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VANESSA COSME FERREIRA

SUSTAINABLE VALORIZATION OF PITAYA PEEL (*HYLOCEREUS UNDATUS*) VIA EXTRACTION AND HYDROLYSIS IN SUBCRITICAL WATER TO RECOVER HIGH-VALUE-ADDED PRODUCTS

VALORIZAÇÃO SUSTENTÁVEL DA CASCA DA PITAIA (*HYLOCEREUS UNDATUS*) VIA EXTRAÇÃO E HIDRÓLISE EM ÁGUA SUBCRÍTICA PARA RECUPERAR PRODUTOS DE ALTO VALOR AGREGADO

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Valorização sustentável da casca da pitaia (*hylocereus undatus*) via extração e hidrólise em água subcrítica para recuperar produtos de alto valor agregado

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

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RESUMO

A pitaia (*Hylocereus* spp.) é uma fruta com grande potencial para a produção de alimentos como geleia, iogurte e vinho, porém, até 40% da fruta se transforma em subprodutos durante o processamento agroindustrial. O reaproveitamento desses resíduos é uma alternativa para minimizar os efeitos negativos gerados no meio ambiente, além de promover ganhos econômicos, visto que muitos destes resíduos podem ser utilizados como matéria prima sustentável para a produção de compostos de alto valor agregado. O objetivo deste trabalho foi avaliar o uso de um processo hidrotérmico de alta pressão semicontínuo para a recuperação de produtos de valor agregado da casca da pitaia. O ensaio experimental usou o processo hidrotérmico a 15 Mpa de pressão, taxa de fluxo de água de 2 mL/min e temperaturas variando de 40 a 210 °C. Uma análise bibliométrica foi feita dos estudos nos últimos 22 anos para identificar as áreas e estudos realizados com pitaia. Os resultados da revisão e análise bibliométrica mostram os gaps do progresso da pesquisa, tendências e perspectivas na aplicação dos subprodutos da pitaia para a recuperação de produtos de valor agregado com processos sustentáveis. O principal campo de pesquisa identificado foi o de extração de compostos bioativos, sendo a betacianina o composto bioativo majoritariamente extraído da pitaia, seguido por antocianinas, flavonoides e flavonas. Os resultados também indicam que, a pitaia e seus subprodutos têm aplicação como corantes e existe uma tendência no uso de técnicas de extração sustentáveis (microondas, ultrassom, líquidos pressurizados e fluidos supercríticos) para a valorização da casca da pitaia. Os resultados do experimento com o processo hidrotérmico mostram que as temperaturas de extração entre 40 e 80 °C promoveram a recuperação de betacianina (1,52 mg/g), ácido málico (25,6 mg/g) e ácido cítrico (25,98 mg/g). Os principais compostos fenólicos obtidos foram ácido p-cumárico (17,95 µg/mL), ácido protocatecuico (11,43 µg/mL) e ácido piperônico (9,27 µg/mL). As temperaturas de hidrólise entre 150 e 210 °C produziram açúcares, enquanto temperaturas acima de 180 °C geraram produtos da reação de Maillard, aumentando os compostos fenólicos totais e a atividade antioxidante dos hidrolisados. Conclui-se que, o processo hidrotérmico de alta pressão semicontínuo demonstra ser uma abordagem promissora e sustentável para a recuperação de compostos valiosos da casca da pitaia, apoiando a economia circular na indústria agroalimentar.

Palavras-chave: *Líquidos pressurizados; Biocompostos; Economia circular; Biorrefinaria.*

ABSTRACT

The pitaya (Hylocereus spp.) is a fruit with great potential for the production of foods such as jam, yogurt, and wine. However, up to 40% of the fruit becomes by-products during agro-industrial processing. Reusing these residues is an alternative to minimize the negative environmental effects, as well as to promote economic gains, since many of these residues can be used as sustainable raw material for the production of high-value-added compounds. The objective of this study was to evaluate the use of a semi-continuous high-pressure hydrothermal process for the recovery of value-added products from pitaya peel. The experimental assay used the hydrothermal process at a pressure of 15 MPa, a water flow rate of 2 mL/min, and temperatures ranging from 40 to 210 °C. A bibliometric analysis was carried out of studies over the last 22 years to identify the areas and studies carried out on pitaya. The results of the review and bibliometric analysis show gaps in research progress, trends, and perspectives on the application of pitaya by-products for the recovery of value-added products using sustainable processes. The main research field identified was the extraction of bioactive compounds, with betacyanin being the major bioactive compound extracted from pitaya, followed by anthocyanins, flavonoids, and flavones. The results also indicate that pitaya and its by-products have applications as dyes, and there is a trend in the use of sustainable extraction techniques (microwaves, ultrasound, pressurized liquids, and supercritical fluids) for the valorization of pitaya peel. The results of the experiment with the hydrothermal process show that extraction temperatures between 40 and 80 °C promoted the recovery of betacyanin (1.52 mg/g), malic acid (25.6 mg/g), and citric acid (25.98 mg/g). The main phenolic compounds obtained were p-coumaric acid (17.95 µg/mL), protocatechuic acid (11.43 µg/mL), and piperonylic acid (9.27 µg/mL). Hydrolysis temperatures between 150 and 210 °C produced sugars, while temperatures above 180 °C generated Maillard reaction products, increasing the total phenolic compounds and antioxidant activity of the hydrolysates. It is concluded that the semi-continuous high-pressure hydrothermal process proves to be a promising and sustainable approach for the recovery of valuable compounds from pitaya peel, supporting the circular economy in the agro-food industry.

Keywords: *Pressurized liquids; Biocompounds; Circular economy; Biorefinery.*

SUMÁRIO

CAPÍTULO 1- Introdução geral, objetivos e estrutura da dissertação

1.1. Introdução Geral

Atualmente as indústrias agroalimentares são uma das maiores produtoras de resíduos sólidos, gerando elevadas quantidades anualmente (MAKRIS; BOSKOU; ANDRIKOPOULOS, 2007). Alguns desses resíduos são usados como adubo ou como ração para alimentação animal. Porém, esses materiais são compostos por substâncias orgânicas que podem ser nocivas ao meio ambiente por apresentarem elevada carga orgânica, o que pode gerar problemas ambientais. A maioria desses resíduos não possui um tratamento adequado para reutilização ou descarte (CASTRO *et al*., 2019; FAGNANI *et al*., 2019). Dentre os resíduos agroindustriais mais comuns, os provenientes das indústrias processadoras de frutas, são os mais encontrados, como cascas, sementes, folhas e outras partes dessas frutas (MARTINS et al., 2019).

Tratando-se da indústria de processamento da pitaia, a casca é um importante subproduto do processamento. A parte comestível da pitaia é a polpa que apresenta uma rica fonte de compostos ativos e pode ser consumida in natura, utilizada na fabricação de doces, bebidas e sorvetes (GEORGIN *et al*., 2022). A casca representa cerca de 40% do peso total do fruto, caracterizada como resíduo sólido sua gestão é um desafio. Na maioria das vezes a casca é descartada em aterros ou utilizada em compostagem e incineração, o que pode causar poluição ambiental devido a emissões de gases nocivos (GEORGIN *et al*., 2022). Ela também pode ser utilizada como fonte de agentes biotativos naturais, pois apresenta em sua composição flavonoides, ácidos fenólicos, betacianinas com potencial antioxidante, antibacteriano e anti-inflamatório(XIN *et al*., 2022).

A pitaia da polpa branca (*Hylocereus undatus*), pertence ao grupo das espécies exóticas de cactos. É cultivada nos países da América Central, sendo o México seu principal produtor. Com o passar dos anos o Brasil vem se destacando como produtor de pitaia devido ao seu crescente cultivo e consumo (GEORGIN *et al*., 2022). No ano de 2017 a produção de pitaia no Brasil segundo o Instituto Brasileiro de Geografia e Estatística (IBGE) foi de 1459 toneladas, sendo São Paulo e Santa Catarina responsáveis por mais de 64% de toda produção (IBGE, 2017). Já na safra de 2021, segundo o a Empresa de Pesquisa Agropecuária e Extensão Rural de Santa Catarina (EPAGRI), o estado de Santa Catarina produziu em torno de 1000 toneladas, demonstrando assim um crescimento de mais de 60% em relação as safras anteriores (EPAGRI, 2021).

O reaproveitamento de resíduos agroindustriais deve ser realizado de forma ambientalmente correta para minimizar problemas de emissão de gases de efeito estufa, evitar fenômenos como a eutrofização nos rios, perda da fauna e flora nativa, o esgotamento de recursos não-renováveis e o acúmulo de lixo no meio ambiente (KAHILUOTO *et al*., 2011). Além disso, a destinação incorreta desses coprodutos pode ocasionar perdas econômicas para a indústria, visto que esses resíduos são uma rica fonte de compostos de alto valor agregado. Esses compostos podem ser aproveitados em diferentes setores industriais (farmacêutico, químico e alimentício) para a fabricação de produtos mais naturais, como suplementos alimentares ricos em antioxidantes e outros compostos bioativos (FERRARI *et al*., 2019; MAKRIS; BOSKOU; ANDRIKOPOULOS, 2007; ZÚÑIGA-MURO *et al*., 2020).

Levando em consideração o contexto de economia circular, o aproveitamento dos resisduos orgânicos produzidos pela indústria alimentícia pode ser realizado através de procedimentos de extração com objetivo de obter produtos de alto valor agregado(UMMAT *et al*., 2021). Os processos de extração e os solventes utilizados dependem das características da matéria prima utilizada e dos produtos de interesse, uma vez que a tecnologia utilizada afeta as características qualitativas e quantitativas dos compostos (UMMAT *et al*., 2021). Atualmente, o interesse por técnicas de extrações ecológicas, seguras, acessíveis e eficientes tem aumentado consideravelmente. Com isso, surgiu o termo “extração verde”. Esse conceito se baseia no desenvolvimento de processos de extração que permitem a redução do consumo de energia e a utilização de solventes alternativos e produtos renováveis, promovendo assim, a obtenção de um extrato seguro e de alta qualidade (GOMEZ *et al*., 2020) . Várias tecnologias estão sendo desenvolvidas e empregadas nesse conceito, como por exemplo, a extração assistida por ultrassom, extração assistida por microondas, extração assistida por enzima, extração com fluido supercrítico e extração via liquido pressurizado (KADAM *et al*., 2015).

Dentre as tecnologias verdes estudadas atualmente tem-se o processo hidrotérmico, que vem sendo considerado uma alternativa ecológica, econômica e simples. Uma das maiores vantagens do tratamento é a utilização da água como principal solvente de extração, evitando assim o uso de solventes tóxicos (PLAZA; TURNER, 2015). No tratamento hidrotérmico, são utilizadas temperaturas acima do ponto de ebulição à pressão atmosférica (100 °C a 1 atm) e abaixo do ponto crítico (374 °C a 218,11 atm), possibilitando a alteração da polaridade da água e permitindo que ela dissolva compostos de média e alta polaridade (TEO *et al*., 2010). A aplicação do tratamento hidrotérmico tem sido utilizada para a recuperação de diversos compostos extraídos de substratos vegetais e tem apresentado resultados satisfatórios. Já foi comprovado que este tratamento promove a ruptura estrutural nas células vegetais, facilitando a liberação de compostos intracelulares (DAS; ARORA, 2023). Contudo, as condições operacionais devem ser bem definidas, pois dependendo das faixas de pressão e temperatura podem ser obtidos diferentes produtos. Sendo a otimização destas condições operacionais essencial para a sua aplicabilidade. Por fim, os tratamentos hidrotérmicos parecem ser uma alternativa conveniente e eficaz para a obtenção de compostos de alto valor.

Para avaliar a eficácia e inovação desses tratamentos, a análise bibliométrica é uma ferramenta valiosa. Ela permite quantificar os trabalhos publicados sobre o tema, identificando o grau de inovação, as áreas de interesse, os assuntos abordados, e os principais países envolvidos (DOS SANTOS *et al*., 2023). A bibliometria é uma ciência que integra matemática, estatística e bibliografia, explorando a estrutura e características da ciência e tecnologia. Estuda a distribuição, as relações quantitativas e os padrões de mudança na literatura (FENG *et al*., 2023).

Neste estudo, o objetivo foi avaliar o uso de um processo hidrotérmico de alta pressão semicontínuo para a recuperação de produtos de valor agregado da casca da pitaia. Através dessa abordagem, busca-se otimizar as condições operacionais e explorar todo o potencial dos tratamentos hidrotérmicos.

1.2. Objetivos

1.2.1. Objetivo Geral

O objetivo geral deste trabalho foi avaliar o uso de um processo hidrotérmico de alta pressão semicontínuo para a recuperação de produtos de valor agregado da casca da pitaia. O reator subcrítico foi operado em modo semi-contínuo para obtenção de um extrato/hidrolisado com alta quantidade desses compostos.

1.2.2. Objetivos específicos

- Realizar análise bibliométrica dos estudos dos últimos 22 anos para verificar as áreas de estudos pouco explorados utilizando a pitaia e as possibilidades para a sua utilização;

- Selecionar os parâmetros operacionais de extração e hidrólise do reator com água subcrítica na pressão 15 Mpa, fluxo 2 mL/min e temperaturas de 40, 60 e 80 °C (extração) e 150, 180 e 210 °C (hidrólise) a partir de cascas de pitaias;

- Analisar os extratos e os hidrolizados quanto a concentração de compostos bioativos, açúcares, inibidores e ácidos orgânicos produzidos na etapa anterior.

1.3. Estrutura da Dissertação

Esta dissertação se encontra dividida em capítulos. Os capítulos apresentam os resultados experimentais correspondem os artigos que foram publicados em revistas científicas da Área de Engenharia de Alimentos.

O **Capítulo 1** é composto pela introdução, que descreve o tema central da dissertação, fazendo uma exposição, de forma sucinta dos pontos mais relevantes, os objetivos e finalidade dessa dissertação.

O **Capítulo 2** trata de um artigo de revisão com título "An updated review on recent applications and future perspectives to the valorization of pitaya (*Hylocereus* spp.) by-product". O trabalho apresenta uma revisão bibliométrica sobre o andamento da pesquisa, atualizações e tendências futuras de recuperação de produtos com maior valor agregado das cascas de pitaia no conceito de biorrefinaria.

O **Capítulo 3** trata de um artigo com título "Sustainable valorization of pitaya (Hylocereus spp.) peel in a semi-continuous high-pressure hydrothermal process to recover value-added products". O trabalho tem como tema central a valorização de subprodutos procedentes do processamento de pitaia devido as preocupações com os impactos ambientais causados pela deposição incorreta. Este estudo apresenta ensaios de extração e hidrólise em água subcrítica, em reatores semi-contínuos, em diferentes condições operacionais de temperatura, pressão e fluxo a partir das cascas de pitaia. Os extratos e os hidrolisados foram caracterizados quanto a concentração de compostos bioativos, açúcares, inibidores, ácidos orgânicos produzidos a partir do resíduo agroindustrial de pitaia.

O **Capítulo 4** apresenta uma discussão dos principais resultados obtidos neste trabalho.

O C**apítulo 5** apresenta as conclusões gerais e sugestões para trabalhos futuros.

O C**apítulo 6** apresenta as referencias bibliográficas utilizadas nessa dissertação.

CAPÍTULO 2 - An updated review on recent applications and future perspectives to the valorization of pitaya (Hylocereus spp.) by-product.

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An updated review on recent applications and future perspectives to the valorization of pitaya (*Hylocereus* spp.) by-product

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Abstract

Pitaya (*Hylocereus* spp.) is a fruit with high potential to produce food products, such as jam, jelly, yogurt, and wine. However, the by-products generated during agro-industrial processing can represent up to 40% of the fruit. This comprehensive review and bibliometric analysis elucidated the research progress, updates, trends, and perspectives in the application of pitaya by-products to the recovery of value-added products with sustainable processes. The main research field is associated with the extraction of bioactive compounds. Betacyanin is the major phytochemical extracted from pitaya, followed by anthocyanins, flavonoids, and flavones. Moreover, pitaya and its by-products have been incorporated as a natural colorant to improve the bioavailability of food products. There is a trend in the use of sustainable extraction techniques (microwave, ultrasound, and pressurized liquids, and supercritical fluids) to the valorization of pitaya peel. The extracts and powder of pitaya can be incorporated in food packaging to reduce the oxidative reactions during storage, monitoring the freshness by changes in pH, and increasing the shelf life of perishable food products. In conclusion, the findings elucidated in this review supports the application of pitaya by-product in the sustainable production of value-added products.

