1} for the treatments conducted at 143 °C, and 12.76 mg GAE g^{-1} for the treatment at 160 °C.
- ✓ The increased hydrolysis temperature (160 °C) positively influenced the antioxidant capacity of the hydrolysate, reaching a maximum yield of 501.91 µmol of TEAC g^{-1} at the end of the 60-minute process.
- ✓ The sugars found in the highest proportion in cambuci peel include glucose, arabinose, and fructose. This suggests that cambuci peel could contribute to the circular economy, as the extracted sugars may find diverse applications in various industries.

- ✓ The utilization of an automated in-line reactor represents a novel technology that provides the capability to monitor the process in real-time. This allows for determining the optimal time for concluding the extraction or hydrolysis. In the case of cambuci peel hydrolysis, this study identified that 42 minutes are sufficient to complete the process. Nevertheless, it is recommended to continue further research to establish operation times for various raw materials.
- ✓ The results obtained in this study are applicable within the temperature range of 4 to 160 °C, as well as a pH range between 4 and 7, with a constant pressure of 15 MPa and a flow rate of 2 ml min^{-1}. It's important to note that these findings are specific to the mentioned conditions and cannot be extrapolated, but they could serve as a foundational basis for future research involving cambuci peel.
- ✓ Hydrolysis with subcritical water is an environmentally friendly technology applicable to a wide variety of raw materials. It is recommended to further explore by-products of native fruits, as this approach can generate added value for both the fruit and the waste from agro-industrial processing, which, in most cases, is disposed of inadequately.
- ✓ For future research, it is recommended to purify the extracts to achieve an accurate quantification of bioactive compounds, such as ellagic acid, as this could attract the attention of industries interested in this compound.

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Title	EXTRACTION AND HYDROLYSIS IN AN AUTOMATED IN-LINE REACTOR AS A TECHNOLOGY FOR THE VALUATION OF CAMBUCI (Campomanesia phaea Berg.) PEEL	**Institution Name**	UNIVERSIDADE ESTADUAL DE CAMPINAS
		Expected Presentation Date	2025-02-26
Instructor Name	TÂNIA FORSTER CARNEIRO		

ADDITIONAL DETAILS

The Requesting Person / Organization to Appear on the License	UNIVERSIDADE ESTADUAL DE CAMPINAS