Keywords: *Dragon fruit; Bioactive compounds; Food products; Natural products; Extraction; Sustainable chemistry.*

1. Introduction

Nowadays, there is a trend in the production of eco-friendly value-added materials and bioenergy from agri-food biomass by-products [1,2]. The recently increase in the cost of electricity, efforts to reduce greenhouse gas emissions, climate change mitigation, and large food lost are some reasons that encourage the discover of novel waste management technologies [3]. In addition, the advantages of applying agri-food by-products to produce value-added bio-products can stimulate the bioeconomy based on biomass [4,5]. Environmentally friendly technologies to the management of organic waste is a challenge for different industrial sectors, especially for the agri-food industry, that generates high amount of wastewater, organic, and lignocellulosic waste [6,7].

The agri-food sector must meet the Sustainable Development Goals (SDG) established by the 2030 Agenda for Sustainable Development. Among the 17 goals, the SDG 6 (clean water and sanitation), 7 (accessible and clean energy), and 13 (climate action) can be achieved by the agri-food industry with the application of waste-to-energy technologies [8]. For instance, the by-products from the food industry (e.g., straw, bagasse, seeds, and husks) could generate biomethane, electricity, and thermal energy through anaerobic digestion, avoiding environmental pollution [9,10]. Innovative and sustainable technologies focused on waste management and bioenergy recovery are essential for sustainable development based on the bioeconomy [11], which supports the environmental conservation and mitigate greenhouse gases [12,13].

The application of sustainable waste management technologies can contribute to the establishment of a circular economy by closing the use of biomass [14–16]. The circular economy is a concept that must be implemented to change the linear economy based on fossil resources and without environmentally correct disposal of the wastes generated [17,18]. The circular economy model is based on the product life cycle, especially in the concept of "reduce – reuse – recycle" [19]. In the case of the agri-food industry, the sustainable waste management and bio-products recovery advocates the circular economy [20]. These technologies and processes are directly associated with the biorefinery concept, which has great importance for the improvement of process intensification and reduction of environmental impacts [21]. The biorefinery concept can be defined as a sustainable (bio)process for transforming lignocellulosic biomass into a range of marketable products and energy [19]. From the biorefinery concept, it is possible to separate biomass resources into their building blocks, such as lipids, proteins, and carbohydrates, which can converted into value-added products in downstream processes [22]. The processing of agri-food by-products in biorefineries can reduce the waste generated, increase the market diversification of bio-based products, acquiring a synergistic effect of several technologies and the possible occurrence of energy self-sufficiency [23].

Notwithstanding, bibliometrics consists of a method of evaluating scientific productions that uses published documents to evaluate the most important authors, institutions, keywords, and citations related to a specific research field [24,25]. Through bibliometric analysis, it is possible to highlight the research gaps, hot topics, perspectives, and futures directions in a field of science [26]. In the case of the agri-food industry, the use of bibliometric analysis can contribute to the sustainable development and towards a waste-biorefinery implementation, advocating a circular bioeconomy. Recently, bibliometric tools was applied to evaluate the bioprospecting and potential of cactus mucilage [27], trends in mulberry and silkworm research [28], probiotics in citrus fruits products [29], extraction of bioactive compounds [30], scientific research on mango by-products [31], strawberry [32], and uvaia [33]. However, there is a lack in the literature regarding a bibliometric analysis of scientific research on pitaya fruits and its by-products.

Based on the mentioned above, this study addressed an updated review and a bibliometric analysis of the scientific research on pitaya and its by-products. The focus of this review is enclosed in the analysis of research progress, updates, and future trends in the recovery of bioproducts from pitaya by-product. This updated review contributes to the description of the available technologies for the valorization of pitaya peel. Therefore, this study effectively proposes the future research and is the basis for the circular economy transition of the pitaya processing industry.

2. Methodology for literature search and bibliometric analysis

The bibliometric analysis was conducted based on the methodology established and reported in previous studies [33]. The bibliometric study used scientific data from the core collection of Science Citation Index Expanded (SCI-E) – Clarivate Analytics' ISI – Web of Science© (WoS). The type of search applied was "advanced search" applying the following logic operation: "*Hylocereus polyrhizus*" OR "*Hylocereus undatus*" OR "*Hylocereus costaricensis*" OR "*Hylocereus megalanthus*" OR "pitaya" OR "pitaya" OR "pitahaya" OR "dragon fruit". The search query was used to obtain reliable and accurate details based on selected words included in the title, abstract, author's keywords, and not at keywords-plus® of each document on the topic. The types of documents were filtered to "article" and "review". The systematic search was conducted in the timespan from 2000 to 2021, aiming to observe the publication's evolution over the years and the most frequent research areas. In total, 770 documents (753 articles and 17 reviews) were selected for the bibliometric analysis.

The data from the selected documents were exported and analyzed using bibliometric software. The "Bibliometrix" package (R language) was used to obtain a three-field plot and the top authors' production over the timing diagram [34]. After that, the dataset was investigated on VOSviewer© software to construct the authors, sources and countries' networks, and the keyword clusters [35]. All the data used in the bibliometric analysis were obtained from the 770 documents exported in the WoS. The most cited documents were evaluated to understand the importance and repercussions of the most impacting works. Finally, the research trends, gaps, hot topics, and perspectives were evaluated to determine the future research on pitaya.

3. Overview of the pitaya research over the last 21 years

3.1. Publication evolution and research areas

Figure 1 presents the number of publications on the research over the last 21 years. In the year 2000, two articles were published. One of them was associated with the botanic and genomic aspects of the plant [36], while the other developed a pollen storage procedure [37]. *Agriculture* was the main research area studied until 2010. After 2010 there is a significant increase in the number of publications, when the number of documents reached 21. From 2011 (29 documents) to 2021 (159 documents), there is an exponential increase in the number of publications. In 2021, the documents recorded in the WoS database corresponded to 20% of all documents published in the research field. The main research area has been oscillating among *Agriculture* and *Food Science Technology*, passing through *Chemistry* and *Plant Sciences*. This increase in the number of publications demonstrate that pitaya represents an interest feedstock for scientific studies, which may be associated with a worldwide concern about human healthy, nutritional food, and its benefits [38–40].

Figure 1. Evolution of the number of publications over the years 2000–2021.

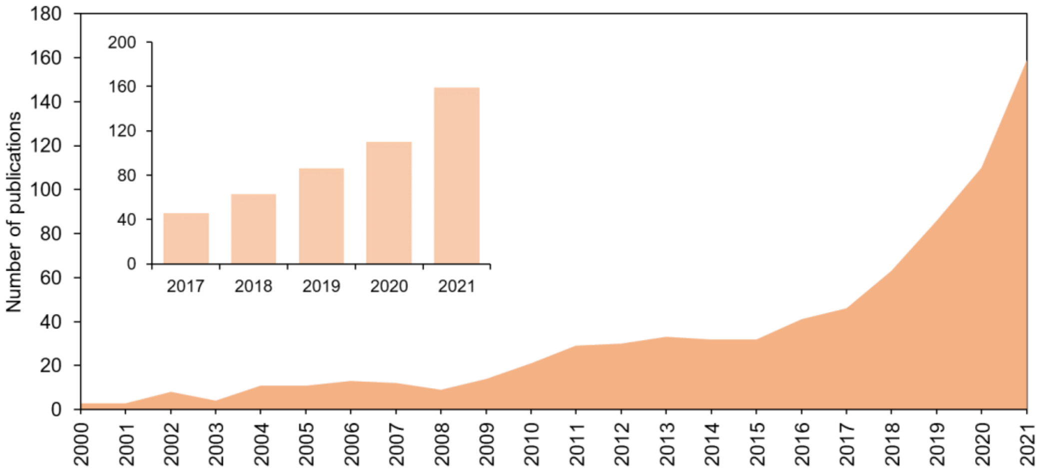


Table 1 presents the ranking of the 10 top research areas, affiliations, countries, journals, and authors based on the number of publications over the last 21 years in the field of pitaya research. Considering the total number of publications related to each research area, *Agriculture* accounts for 26.88%, followed by *Food Science Technology* (26.36%), and *Chemistry* (17.14%). The diversity of research areas increased proportionally to the number of publications on pitaya, which shows a growing interest in multiple applications of this valuable fruit, allowing the development of new research and marketable products. In *Agriculture* research field, the article indicated as the most relevant by the WoS database, studied different rates of nitrogen fertilization in species of pitaya, aiming to improve yield, fruit quality, and nutrient content [41]. The dose of 190 g nitrogen per plant was the best condition for *Hylocereus undatus* [41]. For *Food Science Technology*, the most relevant article investigated the physical stability and antioxidant properties of spray-dried pitaya, indicating a decrease in antioxidant activity when compared with the fresh sample [42]. Finally, for *Chemistry*, the most relevant study evaluated the oil extracted from the seeds of *Hylocereus undatus* and *Hylocereus polyrhizus*, revealing that pitaya seed oil has a good content of functional lipids and bioactive compounds [43] .

Table 1. Ranking of the 10 top publication areas, affiliations, countries, journals, and authors based on the number of publications over the last 21 years in the field of pitaya research.

Ranking	Parameters	Number	%[1]
Research areas			
1st	Agriculture	207	26.88
2nd	Food Science Technology	203	26.36
3rd	Chemistry	132	17.14
4th	Plant Sciences	79	10.26
5th	Engineering	69	8.96
6th	Biochemistry Molecular Biology	49	6.36
7th	Environmental Sciences Ecology	38	4.93
8th	Science Technology other Topics	35	4.54
9th	Materials Science	33	4.28
10th	Biotechnology Applied Microbiology	28	3.63
Affiliations (Country)			
1st	Universiti Putra Malaysia	64	8.31
2nd	Ben Gurion University	37	4.80
3rd	Chinese Academy of Sciences	20	2.59
4th	Instituto Politecnico Nacional Mexico	19	2.46
5th	Universiti Sains Malaysia	19	2.46
6th	Federal University of Lavras (Brazil)	18	2.33
7th	University Hohenheim	18	2.33
8th	South China Agricultural University	17	2.20
9th	Univ Autonoma Chapingo	14	1.81
10th	Hainan University	13	1.68
Countries			
1st	China	168	21.81
2nd	Malaysia	128	16.62
3rd	Mexico	80	10.39
4th	Brazil	72	9.35
5th	United States of America	45	5.84
6th	Israel	43	5.58
7th	Thailand	34	4.41
8th	Vietnam	34	4.41
9th	Indonesia	31	4.02
10th	Taiwan	29	3.76
Journals			
1st	Scientia Horticulturae	20	2.59
2nd	Food Chemistry	17	2.20
3rd	Postharvest Biology and Technology	15	1.94
4th	LWT – Food Science and Technology	14	1.81
5th	International Food Research Journal	13	1.68
6th	International Journal of Food Science and Technology	12	1.55
7th	Molecules	11	1.42
8th	*Revista Brasileira de Fruticultura**	11	1.42
9th	Journal of Agricultural and Food Chemistry	9	1.16

10th	Journal of Food Processing and Preservation	9	1.16
Authors			
1st	Mizrahi Y	28	3.63
2nd	Carle R	16	2.07
3rd	Tel-zur N	15	1.94
4th	Stintzing FC	13	1.68
5th	Ramos JD	11	1.42
6th	Zhang ZK	11	1.42
7th	Ali A	10	1.29
8th	Hua QZ	9	1.16
9th	Qin YH	9	1.16
10th	Chen CB	8	1.03

[1] Percentage of 770 documents (automatically calculated in WoS). Search conducted on March 29th, 2022; * In Portuguese.

3.2. Keywords analysis

The bibliometric analysis investigated the most important keywords in the field. **Table 2** describes the 20 most used keywords in the bibliometric study (rank based on the occurrences). The first four keywords are related to the popular (pitaya and dragon fruit) and scientific names (*Hylocereus undatus* and *Hylocereus polyrhizus*) of the fruit. The other keywords are related to the compounds and name variations presented by the fruit. Keywords associated with extraction of bioactive compounds and antioxidant activity are also reported with high occurrences and link strength.

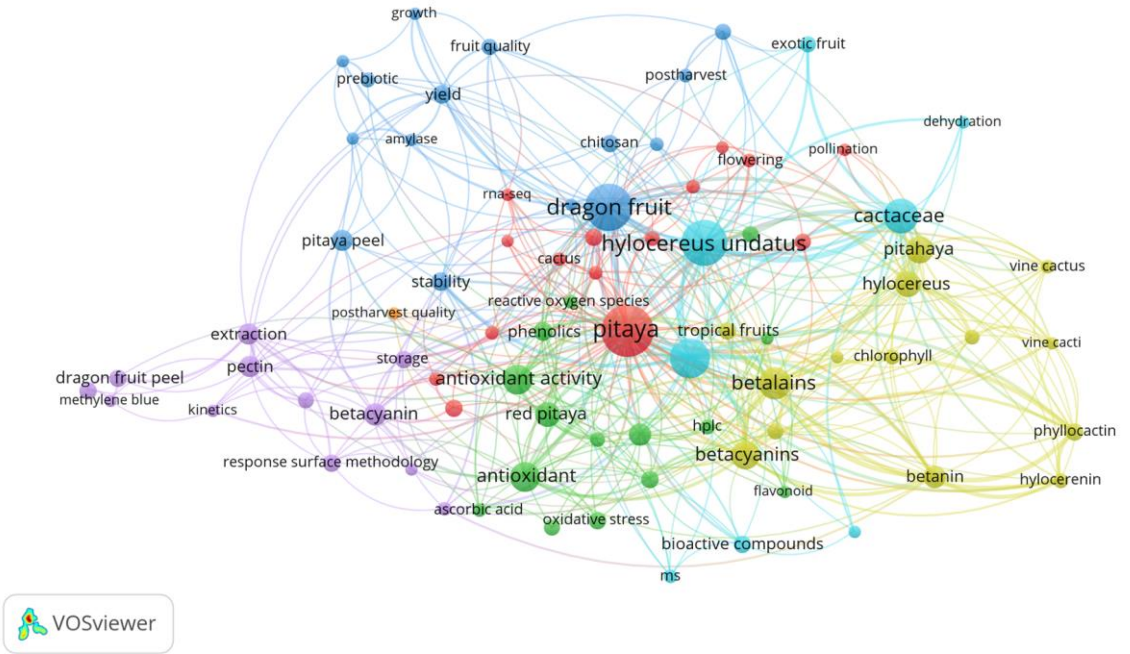
Table 2. The top 20 keywords in the field of pitaya research (rank based on the occurrences).

Ranking	Keyword	Occurrences	Total link strength
1	Pitaya	83	112
2	Dragon fruit	67	92
3	*Hylocereus undatus*	66	74
4	*Hylocereus polyrhizus*	49	102
5	Cactaceae	35	72
6	Betalains	31	84
7	Antioxidant	26	47
8	Antioxidant activity	26	38
9	Betacyanins	23	65
10	Pitahaya	21	36
11	*Hylocereus*	20	42
12	Red pitaya	17	20
13	Betacyanin	15	24
14	Phenolic compounds	15	13
15	Betanin	14	52
16	Pectin	13	20
17	Extraction	12	23
18	Pitaya peel	12	11
19	Phenolics	10	23
20	Stability	10	21

The keywords most used were grouped into clusters (**Figure 2)**. The network map consists of 7 clusters (**Table 3**) where the displayed keywords are directly related its potency and importance. The spacing between the clusters represents the link between the words. According to **Figure 2a**, the keyword *pitaya* occupies a central place when the subject is the studied fruit, and the importance of this keyword can be observed through the high number of links between this word and other terms. The keyword *pitaya* was presented in the red cluster (group 1) and is associated with the green (group 2), dark blue (group 3) and light blue (group 6) clusters. This association demonstrates that the fruit is mostly studied for the physicochemical characterization and extraction of bioactive compounds. This purpose can be observed through the presence of keywords such as *betacyanins*, *antioxidant activity*, and *phenolic compounds*. The presence of the keyword *bark* in some clusters demonstrates that the by-product from pitaya has been studied for purposes better than open-air disposal. Finally, keywords such as *ethylene*, *quality*, and *storage*, suggest the study of the characteristics of the fruit during its processing and shelf life. The VOSviewer software makes it possible to visualize the use of keywords between 2010 and 2018 (**Figure 2b**). Initially, research on pitaya was related to its origin and classification and the quantification of specific betacyanins. Between 2012 and 2014, the fruit became better known, and terms such as *Hylocereus undatus* and *tropical fruit* started to be used in the research field. In 2016, there is a considerably increase in the interest of bioactive compounds present in the pulp and peel of pitaya, and this fact can be observed through the increased use of terms such as *peel*, *amylases*, *antioxidants*, and *pectin*. Finally, in 2018, the extraction of bioactive compounds gained prominence, and the use of terms such as *phenolic compounds*, *betacyanins*, *prebiotics* and *flavonoids* emerged.

Figure 2. Clustering of the most frequent authors' keywords. (a) Term map based on different clusters; (b) Term average year map.

(a)



(b)

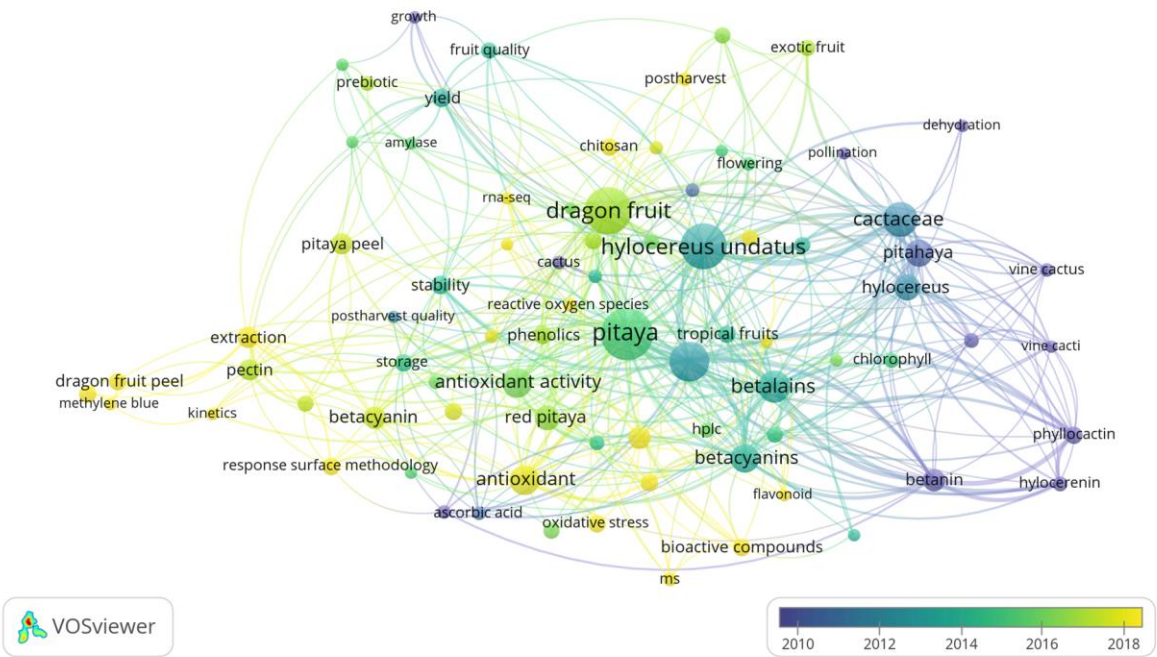


Table 3. Clusters of the main keywords obtained on VOSviewer software.

Cluster	Number of items	Keywords on VOSviewer Network
1	15	Betalain, cactus, columnar cacti, flowering, phenology, pitaya, pitaya fruit, pollination, red dragon fruit, rna-seq, *Stenocereus proinosus*, *Stenocereus stellatus*, temperature, transcriptome, and white dragon fruit
2	15	Antioxidant, antioxidant activity, antioxidant capacity, ascorbic acid, dragon fruits, fermentation, flavonoid, HPLC, *Neoscytalidium dimidiatum*, oxidative stress, phenolic compounds, phenolics, polyphenols, reactive oxygen species, and red pitaya.
3	15	Amylase, chitosan, dragon fruit, ethylene, fruit quality, growth, oligosaccharides, pitaya peel, postharvest, prebiotic, purification, quality, *Selenicereus megalanthus*, stability, and yield.
4	14	Betacyanins, betalains, betanin, betaxanthins, chlorophyll, gas chromatography, *Hylocerenin*, *Hylocereus*, phyllocactin, pitahaya, *Selenicereus*, tropical fruits, vine cacti, and vine cactus.
5	12	Adsorption, betacyanin, dragon fruit peel, extraction, kinetics, methylene blue, optimization, pectin, purple pitaya, response surface methodology, storage, and vitamin C.
6	8	Bioactive compounds, cactaceae, dehydration, exotic fruit, exotic fruits, *Hylocereus polyrhizus*, *Hylocereus undatus*, and MS.
7	1	Postharvest quality

In addition, **Figure 3a** shows the relationship between the centrality and density of the most frequent keywords in the pitaya research. Each set of keywords corresponds to a group of keywords described in **Table 4**. It was possible to observe a diagram that allows the analysis of the most prominent and used keywords when the theme is pitaya. The size of the sphere in **Figure 3a** is related to the number of uses of the keyword group. The diagram is divided into 4 quadrants. In the first quadrant (motor themes), well-developed themes with great importance were presented, such as *antioxidant activity.* In the second quadrant (niche themes), keywords with high density and low centrality were presented, which means that these keywords are specific and important for the research area, such as *betalains*. Emerging or declining themes were presented in the third quadrant, which includes terms that have low density and centrality (i.e., themes that are no longer being studied or that have been addressed recently and are not yet very present in the literature), such as *pectin* and *pitaya peel*. Finally, the basic themes in the fourth quadrant represent the highly relevant themes. The keywords *dragon fruit* and *Hylocereus undatus* are present between two themes and may change classification over the years, demonstrating that studies on pitaya have been conducting to advance the knowledge.

Figure 3. Bibliometric analysis of pitaya research. (a) Thematic map of the most frequent keywords; (b) Most important authors; (c) Network analysis of the most expressive journals; (d) Network analysis of the most expressive countries; (e) Map representation of the number of publications per country; (f) Three fields plot correlating the top authors, keywords, and countries.

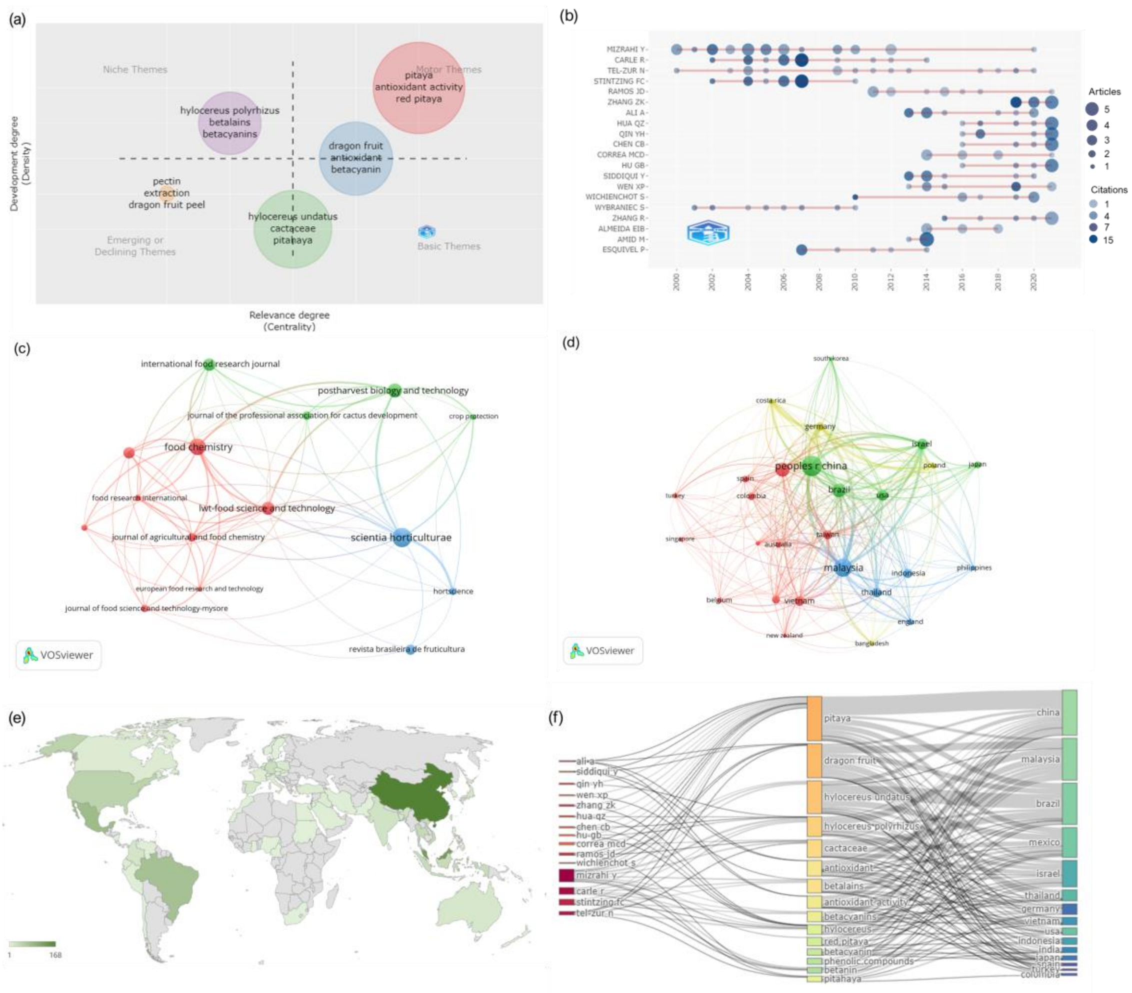


Table 4. Clusters of the main keywords obtained on Bibliometrix software.

Cluster	Main keyword	Keywords on Bibliometrix Network
1	Pitaya	Pitaya, antioxidant activity, red pitaya, phenolic compounds, phenolics, bioactive compounds, pitaya fruit, tropical fruits, chitosan, and fermentation.
2	*Hylocereus polyrhizus*	*Hylocereus polyrhizus*, betalains, betacyanins, betanin, stability, phyllocactin, betaxanthins, chlorophyll, hylocerenin, and anthocyanin
3	Dragon fruit	Dragon fruit, antioxidant, betacyanin, pitaya peel, yield, red dragon fruit, oxidate storage, fruit quality, and genetic diversity.
4	*Hylocereus undatus*	*Hylocereus undatus*, cactaceae, pitahaya, *Hylocereus*, exotic fruit, *Selenicereus megalanthus*, temperature, Alternaria alternata, *Selenicereus*, and cactus.
5	Pectin	Pectin, extraction, dragon fruit peel, adsorption, response surface methodology, optimization, dragon fruit peels, and methylene blue.

3.3. Most cited articles in the research field

Table 5 shows the top 10 most cited articles in the field of pitaya research. The analysis of the most cited documents in a research field is an interesting analysis to highlight the most significant topics related to the theme. Most of the publications presented in this ranking are experimental articles, with only one review. It was possible to identify a trend on the study of betalains and betacyanins from pitaya for food applications. The first publication consists in an experimental research conducted with red pitaya, evaluating its total phenolic content, antioxidant, and antiproliferative activity on melanoma cells [44]. In addition, the study determined that red pitaya is an important source of antioxidants. The antiproliferative study on melanoma cells indicated that the peel is stronger than the pulp as a growth inhibitor. Additionally, the pulp and peel were rich in antioxidants polyphenols [44].

Table 5. Top 10 most cited articles in the field of pitaya research.

Ranking	Title	Journal	Publication year	Total citations	Average citation per year	Reference
1st	Antioxidant and antiproliferative activities of red pitaya	Food Chemistry	2006	287	16.88	[44]
2nd	Total antioxidant activity and fiber content of select Florida-grown tropical fruits	Journal of Agricultural and Food Chemistry	2006	213	12.53	[45]
3rd	Robust pitaya-structured pyrite as high energy density cathode for high-rate lithium batteries	ACS Nano	2017	188	31.33	[46]
4th	Betacyanins in fruits from red-purple pitaya, Hylocereus polyrhizus (Weber) Britton & Rose	Food Chemistry	2002	158	7.52	[48]
5th	Betalains: Natural plant pigments with potential application in functional foods	LWT - Food Science and Technology	2015	154	19.25	[40]
6th	Oligosaccharides of pitaya (dragon fruit) flesh and their prebiotic properties	Food Chemistry	2010	142	10.92	[50]
7th	Fruit peels as efficient renewable adsorbents for removal of dissolved heavy metals and dyes from water	ACS Sustainable Chemistry & Engineering	2015	134	16.75	[52]
8th	Pitaya-like Sn@C nanocomposites as high-rate and long-life anode for lithium-ion batteries	Nanoscale	2014	122	13.56	[47]
9th	Nutraceutical potential and antioxidant benefits of red pitaya (Hylocereus polyrhizus) extracts	Journal of Functional Foods	2012	112	10.18	[51]
10th	Fruit flesh betacyanin pigments in Hylocereus cacti	Journal of Agricultural and Food Chemistry	2002	112	5.33	[49]

The second most cited article evaluated the antioxidant activity, total soluble phenolics, total ascorbic acid, total dietary fiber, and pectin contents in several fruits, including red and white pitaya [45]. The study indicates that red pitaya presents more than double the values of total soluble phenolics, total ascorbic acid, oxygen radical absorbance capacity and antioxidant activity when compared to white pitaya [45]. In general, red pitaya presented higher antioxidant activity than the other tropical fruit varieties. However, the consumption of these fruits is a natural source of antioxidants and dietary fiber, which protect the cells from damage while helping improve digestion and maintain blood sugar levels [45].

The articles found in the third and eighth positions studied lithium batteries with their structure based on pitaya fruits [46,47]. On the fourth and tenth positions, there are two experimental articles regarding betacyanin pigments on red–purple pitaya [48] and *Hylocereus* species [49], respectively. The first study indicated the same betalain composition of mesocarp and pulp, indicating similar enzymes in both tissues. Moreover, both articles mentioned *H. polyrhizus* as the species containing the highest relative concentration of the pigment hylocerenin, which is probably responsible for the fluorescent color of the fruit [46,47]. The study on the fifth position of the ranking is also regarding betalains, which are plant-derived natural pigments, discussing their potential for food application, their pharmacological properties and the sources from which betalains can be provided [40].

The sixth and ninth positions in the ranking of the most cited studies are related to nutritional aspects of pitaya, especially due to the presence of oligosaccharides with prebiotic properties [50], and the nutraceutical potential and antioxidant benefits of the fruit [51]. Both white and red pitayas were investigated as a potential source of oligosaccharides for prebiotic production, evaluating the optimal extraction method, sugar content, and prebiotic properties [50]. It was found that pitayas are mostly composed of glucose, fructose, and some oligosaccharides, and the fruit is a potential source of prebiotics, with applications in functional food and nutraceutical products [50]. Another study quantified polyphenols from pulp and peels of red pitaya. Betacyanin elements exhibited reducing and radical-scavenging capacities, while the polyphenolic fractions showed intense antimicrobial activity. The results indicated that fruit flesh presents a good amount of antioxidants that can benefit the human diet, while the peels could be used for nutraceutical formulation and food applications [51].

Different from the other articles in the ranking, the seventh position discusses other possible applications of pitaya peels, which is its use as an adsorbent for removing dissolved heavy metals and dyes from water [52,53]. In this study, spectroscopic and electron microscopic techniques were used to determine the presence of surface functional groups (−CO_2H, −OH) and the morphologies of the peels. The results show that the extraction capacity of peels increased with time and then reached equilibrium. Pitaya peels showed the highest extraction efficiency toward alcian blue (71.85 mg g^{-1}) and methylene blue (62.58 mg g^{-1}). In addition, the adsorbents can be regenerated at acidic pH and reused, consisting of a low-cost alternative treatment for existing technologies [52].

The most cited studies make it possible to identify a strong research theme related to the nutritional aspects of pitaya, establishing its potential for food fortification, bringing benefits to human health. Another important topic that is under discussion is the use of pitaya as a source of healthier pigments that could be valuable to the food industry, helping the transition from the use of synthetic pigments and additives by replacing them with natural products. Finally, the most distinguished research theme addressed by the most cited articles was contemplating the use of pitaya peels in producing adsorbent materials for water and wastewater treatment. In the biorefinery concept, it is possible to visualize pitaya being used for several food applications. At the same time, the by-product application is a topic that deserves attention and the development of new research, testing different routes for the revalorization of materials that were considered waste for obtaining new added-value products. Once the research around by-products valorization is developed, the biorefinery and circular economy concepts will be able to occur in real situations.

3.4. Study of authors, journals, institutions, and countries

The bibliometric study also included the main authors, journals, institutions, and countries related to pitaya research (**Table 1**). The results show the main authors, as well as the number of publications and citations over the last 20 years (**Fig. 3b**). Among the main authors, *Mizrahi Y* is in first place with 28 publications, which is equivalent to 3.63% of the 770 documents found, followed by *Carle R* (16 documents), *Tel-zur N* (15 documents), *Stintzing FC* (13 documents) and *Ramos JD* (11 documents). The journals *Scientia Horticulturae*, *Food Chemistry*, *Postharvest Biology and Technology*, and *LWT - Food Science and Technology* are the most influential journals on the research field (**Fig. 3c**). The 10 most prestigious journals in the study of pitaya are related to the scope and research areas of *Food Science Technology*, *Agriculture*, and *Chemistry*, corroborating with the analysis of the most important research areas. **Figure 3d** and **Figure 3e** presents the most expressive countries in the field of pitaya research. China is responsible for 168 publications and corresponds to 21.81% of the publications. Malaysia, Mexico, and Brazil are responsible for almost 60% of the 770 publications. The collaboration between the main countries demonstrates an intense partnership between the countries in the search for scientific discoveries.

Figure 3f shows the three-field plot correlating the top authors, keywords, and countries. The results show the top 15 authors along with the top 15 keywords used by them and the top 15 countries. Practically all authors used the word pitaya, which characterized it as the most used keyword, and this can be explained by the fact that the fruit is popularly known as pitaya. Analyzing the keywords, removing the popular and scientific names, the keywords *antioxidant*, *anthocyanin*, *phenolic compounds*, and their variations are found, which demonstrates a great interest of the scientific community in the study of bioactive compounds from this fruit. Research is intensely concentrated in countries such as China, Malaysia, Brazil, and Mexico. This intensity can be explained by the fact that dragon fruit is an exotic fruit and is easily found in the countries mentioned above.

4. Overview of pitaya: Characteristics, processing, and generation of by-products

The demand for fruits with beneficial health properties is growing considerably in recent years. The pitaya is a fruit from the *Cactaceae* family and originated from Central and South America. Pitaya is also known as dragon fruit or pitahaya [54]. Pitaya is widely cultivated in tropical or subtropical areas and can be classified by the appearance of pulp (red or white) and peel (red or yellow) [55–57].

The physicochemical (**Table 6**) and mineral (**Table 7**) characterization of pitaya was summarized to better understand the nutritional value of the fruits and the potential to produce commercial products. In general, the physicochemical characteristics of pitaya vary according to the species due to the great variety presented by this fruit [53,58–60]. Pitaya presents a high moisture content, with higher content of carbohydrates and proteins. In addition, the average values of crude fibers in pitaya is 11.28%, being considered a rich source of dietary fibers and significant when included in the human diet [53]. Pitaya is a rich source of phenolic compounds, vitamins, and minerals. Among the vitamins present in the fruit are B_1, B_2, B_3, C, niacin, pyridoxine, and cobalamin [61]. Nitrogen, potassium, and calcium are the minerals with the highest amount. Among the microminerals, iron and manganese stand out comparing with the other minerals [53,58,59,62]. Otherwise, pitaya peel, which represents approximately 40% of the whole fruit mass, also contains several essential nutrients, including lipids, proteins and carbohydrates [55,63–65]. Pitaya peel has 57.25 g of fiber per 100 g of peel, which characterizes it as a source of fiber. In addition to the high fiber content, the peel has 729.7 mg vitamin C per kg of residue [55].

Table 6. Physicochemical characterization of the pulp and peel of pitaya.

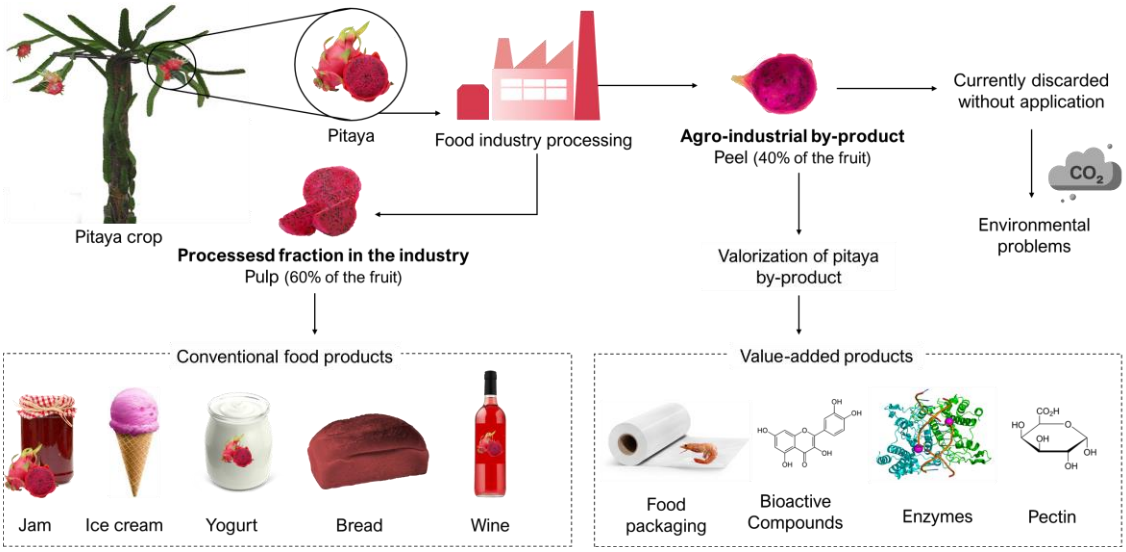
Pitaya pulp (fresh weight)	Ramli and Rahmat [58]	Ramli and Rahmat [58]	Rohin et al.[59]	Unit
Moisture	85.05	89.98	83.00	%
Ash	0.54	1.19	0.28	%
Carbohydrates	12.97	8.42	-	%
Protein	1.45	0.41	0.23	%
Lipids	-	-	0.61	%
Total dietary eiber	2.65	-	0.90	%
Pitaya peel (dry weight)	Utpott et al. [63]	Morais et al. [64]	Madane et al.[65]	Unit
Ash	17.56	18.21	4.48	%
Carbohydrates	8.92	70.69	-	%
Protein	6.30	6.16	10.36	%
Lipids	1.31	4.13	2.34	%
Total dietary fiber	65.59	57.25	56.91	%

Table 7. Mineral characterization of the pulp of pitaya.

Parameters	Cordeiro et al. [53]	Nurul and Asmah [60]	Rohin et al.[59]	Unit
Bromine	18.7	-	-	mg kg^{-1}
Calcium	-	67.2	57.0	mg kg^{-1}
Copper	21.7		0.30	mg kg^{-1}
Iron	337.6	300	34.0	mg kg^{-1}
Phosphor	-		230.0	mg kg^{-1}
Magnesium	-	264.0	283.0	mg kg^{-1}
Manganese	113.9	-	-	mg kg^{-1}
Nitrogen	-	-	-	mg kg^{-1}
Potassium	-	1580	56.96	mg kg^{-1}
Sodium	-	356.3	501.5	mg kg^{-1}

Considering that the edible part of the fruit is the pulp, which is equivalent to approximately 60% of the fruit mass, there is a high generation of by-products during the processing of pitaya. Currently, the pulp of pitaya is used to produce ice cream, jellies, yogurt, bread, and wine [66]. The pitaya peel is the main by-product generated during processing. Studies have been conducted to promote the sustainable destination of pitaya by-products, aiming the circular economy transition of this industrial sector. **Figure 4** shows a general representation of the pitaya agro-industrial processing and the generation of pitaya by-product. The valorization of pitaya peel has been studied mainly into bioactive compounds, food packaging, enzymes, and pectin. These value-added products can be produced from the peel, which acts as a strategy to promote the circular economy transition of this agro-industrial sector. The next section presents a deep discussion regarding the research trends, gaps, hot topics, and perspectives to produce value-added products from pitaya.

Figure 4. Schematic representation of the food industry processing, generation of by-product, and valorization.



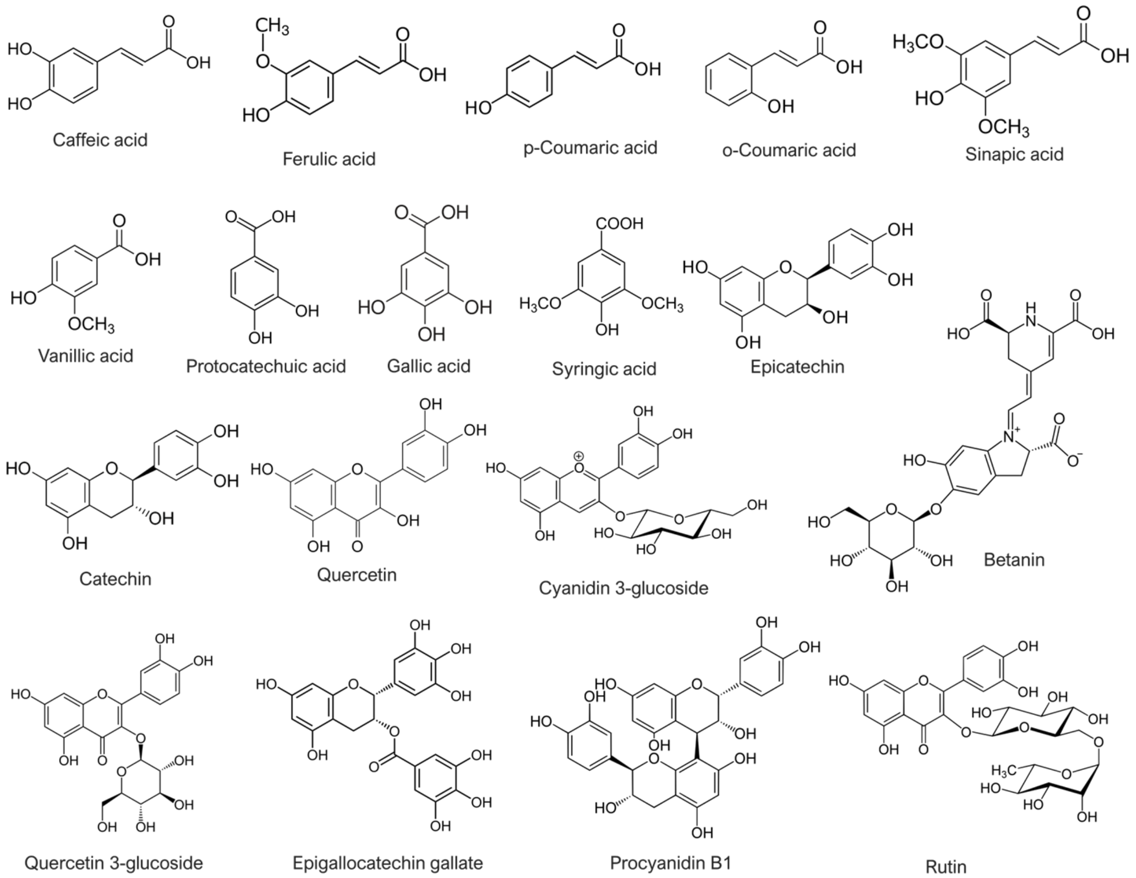
5. Research trends, gaps, hot topics, and perspectives

5.1. Bioactive compounds

The pulp of pitaya has an extensive content of phytochemicals, especially phenolic compounds, betacyanin, and terpenoids [55,67–69]. Recently, with the increasing popularity of functional food, pitaya has become an agricultural product that has drawn attention and interest from researchers. Pitaya is widely used for medicinal purposes since its leaves contain a hypoglycemic and diuretic effect, where the continuous consumption can reduce chronic diseases [70]. In addition, plant-derived by-product from pitaya processing has been used as functional and nutritional ingredients, especially due to the rich source of bioactive compounds.

The betacyanin is the main phytochemicals obtained in pitaya peel [55,71,72]. In addition, phenolic compounds, anthocyanins, flavonoids, and flavones can be obtained in lower amount. **Figure 5** shows the main bioactive compounds obtained from pitaya.

Figure 5. Main bioactive compounds obtained from pitaya pulp and peel.



The peel of pitaya presents higher content of phenolic compounds than the pulp [55]. Terpenoids and alkaloids are also found in pitaya by-product, however, studies involving these substances are scarce in the literature, and should be further investigated. According to o Roriz et al. [73], pitaya peel presents α-tocopherol (3.10 mg 100 g^{-1} d.w.), β-tocopherol (0.16 mg 100 g^{-1} d.w.), γ-tocopherol (11.8 mg 100 g^{-1} d.w.), and δ-tocopherol (0.84 mg 100 g^{-1} d.w.). The extracts obtained from pitaya presents considerable high antioxidant, antimicrobial, and anticancer activity

[72,74–76]. Due to the abundance of phytonutrients in its composition, the effect on *in vitro* fermentation of pitaya bark powder was evaluated [77]. The addition of pitaya by-product improved the fermentation, nutrient degradation, and significantly promoted the reduction of methane production, which is a very promising result when considering the search for reduction of greenhouse gas emissions [77]. Pitaya has been evaluated as a promising source of betacyanin, especially betalain, which is the main red dye used in the food industry, usually extracted from red beets. In recent years the extraction of beet dyes is being reduced, and there is a possibility to obtain betalain from pitaya by peel [78,79]. Pitaya peel presents betanin (10.1 mg g^{-1} extract), isobetanin (11.8 mg g^{-1} extract), phyllocactin (14.8 mg g^{-1} extract), and isophyllocactin (11.7 mg g^{-1} extract) as the major betacyanins [73]. In addition, the seeds of pitaya are a source of flavonoids, such as catechin (3.6 mg g^{-1}), epicatechin (0.6 mg g^{-1}), rutin (0.53 mg g^{-1}), quercetin (1.31 mg g^{-1}), and myricetin (0.63 mg g^{-1}) [80]. Finally, the presence of bioactive compounds in pitaya pulp and by-products (peel and seeds) demonstrated that it is possible to apply extraction methods to promote its valorization, avoiding the inappropriate disposal and producing value-added products.

5.2. Green extraction techniques of bioactive compounds

For the extraction of bioactive compounds, several extraction methods can be applied [55]. **Table 8** summarizes the extraction methods of bioactive compounds form pitaya. The extraction of bioactive compounds can be classified into “classical” and “modern” methods. The classical extraction methods are designated by utilizing larger volume of extraction organic solvents, manual procedures, and large time. Modern extraction methods have been recently used due to the automation capacity, higher extraction efficiency, and reduced use of solvents [81].

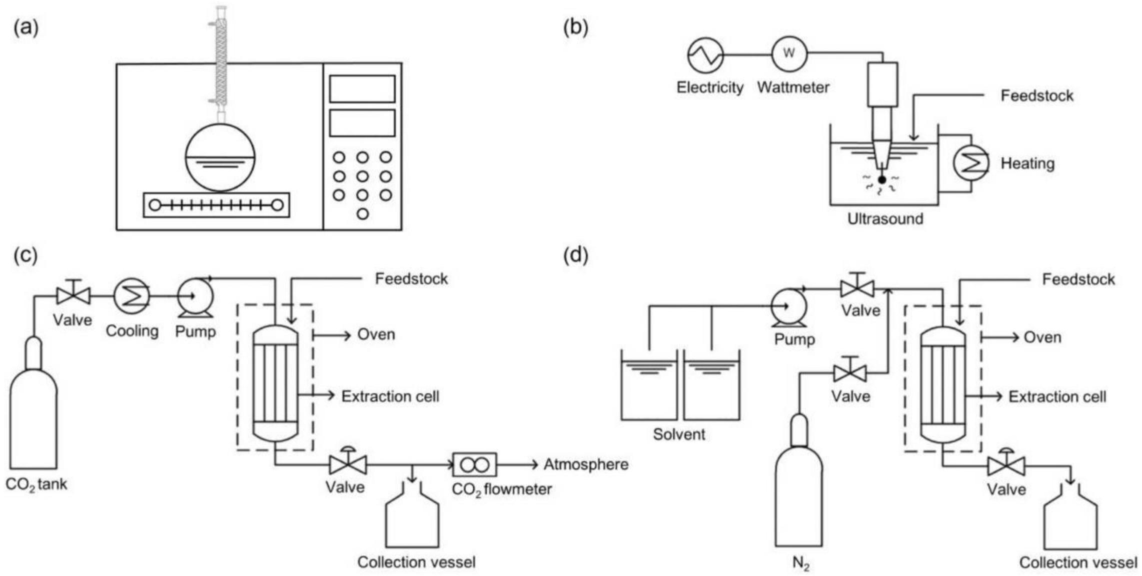
Table 8. Recovery of value-added products from pitaya with different extraction methods.

Value-added product	Extraction method	Operational parameters	Yield	Reference
Amylase	Aqueous extraction	2.5 ºC, 2 min, pH 5, S/F 4:1 w/w	-	[109] [105]
Pectinase	Aqueous extraction	S/F 5:1 w/w, 5 ºC, pH 8	-	[111]
Pectin	Microwave	400 W, 45 ºC, 20 min, S/F 24 w/w	7.5%	[106]
Pectin	Microwave	300 – 800W, pH 2.07, 65 s, S/F 66.57 w/w	11.8 – 18.5%	[107]
High methoxyl-pectin	Ultrasound	250 rpm, 70 ºC 120 min, pH 2, S/F 10 w/w	28.20%	[108]
High methoxyl-pectin	Ultrasound	250 rpm, 65 ºC 70 min, pH 2, S/F 12 w/w	30.11%	[71]
Betacyanin	Alcoholic solution	Ethanol 30%, 4 ºC, overnight	32%	[123]
Betacyanin	Aqueous extraction	S/F 1/4 w/w, pH 6	-	[110]
Betacyanin	Pressurized liquid	25 MPa, 50 ºC, 1.5 h, ethanol/water 10/90 v/v	4.12%	[83]
Betacyanin	Net biphasic float	Ethanol 100 %, 15 min, 25 ºC	-	[84]
Betacyanin	Net biphasic float	27 % v/v ethanol, 20% v/v K_2HPO_4, 50% v/v water, 1 % v/v sample, 2% v/v NaCl 0.4M	-	[79]
Polyphenols	Microwave	S/F 1/150 w/w, 49.33 ºC, 5 min	17.36%	[91]
Polyphenols	Ultrasound	100 W, 60 ºC, S/F 25/1 w/w, 20 min	8.536 mg g^{-1}	[93]
Polyphenols	Aqueous extraction	Methanol 80%, 25 ºC, 3 days, occasional sonication (max 60Hz 30 min/day)	1.5%	[77]
Flavonoids	Aqueous extraction	Methanol 80%, 25 ºC, 3 days, occasional sonication (max 60Hz 30 min/day)	1.4%	[77]
Flavonoids	Aqueous extraction	S/F 1/50 v/v, 300 rpm, 24 h, 25 ºC	5.99%	[76]

In the case of pitaya, classical extraction methods have been widely investigated. Organic solvents have been used for the extraction of bioactive compounds, however, the use of organic solvents should be reduced due to safety issues and the toxicity [82]. Another reason for seeking alternative to replace organic solvents in the extraction of phytochemicals is associated with the high cost and time-consuming [83]. The extract obtained from conventional extraction techniques have disadvantages of deterioration of thermosensitive compounds and promote damage to the environment [84].

Notwithstanding, sustainable extraction techniques have being evaluated to promote sustainability in the extraction of bioactive compounds [85,86]. The advantages of modern extraction methods are associated with the use of renewable resources (e.g., by-products as feedstock), low energy consumption, high yield of extraction of the target compound, non-generation of inhibitors, automated process, and high degree of purity of the target compound [84,87].The most applied sustainable and modern techniques are microwave-assisted extraction, ultrasound-assisted extraction, pressurized liquid extraction, and supercritical fluids extraction (**Figure 6**). These new techniques are classified as eco-friendly and can be an alternative to the classical methods.

Figure 6. Representation of the modern and sustainable extraction techniques of bioactive compounds. (a) Microwave-assisted extraction; (b) Ultrasound-assisted extraction; (c) Supercritical fluid extraction; (d) Pressurized liquid extraction. Reproduced from [7], with permission from Elsevier.



Microwave-assisted extraction (**Figure 6a**) consists of the passage of microwave through a fluid absorbing energy and converting it into thermal energy. In this process, the increase in temperature promotes the evaporation of moisture by increasing the pressure inside the cell wall and organelles [88]. This increase in pressure generates the increase of the pores of the matrix thus facilitating the passage of the solvent [84]. The use of the microwave has as a disadvantage the degradation of heat-sensitive bioactive compounds and is less compatible with flow system than other extraction methods [89]. Ferreres et al. [90], optimized the recovery of high-value compounds from pitaya fruit by-products using microwave-assisted extraction. The maximum betacyanin content was achieved with a solid/solvent ratio of 1/150 g/mL, temperature of 49.33 °C, and the extraction time of 5 min [90]. In addition, the optimal microwave-assisted extraction parameters were 400 W, 45°C, 20 min, and 1.2 g of sample weight per 50 mL water [91].

Ultrasound-assisted extraction (**Figure 6b**) is one of the most widely used emerging techniques in the extraction of bioactive compounds from plant tissues. The extraction process occurs via cavitation, which promotes the rupture of plant tissues facilitating the permeation of the solvent and the extraction of bioactive compounds. This method presents good specificity, economic viability, and high yield. However, it presents wave attenuation in phase systems dispersed over long distances due to the reduction of wave amplitude and presents difficulties in the extraction of unstable compounds [89]. The influence of ultrasonic temperature (30–70 °C), solvent to solid ratio (10:1–30:1 mL/g), solvent concentration (30–60%), and ultrasonic treatment time (5–25 min) was investigated on the recovery of total polyphenolic content, antioxidant activity, and betacyanin content [92]. The optimal ultrasonic conditions were obtained at 60 °C, solvent to solid ratio 25:1 mL/g, solvent concentration 60%, and ultrasonic treatment time of 20 min [92]. The optimal variable for ultrasound-assisted extraction were 54.6 °C of extraction temperature, 0.3 s^{-1} cycle, 20% ultrasound power, 21.4% methanol in water solvent composition, 0.2:10 sample to solvent ratio, and 5 min extraction time [93]. Finally, ultrasound-assisted extraction is a sustainable, reliable, economical, and rapid technique for obtaining betacyanin from pitaya.

The extraction process with supercritical fluids (**Figure 6c**) has been investigated to recovery value-added bioactive compounds [89]. The supercritical state is dyed through temperature and pressure control. Usually the fluid used is carbon dioxide because it has low temperature (31.2 ºC) at the critical pressure (73.8 bar) [94]. This extraction method can be used in the extraction of volatile and thermosensitive compounds, since depending on the fluid used the temperatures and pressures vary. The extracted compounds have a high degree of purity and is free of solvents, the extract has no contact with oxygen thus avoiding oxidation [84]. The supercritical fluid extraction presents the disadvantage of extraction of low polarity compounds [84,94]. According to Fathordoobady et al. [82], 91.27 mg 100 mL^{-1} total betacyanin can be produced by supercritical fluid extraction at 25 MPa pressure and 10% EtOH/water (v/v) mixture as a co-solvent contained 24.58. The highest oil yield obtained from pitaya with supercritical fluid extraction was predicted to be about 6.93 wt%, under optimal conditions temperature of 47 ºC and pressure of 4750 psi [95]. At optimal condition of supercritical fluid extraction (25 MPa, 50°C, and 15% co-solvent), the response variables were assessed as maximum extraction yield of 4.09 ± 0.69%, total betacyanins content of 25.49 mg 100 mL^{-1} [96]. The main components of pitaya peel extracted by supercritical carbon dioxide were β-amyrin (15.87%), α-amyrin (13.90%), octacosane (12.2%), γ-sitosterol (9.35%), octadecane (6.27%), 1-tetracosanol (5.19%), stigmast-4-en-3-one (4.65%), and campesterol (4.16%) [97]. Finally, the use of supercritical fluid extraction demonstrated a suitable and sustainable technique recovery bioactive compounds from pitaya.

The pressurized liquid extraction (**Figure 6d**) and subcritical water extraction have been studied to extract bioactive compounds. In the subcritical state the system has a temperature lower than the critical temperature and the pressure is higher than the saturation pressure presented by the solvent, which is possible to keep the water in the liquid state [98]. Currently, the subcritical water extraction in temperature below 100 ºC has been applied to extract anthocyanins from fruit by-products [99,100]. In addition, subcritical water at temperatures higher than 180 ºC can be used for the dissociation of cellulose and hemicellulose, releasing oligosaccharides from biomasses [101]. The advantages of pressurized liquid extraction and subcritical water extraction are the high efficiency, low waste generation, and short extraction time [102,103]. However, the implementation of these processes is considered as expensive, and the scale-up should be further investigated.

5.3. Enzymes and pectin

The use of pitaya by-products has been studied for the extraction of enzymes and pectin, which are value-added products that can be obtained in a context of “waste to bioproducts”. The pitaya peel has different types of enzymes, which makes it a rich source and an economical alternative for the commercial production of natural enzymes such as amylose and pectinase [104–106]. Commercial pectins are classified according to the degree of esterification, pectins that present a degree of esterification greater than 50% are classified as high degree of esterification and pectins that present lower values are pectins of low degree of esterification [107]. The main sources of commercial pectin are citrus peels [108,109], and pitaya peels can be used as an unconventional source. The pitaya peel was evaluated as a potential source of pectin. Ultrasonic extraction demonstrates that the process should be carried out at 45°C for 30 min [110]. The extracted pectin showed high antioxidant capacity and is characterized as low methoxylic pectin or as high-esterification pectin (i.e. the extracted pectin can form gels with low action levels) [110]. The degree of esterification presented by the pectins extracted with the aid of microwaves was 50.41 % [107]. Muhammad et al. [111] evaluated the degree of esterification presented in the extraction of pectins from dried and fresh pitaya skins, ranging from 51.71 to 68.00 %, that is, all pectins extracted were classified as pectins with a high degree of esterification.

Pectin obtained from pitaya peel also has a significant capacity for film formation, thus demonstrating its possible application as a biopolymer in edible films or packaging [108]. The pectin yield obtained from pitaya peel can vary according to the type of extraction used. When compared to conventional extraction with ultrasound-assisted extraction, it obtained a higher yield presenting 19.48% when used at a temperature of 75ºC for 60 minutes [110]. Various studies using different types of extraction showed yields between 14.86 and 26.38% [111–114]. The yield found for pectin extraction using the inner part of the pitaya peel (26.38%) was higher than that found for other foods such as passion fruit peel (14.80%), apple pomace (14.55%), lemon by-product (11.21%) and peach pomace (11.40%). Currently, research on obtaining pectin from pitaya peels is limited, focusing mainly on the optimization of extraction processes, thus leaving its specific properties unknown [107,115]. Few publications are focused on the properties of the extracted pectins. [115], evaluated the emulsifying capacity of pectins extracted from pitaya peels, which showed excellent antioxidant and emulsifying activity when compared to commercial pectins, thus allowing pitaya peel pectins to be considered as promising natural emulsifiers.

5.4. Incorporation in food products

The use of pitaya extracts in the food industry has also been studied. The extracts obtained by the classical and modern extraction techniques presented high concentration of bioactive compounds (flavonoids, hydroquinone phenols, sterols, triterpenoids, saponin, and tannins). The flavonoids and hydroquinone phenols are directly related to antioxidant activity and steroids and triterpenoids contribute to antibacterial activity. In addition, the application of pitaya extract can increase the resistance to oxidative stress and reduce the lipid oxidation of beef sausage [74]. The food functionalization with pitaya extracts have been studied in the bakery and dairy products. The addition of pitaya peel powder in the composition of bread promoted the increase of the content of betacyanin, phenolic compounds and antioxidant capacity [116]. The bread presented a change of color due to the presence of betacyanin, which was expected due the characteristics of the pigment. The final product with the addition of 3% of pitaya peel flour was sensorially accepted. The food can be characterized as a healthy and colorful with high phytochemical composition [116]. In dairy products, the high content of fibers in pitaya peel was evaluated as a promising fat substitute in ice cream [63,75]. The addition of peel flour promoted the formation of a low-fat ice cream without harming the physical and sensory characteristics, with the improvement of the rheological characteristics. The final product besides fat reduction presented high antioxidant capacity, which can provide benefits to a healthy diet [63,75].

5.5. Food packaging

Another promising application of pitaya by-products and extracts is in the production of films and coatings, that can be applied as food packaging [117]. The extracts of pitaya can be incorporated in food packaging to monitor the freshness of crayfish [118]. The addition of pitaya extracts decreased the deterioration process, maintained the crayfish within acceptable quality parameters and extending the shelf life. The performance is directly associated with the content of phenolic compounds present in their composition [118]. Similar results were obtained when encapsulated extracts from pitaya were added in pork, reducing oxidative stress during storage, and increasing the shelf life [119]. Intelligent active packaging was produced with the addition of pitaya bark extract and the films produced showed improvement in the water vapor barrier and mechanical properties. In addition, the films showed improvement in the light barrier, antioxidant, and antimicrobial capacity. The study suggested that the packaging produced in addition to serving to extend the shelf life of the commercialized product can be used as a quality indicator, since the betalain present in the film is sensitive to the pH change promoted during food degradation [120]. Studies involving the application of extracts and pitaya peel flour in foods are growing, but there is still a need for further studies related to the subject [55].

6. Conclusion

The growth in waste generation has been caused by the high demand for food production. Due to this, several reuse methods are being developed. Pitaya is an exotic fruit that is widely consumed in Asia and has about 30% of its weight in the form of waste. Research involving pitaya pulp and peel has grown more than eight times in the last 11 years. Among the main subjects researched is the obtaining of antioxidant compounds such as betacyanins and phenolic compounds. Studies show that extracts from pitaya bark also have antimicrobial, anticancer activity and other functional properties. Despite the increase in research related to extraction, issues related to other functional properties and the application of these compounds are still scarce.

In view of the concern with environmental impacts, the need to reduce costs and favor the circular economy, new methods of extracting and using waste are being used. Therefore, a greater focus is needed on the use of pitaya peel as a commercial source of compounds with high added value, to evaluate the other functional activities of the extracted compounds and to study their possible applications.

CRediT authorship contribution statement

Vanessa Cosme Ferreira: Writing - original draft. **William Gustavo Sganzerla**: Conceptualization, Writing - original draft, Writing - review & editing. **Larissa Castro Ampese:** Writing - original draft, Writing - review & editing. **Leda Maria Saragiotto Colpini**: Supervision, Writing - review & editing. **Tânia Forster-Carneiro:** Conceptualization, Supervision, Resources, Project administration, Funding acquisition, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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CAPÍTULO 3 – Sustainable valorization of pitaya (Hylocereus spp.) peel in a semi-continuous high-pressure hydrothermal process to recover value-added products

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Sustainable valorization of pitaya (*Hylocereus* spp.) peel in a semi-continuous high-pressure hydrothermal process to recover value-added products

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Abstract

This study evaluated the use of a semi-continuous high-pressure hydrothermal process for the recovery of value-added products from pitaya peel. The process was carried out at 15 MPa, a water flow rate of 2 mL min^{-1}, a solvent-to-feed ratio of 60 g water g^{-1} pitaya peel, and temperatures ranging from 40 to 210 °C. The results show that extraction temperatures (between 40 and 80 °C) promoted the recovery of betacyanin (1.52 mg g^{-1}), malic acid (25.6 mg g^{-1}), and citric acid (25.98 mg g^{-1}). The major phenolic compounds obtained were *p*-coumaric acid (17.95 ± 5.01 μg mL^{-1}), protocatechuic acid (11.43 ± 4.49 μg mL^{-1}), and piperonylic acid (9.27 ± 3.65 μg mL^{-1}). The hydrolysis temperatures (between 150 and 210 °C) could produce sugars (18.09 mg g^{-1}). However, the hydrolysis process at temperatures above 180 °C generated Maillard reaction products, which increased the total phenolic compounds and antioxidant activity of the hydrolysates. Finally, the use of semi-continuous high-pressure hydrothermal process can be a sustainable and promising approach for the recovery of value-added compounds from pitaya peel, advocating a circular economy approach in the agri-food industry.

Keywords: *Food industry; Biomass; Betalains; Bioactive compounds; Green extraction; Circular economy.*

1. Introduction

In recent years, food processing has expanded in response to rising global food demand, which has increased the generation of agri-food by-products, such as pomace, seeds, stalks, and peels (Rodrigues et al., 2022). The incorrect destination (e.g., open-air, burning, and landfills) of organic waste generated during agri-food processing causes environmental impacts by the emission of greenhouse gases (Nattassha et al., 2020). The by-products generated during the food processing industry must be disposed of in an environmentally appropriate manner within the circular bioeconomy (Ghosh et al., 2020; Yousefloo and Babazadeh, 2020). The implementation of new sustainable technological processes that could transform agri-food by-products into high value-added materials is a promising approach for the circular economy transition of the food industry (Chhandama et al., 2022; Gianico et al., 2021).

Pitaya (*Hylocereus* spp.) is a tropical fruit popular for its attractive bright red skin color studded with green scales (Madane et al., 2020). In the case of the pitaya processing industry, the peel is an abundant by-product generated during industrial processing, accounting for approximately 30% of the total weight of the fruit (Jimenez-Garcia et al., 2022a). The correct destination for pitaya peel represents a challenge for the pitaya processing industry. Currently, the peel is discarded in landfills, used in composting, and incinerated, which can cause environmental pollution due to harmful greenhouse gas emissions (Georgin et al., 2022). Over the years, Brazil has stood out as a pitaya producer due to its growing cultivation and consumption (Georgin et al., 2022). In 2021, the worldwide market for pitaya reached USD 425.7 million, with an average price of USD 4.56 per kg of fruit. For 2023, pitaya production is expected to reach 93 million tons (Tridge, 2022 ; Puttipan and Khankaew, 2023).

The application of agri-food by-products for the recovery of value-added products has been widely investigated. The biobased products obtained can be used in different industrial sectors (pharmaceutical, chemical, and food) to manufacture natural products, such as antioxidant-rich food supplements (Ferrari et al., 2019; Makris et al., 2007; Zúñiga-Muro et al., 2020). Pitaya peel can be used as a source of natural bioactive agents since it contains flavonoids, phenolic acids, and betacyanin with antioxidant, antibacterial, and anti-inflammatory properties (Xin et al., 2022). For the extraction of bioactive compounds, several extraction methods can be applied (da Silva et al., 2022; Jiang et al., 2021; Lucía et al., 2021). Classical extraction methods use larger organic solvents, manual procedures, and long times. Modern extraction methods have been used due to their automation capacity, higher extraction efficiency, and lower use of toxic organic solvents (da Silva et al., 2022; More et al., 2022; Sanches et al., 2022), promoting sustainability in the extraction process (da Silva et al., 2022; Lucía et al., 2021). The advantages of modern extraction methods are associated with the use of renewable resources (e.g., by-products as raw material), low energy consumption, high extraction yield of the target compound, non-generation of inhibitors, automated processes, and high purity of the target compound (Chemat et al., 2020; More et al., 2022).

Ultrasound-assisted, microwave-assisted, pressurized liquid, supercritical, and enzymatic extractions are commonly used techniques that are green and sustainable (Armenta et al., 2019; Ferreira et al., 2023). Each method has its characteristics and limitations. In the case of ultrasound extraction, there is wave attenuation in dispersed phase systems and difficulties in extracting unstable compounds. Microwave-assisted extraction, on the other hand, has the potential to degrade heat-sensitive compounds (Ferreira et al., 2023). Lastly, supercritical and enzymatic extractions are associated with high costs (Armenta et al., 2019; Das et al., 2021). However, these techniques offer efficient and environmentally friendly approaches to extracting bioactive compounds.

Notwithstanding, there is a challenge regarding using lignocellulosic materials to produce soluble molecules (e.g., sugars and organic acids) (Raj et al., 2022; Zhao et al., 2022). Several conventional pretreatments based on acid, alkaline, and mechanical hydrolysis have been used to promote the breakdown of lignocellulosic structure (Akhtar et al., 2016; Lay et al., 2022). However, conventional pretreatments have high operational costs, high demand for reagents, and high energy consumption and generate contaminants after hydrolysis, requiring additional steps to treat the residues generated after pretreatment (Ma et al., 2022). Therefore, novel sustainable pretreatment methods should be evaluated to promote a sustainable approach to the rupture of the lignocellulose structure. Recently, high-pressure hydrothermal pretreatment has been considered a promising approach to produce value-added materials from biomass (Ruiz et al., 2023; Yue et al., 2022). The main advantage of the high-pressure hydrothermal process is that it can extract bioactive compounds at low temperatures (lower than 100 °C) and hydrolyse biomass at high temperatures (higher than 100 °C). In addition, previous studies elucidated the development of a semi-continuous high-pressure hydrothermal process for the sustainable valorization of grape pomace (Castro et al., 2023), jabuticaba peel (Barroso et al., 2022a, 2022b), brewer's spent grains (Sganzerla et al., 2023, 2022), and poultry feathers (Di Domenico Ziero et al., 2022) into value-added products (bioactive compounds, sugars, xylooligosaccharides, amino acids, and organic acids). Therefore, high-pressure hydrothermal pretreatment is a relevant strategy for the development of the circular bioeconomy, as it is extremely advantageous in the valorization of agri-food by-products (Singh et al., 2023). However, the application of hydrothermal pretreatment of pitaya peel has been poorly investigated. Scientific knowledge should be better developed to verify the application of high-pressure hydrothermal processes for the valorization of pitaya peel into value-added products.

Therefore, this study investigated the valorization of pitaya peel in a semi-continuous high-pressure hydrothermal process to recover value-added products. The study focuses on the application of low temperatures to the recovery of bioactive compounds and high temperatures to the hydrolysis of biomass and recovery of sugars and organic acids. This study provides the first approach for producing value-added products from pitaya peel using a semi-continous high-pressure hydrothermal process.

2. Materials and methods

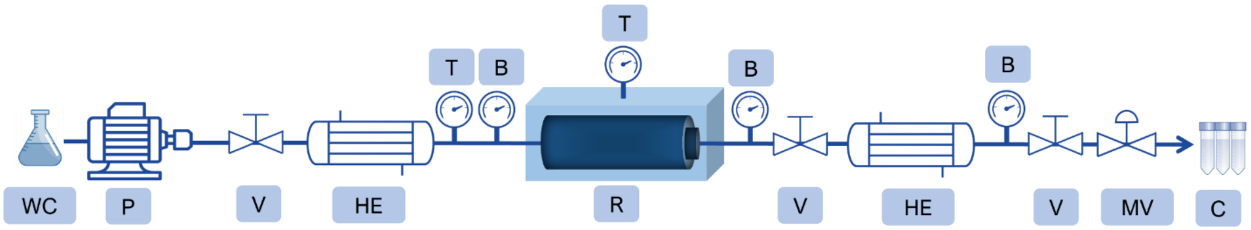
2.1. Raw material

Pitaya fruits (5 kg) were obtained from a local market (Campinas, SP, Brazil). The fruit was manually separated into pulp and peel. The peel was dried (60 °C, 24 h), crushed in a knife mill, and sieved to reach an average particle size of 1 mm. The dried pitaya peel was stored (−18 °C) for characterization and use in high-pressure hydrothermal processes. The pitaya peel was characterized by the content of moisture, lipids, protein, crude fiber, neutral detergent fiber, acid detergent fiber, lignin, hemicellulose, cellulose, starch, total reducing sugars, and total sugars (AOAC, 2012).

2.2. Semi-continuous high-pressure hydrothermal process

The valorization of pitaya peel was conducted in a semi-continuous high-pressure hydrothermal process (**Figure 1**). Previous studies determined the optimal operational conditions of the process for extraction and hydrolysis (Barroso et al., 2022a, 2022b; Maciel-Silva et al., 2022; Sganzerla et al., 2022). The process was operated at 15 MPa, a water flow rate of 2 mL min^{-1} at pH 2 (pH adjusted with phosphoric acid 85%), an initial feed of 1.5 g pitaya peel, a total time of 60 min, and a solvent-to-feed ratio of 60 g water g^{-1} pitaya peel. This study investigated the effect of temperature during semi-continous high-pressure hydrothermal process: 40, 60, and 80 °C for extraction and 150, 180, and 210 °C for hydrolysis. At each extraction, the reactor was properly cleaned and refilled, and the parameters were modified. The samples were directly subjected to the pressure and temperature of the indicated treatment without the need for thermal pretreatment. Extraction and hydrolysis kinetics were performed every 6 min to verify the effect of operational conditions during the semi-continuous process. After obtaining the extracts and hydrolysates, they were centrifuged (3000 rpm, 10 min), and the supernatant was stored at −18 °C for further analysis.

Figure 1. Schematic diagram of the experimental unit for the semi-continous high-pressure hydrothermal process of pitaya peel. Label: WC, water container; P, pump; V, valve; HE, heat exchanger; T, thermocouple; B, barometer; R, reactor; MV, micrometric valve; C, collecting vessel.



2.3. Extraction by conventional techniques

Three conventional techniques (mechanical extraction at pH 7, ultrasound-assisted extraction, and mechanical extraction at pH 2) were performed to compare the results and verify the efficiency of the semi-continuous high-pressure hydrothermal process.

2.3.1. Mechanical extraction (pH 7)

For mechanical extraction at pH 7, 1.5 g pitaya peel (dry basis) was mixed with 50 mL of a hydroethanolic solution (70% ethanol, v/v) at pH 7. The sample was mixed in an orbital shaker for 1 h (150 rpm and 25 °C). The extract obtained was centrifuged (3000 rpm, 10 min), and the supernatant was stored at −18 °C for further analysis.

2.3.2 Ultrasound-assisted extraction

For ultrasound-assisted extraction, 1.5 g pitaya peel (dry basis) was mixed with 50 mL of a hydroethanolic solution (70% ethanol, v/v) at pH 7. The sample was sonicated for 1 h at 37 kHz and 25 °C (Solidsteel, Piracicaba, SP, Brazil). The extract obtained was centrifuged (3000 rpm, 10 min), and the supernatant was stored at −18 °C for further analysis.

2.3.1. Mechanical extraction (pH 2)

For mechanical extraction at pH 2, 1.5 g pitaya peel (dry basis) was mixed with 50 mL of a hydroethanolic solution (70% ethanol, v/v) at pH 2 (pH adjusted with phosphoric acid 85%) (i.e., the same pH used in the semi-continuous high-pressure hydrothermal process). The sample was mixed in an orbital shaker for 1 h (150 rpm and 25 °C). The extract obtained was centrifuged (3000 rpm, 10 min), and the supernatant was stored at −18 °C for further analysis.

2.4. Characterization of extracts and hydrolysates

2.4.1. Color Parameters

The color parameters of the extracts and hydrolysates were determined according to the CIELab scale using a digital colorimeter (CM-600d, Konica Minolta, Osaka, Japan). The color parameters determined were L* (lightness), a* (redness/greenness), b* (yellowness/blueness), and C* (chroma).

2.4.2. pH

The pH values of the extracts and hydrolysates were obtained from a digital pH meter (IonLab, model THS-3E, New York, NY, USA). The measurements were performed at 25 °C with the pH meter properly calibrated.

2.4.3. Total phenolic compounds

The determination of total phenolic compounds was performed according to Sigleton and Rossi (1965). Initially, 60 μL of the sample was mixed with 3000 μL of distilled water and 300 μL of Folin-Cicalteau reagent. After 3 min, 900 µL of sodium carbonate solution (15%) and 1740 µL of distilled water were added to the reaction. The reaction was maintained in a dark environment, and after two hours, the absorbance was measured in a spectrophotometer (Hach, model DR 4000 U, São Paulo, SP, Brazil) at 765 nm. Gallic acid was used as a standard, and the results were calculated from the calibration curve with seven points (y = 0.0015x – 0.0094; R^2 = 0.9959). The results were expressed as mg gallic acid equivalent (GAE) g^{-1} pitaya peel (mg g^{-1}).

2.4.4. Betacyanin

The content of total betacyanin was determined by measuring the absorbance of the extracts at 535.5 nm in a spectrophotometer (Hach, model DR 4000U, São Paulo, SP, Brazil). The content of betacyanin was calculated according to **Eq. (1).**

$$\text{Betacyanin (g g}^{-1}\text{)} = \frac{A \times MM \times DF}{\varepsilon \times l} \quad (1)$$

where A is the absorbance of the extract; MM is the molecular mass of betacyanin (550 g mol^{-1}); DF is the dilution factor used for the measurement of the absorbance; ε is the molar extinction coefficient (60,000 L mol^{-1} cm^{-1}) of betacyanin; and l is the optical path length of the cuvette (1 cm).

2.4.5. Phenolic compounds by LC–MS/MS

Phenolic compounds were analysed by high resolution/accurate mass (HRAM) LC–MS/MS using a Shimadzu Prominence HPLC coupled to a ThermoFisher Scientific LTQ-Orbitrap Velos mass spectrometer. The phenolic compounds were separated using a Phenomenex Synergi C18AQ column (2.0×150 mm, 4-micron particle size, 80 Angstrom pore size) with a gradient of 5% to 80% acetonitrile over 20 min at a flow rate of 200 µL min^{-1}. Phenolic compounds were detected using negative ion mode electrospray ionization with the Orbitrap analyzer set to 60,000 resolution. The phenolic compounds were targeted for higher-energy collisional dissociation

(HCD) MS/MS fragmentation triggered by an inclusion list of known compound masses and compound identifications by comparison of retention times, accurate mass MS data, and MS/MS fragmentation profiles against those of authentic standards. The chromatographic peak alignment, peak area quantification, and identification of phenolic compounds were performed using El-MAVEN software (Elucidata). The quantification was calculated using standard curves obtained from commercially available phenolic compound standards.

2.4.6 Antioxidant activity by DPPH and FRAP assays

The *in vitro* antioxidant activity was determined by the inhibition of DPPH free radicals (Brand-Williams et al., 1995). Initially, 150 µL of the samples were reacted with 5850 µL of DPPH solution (0.06 mmol L^{-1}). After 30 min, the absorbance was measured at 515 nm in a spectrophotometer (Hach, model DR 4000U, São Paulo, SP, Brazil). Trolox was used as the standard ($y = 0.0017x + 0.8518$; $R^2 = 0.9823$). The results were expressed as µg Trolox equivalent antioxidant capacity (TEAC) g^{-1} pitaya peel (µg g^{-1}).

The antioxidant activity by the ferric reducing antioxidant power (FRAP) assay was conducted according to Benzie and Strain (1996), with modifications. The reaction was composed of 100 µL of the sample, 100 µL of ferric chloride (3 mmol L^{-1}), and 1800 µL of the TPTZ (2,4,6-Tris(2-pyridyl)-1,3,5-triazine) solution. The samples were kept at 37 °C for 30 min. The absorbance was measured in a spectrophotometer (Hach, model DR 4000U, São Paulo, SP, Brazil) at 620 nm. Trolox was used as the standard ($y = 0.0021x + 0.0644$; $R^2 = 0.9923$). The results were expressed as µg Trolox equivalent antioxidant capacity (TEAC) g^{-1} pitaya peel (µg g^{-1}).

2.4.7. Total reducing sugars

The reducing sugars were analyzed according to the Somogyi-Nelson method (Nelson, 1944). Prior to the analysis, the samples (extracts and hydrolysates) were subjected to acid hydrolysis (HCl, 2 mol L^{-1}) at 95 °C for 1 h and further neutralized with NaOH (2 mol L^{-1}). The samples were submitted to the reaction by adding the Somogyi-Nelson I and II reagents, followed by heating and cooling according to the conventional method. The calibration curve of glucose (y = 310.43x + 2.93; R^2 = 0.9846) was adopted to calculate the total reducing sugar content. The results were expressed as mg glucose g^{-1} pitaya peel (mg g^{-1}).

2.4.8. Maillard reaction products

The possible formation of Maillard reaction products during the extraction and hydrolysis processes was analyzed by measuring the absorbance at 294 (intermediate state of Maillard reaction products) and 420 nm (final stage of Maillard reaction products) (Rodrigues et al., 2021).

2.4.9. Organic acids

The organic acids were evaluated by high-performance liquid chromatography (HPLC) with a refractive index detector (RID). The separation was performed by a Rezex column (Phenomenex, model ROA-Organic Acid H+ (8%), 8 µm, 300×7.8 mm, Torrance, CA, USA), adopting an isocratic flow rate of 0.6 mL min^{-1} of H_2SO_4 (5 mmol L^{-1}) at 60 °C. The RID was adjusted to 40 °C. The extracts and hydrolysates were centrifuged (10,000×g, 15 min) and filtered (0.22 µm nylon filter). Then, 10 µL of the sample was injected and run for 50 min. The concentrations of oxalic acid, citric acid, malic acid, formic acid, and acetic acid were calculated according to the calibration curves of each standard. The results were expressed as mg g^{-1}.

2.5. Calculations

The yield of each compound produced during the extraction and hydrolysis was calculated according to **Eq. (2),** considering the amount of each compound, the total mass of pitaya peel used in the reactor, and the solvent-to-feed ratio (S/F, g water g^{-1} pitaya peel) employed throughout the kinetics.

$$\text{Yield}\left(\frac{\text{mg}}{\text{g}}\right) = \frac{\text{Compound produced}}{\text{Initial mass pitaya}} \times \frac{\text{S}}{\text{F}} \qquad (2)$$

2.6. Statistical analysis

All analyses were conducted at least in triplicate (n=3). Analysis of variance (ANOVA) was used to verify statistically significant values and the relationship between the variables. Tukey's test evaluated significant differences between samples ($p \leq 0.05$). The statistical analysis was performed using Statistica software (version 10.0, StatSoft Inc., Tulsa, OK, USA).

3. Results and discussion

3.1. Raw material characterization

Table 1 shows the physicochemical characterization of pitaya peel. The values obtained for neutral detergent fiber (34.04 g 100 g^{-1}) and protein (5.04 g 100 g^{-1}) are in agreement with those found for pitaya shell flour (Matra et al., 2019). The moisture of dried pitaya peel was 11.63 g 100 g^{-1}, and the total lipids obtained was 0.85 g 100 g^{-1}. These values are similar to those obtained by Utpott et al. (2020), 12.38 g 100 g^{-1} for moisture and 1.31 g 100 g^{-1} for total lipids. The concentrations of total reducing sugars (3.01 ± 0.11 g 100 g^{-1}) and total sugars (14.04 ± 0.45 g 100 g^{-1}) found in this study were similar to those in the literature (De Mello et al., 2014; Dos Santos et al., 2017), which when analyzing different drying temperatures verified the reduction in sugar content with increasing temperature. The pitaya peel presented high acidity (14.57 ± 0.15 g malic acid 100 g^{-1}), where the composition can be influenced by several factors, such as the place of cultivation, ripening time, and species (Franco et al., 2022; Hua et al., 2018; Lee et al., 2014).

Table 1. Composition of raw pitaya peel (dry basis).

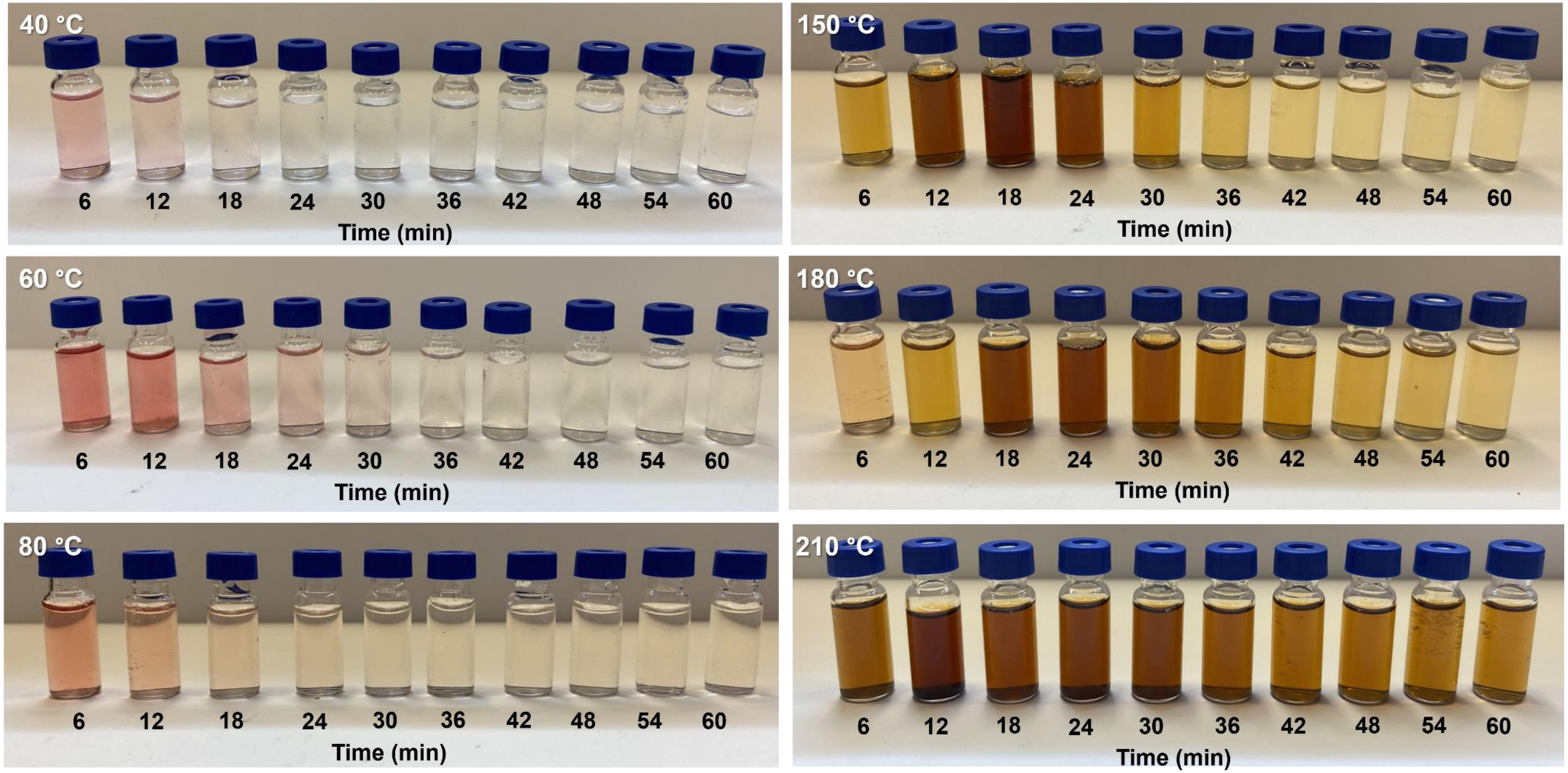
Parameters	Results	Unit
Moisture	11.63 ± 0.44	g 100 g^{-1}
Lipids	0.85 ± 0.02	g 100 g^{-1}
Proteins	5.04 ± 0.21	g 100 g^{-1}
Fibra bruta	23.48 ± 0.11	g 100 g^{-1}
Neutral detergent fiber	34.04 ± 0.41	g 100 g^{-1}
Acid detergent fiber	33.64 ± 0.52	g 100 g^{-1}
Lignin	26.44 ± 0.35	g 100 g^{-1}
Hemicellulose	0.42 ± 0.01	g 100 g^{-1}
Cellulose	7.19 ± 0.04	g 100 g^{-1}
Starch	6.46 ± 0.12	g 100 g^{-1}
Total reducing sugar	3.01 ± 0.11	g glucose 100 g^{-1}
Total sugar	14.04 ± 0.45	g glucose 100 g^{-1}
Total acidity	14.57 ± 0.15	g malic acid 100 g^{-1}

The results are expressed as the mean ± standard deviation. Analysis conducted in triplicate (n=3).

3.2. Visual appearance and color parameters

Figure 2 shows the visual aspect of the extracts and hydrolysates obtained during the semi-continous high-pressure hydrothermal process. In the treatments with lower temperatures (40, 60, and 80 °C), the extraction biocompounds can be observed in the first 20 min, where pink coloration is the main characteristic of the presence of bioactive compounds. The treatment at 60 °C had the highest yield of betacyanins and at same time showed a saturated coloration of the extract at 6 and 12 min, indicating a possible greater recovery. The extracts obtained from 30 min presented a lower color intensity, which can be associated with the end of the extraction process. Similar results were observed in pressurized liquid extraction coupled in-line with solid-phase extraction of açaí (Maciel-Silva et al., 2022) and subcritical water hydrolysis from rosemary plants (Sánchez-Camargo et al., 2019).

Figure 2. Visual appearance of the extracts and hydrolysates obtained from the semi-continous high-pressure hydrothermal process of pitaya peel.



Evaluating the treatments focused on hydrolysis (150, 180, and 210 °C), it is possible to observe a darker and brownish coloration. This darker coloration is characteristic of sugar hydrolysates, reflecting the process of caramelization and Maillard reaction products (Hemmler et al., 2018). It is also possible to observe that the most intense colors can be associated with the hydrolysates with the highest production of reducing sugars. The experiment at 150 °C showed higher production of sugars at 12 to 24 min. The experiment at 210 °C demonstrated that the highest production of sugars was obtained from 12 to 36 min. Similar results were observed in the subcritical water hydrolysis of jabuticaba peel (Barroso et al., 2022a) and brewer's spent grains (Sganzerla et al., 2022).

The CIELab color parameters (L*, a*, b* and C*) were evaluated in the extracts and hydrolysates obtained during the semi-continuous high-pressure hydrothermal process (**Table 2**). The extraction process (40, 60 and 80 °C) resulted in L* values ranging between 80.53 and 102, demonstrating that the samples presented high luminosity. For the 30 and 36 minute time intervals, the extracts at all three temperatures exhibited values that did not differ significantly. The same trend was observed for the readings obtained between 42 and 60 minutes at temperatures of 40 and 80 ºC. However, when comparing the obtained readings, it was found that the temperature of 60 ºC showed the most significant difference in results compared to the other temperatures.

Table 2. Color parameters of the extracts and hydrolysates obtained from the semi-continuous high-pressure hydrothermal process of pitaya peel.

Parameters	40 °C	60 °C	80 °C	150 °C	180 °C	210 °C
*L**						
6 min	86.79 ± 0.76^{bc}	90.97 ± 0.05^{b}	80.53 ± 0.37^{cd}	76.48 ± 1.26^{d}	82.56 ± 2.40^{cd}	57.12 ± 1.88^{a}
12 min	95.60 ± 1.13^{c}	81.50 ± 2.41^{de}	89.17 ± 1.63^{cd}	55.55 ± 0.22^{a}	76.18 ± 1.34^{e}	41.21 ± 2.77^{b}
18 min	90.02 ± 0.04^{a}	82.45 ± 0.57^{b}	89.44 ± 1.15^{ab}	45.30 ± 1.45^{d}	54.01 ± 2.54^{c}	51.77 ± 0.01^{cd}
24 min	96.29 ± 0.81^{a}	87.35 ± 0.13^{ab}	91.37 ± 1.60^{b}	55.36 ± 1.94^{c}	60.01 ± 1.79^{c}	55.10 ± 0.05^{c}
30 min	92.06 ± 0.29^{c}	88.88 ± 3.54^{c}	87.34 ± 0.95^{c}	75.86 ± 2.20^{a}	65.57 ± 1.84^{ab}	58.42 ± 1.40^{b}
36 min	90.80 ± 1.20^{c}	92.39 ± 1.20^{c}	91.32 ± 1.25^{c}	74.44 ± 1.62^{a}	64.37 ± 1.85^{ab}	60.28 ± 2.09^{b}
42 min	88.73 ± 1.31^{c}	102.01 ± 0.40^{a}	89.86 ± 1.45^{c}	84.75 ± 4.03^{c}	65.55 ± 1.85^{b}	65.40 ± 0.30^{b}
48 min	87.74 ± 0.63^{d}	102.64 ± 2.01^{a}	91.95 ± 1.76^{d}	84.55 ± 0.59^{d}	74.37 ± 2.75^{b}	60.86 ± 2.04^{c}
54 min	80.45 ± 1.09^{c}	101.30 ± 1.40^{a}	87.23 ± 3.50^{c}	84.91 ± 2.56^{c}	77.67 ± 0.32^{c}	65.43 ± 1.59^{b}
60 min	86.03 ± 1.56^{d}	98.97 ± 0.83^{a}	91.21 ± 0.31^{d}	88.62 ± 0.93^{d}	79.63 ± 0.67^{b}	71.51 ± 0.98^{c}
*a**						
6 min	4.53 ± 0.06^{b}	13.06 ± 0.09^{a}	6.45 ± 0.06^{e}	1.72 ± 0.01^{d}	2.27 ± 0.04^{c}	6.68 ± 0.20^{e}
12 min	2.63 ± 0.06^{cd}	10.95 ± 0.19^{b}	3.67 ± 0.16^{c}	10.58 ± 0.40^{b}	1.65 ± 0.05^{d}	19.48 ± 0.06^{a}
18 min	1.08 ± 0.02^{e}	5.61 ± 0.14^{d}	1.63 ± 0.02^{e}	17.21 ± 0.43^{a}	11.94 ± 0.36^{b}	10.56 ± 0.12^{c}
24 min	0.36 ± 0.01^{d}	2.95 ± 0.00^{b}	0.86 ± 0.01^{d}	11.54 ± 0.28^{c}	14.73 ± 0.61^{a}	10.41 ± 0.21^{c}
30 min	0.11 ± 0.02^{d}	1.56 ± 0.05^{c}	0.45 ± 0.01^{cd}	3.03 ± 0.01^{a}	9.71 ± 0.49^{b}	9.75 ± 0.04^{b}
36 min	0.08 ± 0.01^{c}	0.85 ± 0.01^{d}	0.29 ± 0.02^{cd}	0.84 ± 0.05^{d}	6.36 ± 0.20^{b}	8.37 ± 0.24^{a}
42 min	0.09 ± 0.01^{cd}	0.53 ± 0.01^{c}	0.28 ± 0.02^{cd}	-0.04 ± 0.01^{d}	3.45 ± 0.14^{b}	6.53 ± 0.14^{a}
48 min	0.04 ± 0.02^{e}	0.35 ± 0.07^{d}	0.14 ± 0.02^{de}	-0.25 ± 0.02^{c}	1.67 ± 0.01^{b}	6.69 ± 0.08^{a}
54 min	0.03 ± 0.01^{c}	0.22 ± 0.02^{e}	0.26 ± 0.01^{e}	-0.30 ± 0.00^{d}	1.03 ± 0.02^{b}	5.02 ± 0.02^{a}
60 min	0.02 ± 0.01^{d}	0.19 ± 0.01^{d}	0.21 ± 0.02^{d}	-0.33 ± 0.03^{c}	0.43 ± 0.03^{b}	5.00 ± 0.07^{a}
*b**						
6 min	3.21 ± 0.06^{c}	8.70 ± 0.08^{d}	8.77 ± 0.04^{d}	27.29 ± 0.16^{b}	9.54 ± 0.28^{d}	38.85 ± 0.66^{a}
12 min	2.76 ± 0.11^{d}	6.87 ± 0.25^{d}	7.35 ± 0.16^{d}	54.23 ± 1.25^{b}	27.70 ± 0.50^{c}	66.07 ± 2.94^{a}
18 min	1.98 ± 0.01^{e}	4.23 ± 0.09^{de}	4.86 ± 0.10^{d}	66.83 ± 0.20^{a}	58.32 ± 1.01^{b}	54.28 ± 0.57^{c}
24 min	1.95 ± 0.10^{c}	3.34 ± 0.02^{c}	3.61 ± 0.09^{c}	57.95 ± 0.79^{b}	66.85 ± 1.85^{a}	53.07 ± 0.85^{b}
30 min	1.42 ± 0.02^{d}	2.64 ± 0.22^{d}	3.08 ± 0.04^{d}	30.29 ± 0.46^{c}	54.98 ± 1.82^{a}	50.14 ± 0.25^{b}
36 min	1.24 ± 0.05^{d}	2.31 ± 0.05^{d}	3.08 ± 0.09^{d}	17.65 ± 0.15^{c}	42.61 ± 0.72^{b}	46.78 ± 1.18^{a}
42 min	0.92 ± 0.09^{d}	2.71 ± 0.02^{e}	2.75 ± 0.12^{e}	11.95 ± 0.58^{c}	29.88 ± 0.22^{b}	41.06 ± 0.43^{a}
48 min	0.72 ± 0.06^{d}	2.62 ± 0.19^{e}	2.83 ± 0.20^{e}	9.95 ± 0.05^{c}	21.07 ± 0.42^{b}	40.40 ± 0.05^{a}
54 min	0.41 ± 0.03^{b}	2.28 ± 0.05^{b}	3.28 ± 0.58^{b}	8.56 ± 0.14^{b}	8.90 ± 7.89^{b}	33.79 ± 0.29^{a}
60 min	0.43 ± 0.08^{d}	1.99 ± 0.06^{e}	2.83 ± 0.01^{e}	7.88 ± 0.17^{c}	11.58 ± 0.19^{b}	34.09 ± 0.27^{a}
*C**						
6 min	5.55 ± 0.08^{d}	15.69 ± 0.12^{c}	10.88 ± 0.06^{e}	27.34 ± 0.16^{e}	9.81 ± 0.28^{b}	39.42 ± 0.69^{a}
12 min	3.81 ± 0.12^{e}	12.92 ± 0.29^{d}	8.22 ± 0.21^{de}	55.25 ± 1.30^{b}	27.74 ± 0.50^{c}	68.89 ± 2.80^{a}
18 min	2.25 ± 0.01^{e}	7.03 ± 0.17^{d}	5.12 ± 0.10^{de}	69.01 ± 0.30^{a}	59.53 ± 1.06^{b}	55.29 ± 0.59^{c}
24 min	1.98 ± 0.10^{c}	4.45 ± 0.01^{c}	3.71 ± 0.09^{c}	59.08 ± 0.82^{b}	68.45 ± 1.94^{a}	54.08 ± 0.87^{b}
30 min	1.42 ± 0.02^{d}	3.06 ± 0.21^{d}	3.11 ± 0.04^{d}	30.44 ± 0.46^{c}	55.83 ± 1.87^{a}	51.07 ± 0.26^{b}
36 min	1.24 ± 0.05^{d}	2.46 ± 0.04^{d}	3.09 ± 0.09^{d}	17.67 ± 0.15^{c}	43.08 ± 0.74^{b}	47.52 ± 1.20^{a}
42 min	0.92 ± 0.09^{d}	2.76 ± 0.03^{e}	2.76 ± 0.12^{e}	11.95 ± 0.58^{c}	30.08 ± 0.23^{b}	41.58 ± 0.45^{a}
48 min	0.72 ± 0.06^{d}	2.64 ± 0.19^{e}	2.83 ± 0.20^{e}	9.95 ± 0.05^{c}	21.14 ± 0.42^{b}	40.95 ± 0.04^{a}
54 min	0.41 ± 0.03^{b}	2.29 ± 0.05^{b}	3.29 ± 0.57^{b}	8.57 ± 0.14^{b}	9.13 ± 7.68^{b}	34.16 ± 0.29^{a}
60 min	0.43 ± 0.08^{d}	1.99 ± 0.07^{e}	2.83 ± 0.02^{e}	7.88 ± 0.16^{c}	11.58 ± 0.19^{b}	34.45 ± 0.28^{a}

The results are expressed as the mean ± standard deviation. Analysis conducted in triplicate (n=3). Different letters in each line indicate significant differences by Tukey's test at $p \leq 0.05$.

The hydrolysis process (150, 180 and 210 °C) resulted in darker hydrolysates with L* ranging from 41.21 to 88, demonstrating that hydrolysis products were obtained. The extracts obtained at 24 minutes did not show significant differences at the three temperatures. Similarly, at 54 minutes, there were no significant differences in the extracts at temperatures of 150 and 180. However, in the remaining readings, the values were found to be significantly different based on Tukey's analysis. For the color parameter a*, which describes the variation between green and red, the highest values were obtained in the extraction conditions, demonstrating that the extract presented a reddish coloration, corroborating the visual appearance and with the possible presence of betacyanins. Considering the extraction kinetics, the highest values were obtained at 6 min in the process operated at 60 °C, indicating the possible presence of betacyanins (Chew et al., 2019). According to the statistical analysis, no significant differences were observed between the temperatures of 40 and 80 ºC at the time points of 18 and 24 minutes. Similarly, no significant differences were found between 60 and 80 ºC at 54 minutes, as well as between all three temperatures at 60 minutes. However, significant differences were observed in the other readings, as determined by Tukey's analysis. For the hydrolysates obtained at 180 and 210 °C, there is an increase in the color parameter a* during the hydrolysis kinetics, indicating a possible formation of Maillard reaction products due to the high temperatures (Coimbra et al., 2011; Murata, 2021). Among the results obtained, only the reading taken from the extracts at 30 minutes and temperatures of 180 and 210 ºC did not show a significant difference

The b* values for the hydrolysis temperatures were higher than the extraction temperatures. The results demonstrate that the hydrolysates present a yellowish coloration, which is the main characteristic of the hydrolysis process. The results obtained for the extraction temperatures were consistent. At 18 minutes, all extraction temperatures showed values with significant differences. However, at 60 and 80 ºC, no significant differences were observed for the other time intervals. For the hydrolysis temperatures, similar values were obtained at 24 minutes for temperatures of

150 and 210 ºC, as well as for temperatures of 150 and 180 ºC at 54 minutes. All other results were considered different. The parameter C* is related to the scale of gray coloration, and the use of high temperature (150, 180 and 210 °C) promoted the highest values, which is associated with the hydrolysis reactions. In this parameter, the extraction temperatures exhibited different values at the time intervals of 6, 12, and 18 minutes. However, no significant difference was found between the temperatures of 60 and 80 ºC in the other readings. Regarding the hydrolysis temperatures, similar values were obtained between the temperatures of 150 and 210 ºC at 24 minutes, as well as between temperatures of 150 and 180 ºC at 54 minutes. All other results were considered different.

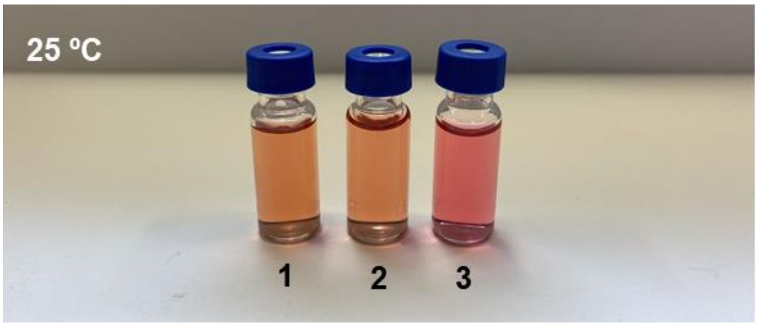
The L* parameter for the conventional extracts exhibited similar values (Table 3). The treatments of mechanical extraction (pH 7) and ultrasound-assisted extraction showed no significant difference in the values of parameters a* and b*. However, for the parameter C*, the ultrasound-assisted extraction treatment was the only one that demonstrated a significant difference compared to the other treatments. These results are guaranteed, as evidenced by the visual aspect shown in **Figure 3**. It is possible to observe that the ultrasound-assisted extraction exhibited a similar coloration to that achieved through mechanical extraction at pH 7, while both differed from the coloration obtained through mechanical extraction at pH 2. Compared with the results obtained by conventional extraction, it is possible to observe that the L* of the conventional extracts was lower than that of the extracts obtained by the high-pressure hydrothermal process. For the color parameters a*, b*, and C*, the conventional extracts promoted a high-intensity extract when compared with the high-pressure hydrothermal process, which can be mainly associated with the fact that the conventional extracts were performed under batch mode. The semi-continous process has the tendency to dilute the product during extraction. In addition, different parameters can affect the color parameters; however, temperature is one of the most significant, requiring a deep investigation on the presence of pigments (e.g., betacyanins) and the occurrence of Maillard reaction products under hydrolysis conditions.

Table 3. Summary of the results obtained by applying conventional extraction techniques to pitaya peel.

Parameters	Mechanical extraction (pH 2)	Ultrasound-assisted extraction	Mechanical extraction (pH 7)	Unit
Reducing sugars	14.36 ± 0.01[b]	13.79 ± 0.41[b]	12.34 ± 0.01[a]	mg g^{-1}
Phenolic compounds	7.25 ± 0.094[ab]	6.91 ± 0.079[b]	7.63 ± 0.18[a]	mg GAE g^{-1}
Malic acid	43.35 ± 0.25[a]	39.58 ± 0.09[c]	42.17 ± 0.27[b]	mg g^{-1}
Citric acid	24.22 ± 0.15[a]	11.05 ± 0.36[c]	19.91 ± 0.23[b]	mg g^{-1}
pH	4.52	5.59	5.52	-
Antioxidant activity				
DPPH	7.81 ± 0.04[b]	8.59 ± 0.03[a]	7.07 ± 0.01[c]	µg TEAC g^{-1}
FRAP	9.59 ± 0.01[b]	9.58 ± 0.13 [b]	10.22 ± 0.07 [a]	µg TEAC g^{-1}
Color parameters				
L*	63.52 ± 0.48[a]	68.94 ± 0.45[a]	71.76 ± 2.86[a]	-
a*	16.55 ± 0.07[a]	10.22 ± 0.03[b]	9.91 ± 0.22[b]	-
b*	6.00 ± 0.02[a]	15.92 ± 0.03[b]	17.25 ± 0.45[b]	-
C*	17.60 ± 0.07[a]	18.91 ± 0.04[ab]	19.89 ± 0.49[a]	-
Mailard reaction products				
Absorbance at 294 nm	5.60[b]	5.67[a]	4.12[c]	-
Absorbance at 420 nm	0.23[c]	0.63[a]	0.47[b]	-

The results are expressed as the mean ± standard deviation. Analysis conducted in triplicate (n=3). Different letters in each line indicate significant differences by Tukey’s test at $p \leq 0.05$.

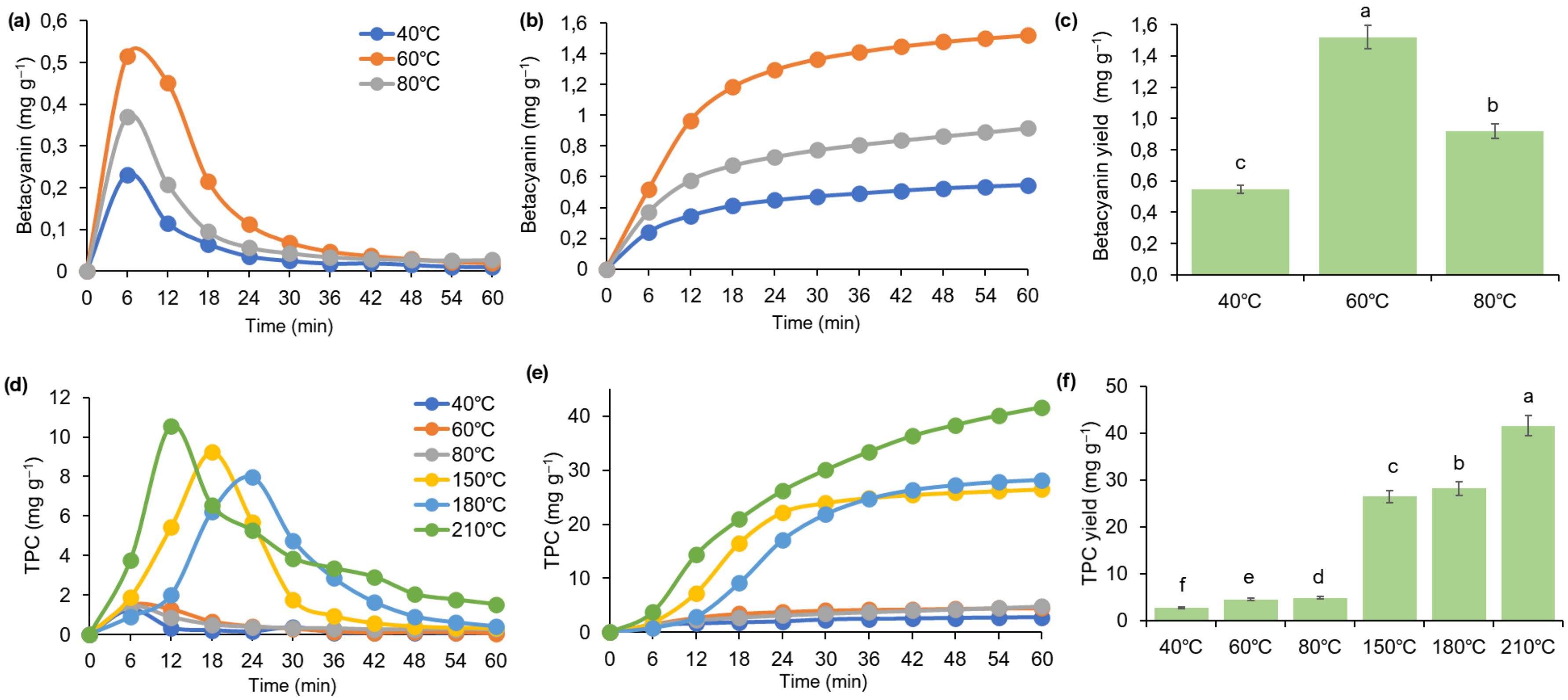
Figure 3. Visual appearance of the extracts obtained from applying conventional extraction techniques to pitaya peel. 1) Mechanical extraction (pH 7) 2) Ultrasound-assisted extraction 3) Mechanical extraction (pH 2)



3.3. Betacyanin

During the semi-continuous high-pressure hydrothermal process of pitaya peel, only the extraction conditions were favorable to the recovery of betacyanin (**Figure 4**). These results can be associated with the fact that betacyanin is a thermolabile compound and is easily degraded when exposed to high temperatures (Rodriguez-Amaya, 2019). The highest recovery of betacyanin was obtained at 60 °C (1.52 mg g^{-1}), followed by 80 (0.92 mg g^{-1}) and 40 °C (0.55 mg g^{-1}). Hence, the extraction was effective up to 60 °C, and at higher temperatures, the betacyanins were degraded. Similar results were reported for the production of betacyanins with ultrasonic-assisted extraction over a temperature range from 30 to 70 °C, where the highest production was achieved at 60 °C with a yield of 1.56 mg g^{-1} (Bhagya Raj and Dash, 2020). In addition, the results obtained in this study were similar when compared with other extraction methods, such as microwave-assisted extraction (1.66 mg g^{-1}) (Ferreres et al., 2017), and ultrasound-assisted extraction (1.61 mg g^{-1}) (Bhagya Raj and Dash, 2020). The betalain content is influenced by several factors, such as the part of the fruit analyzed, peel, or pulp (Ferreira et al., 2023). The ripening period that the fruit was harvested, since the concentration of betalains increases according to maturation (Wu et al., 2019). Other important factors are the place of planting and the type of pitaya (Jiang et al., 2021). The semi-continuous process was demonstrated as a promising approach to producing betacyanins. However, several factors can affect the yield of this compound, such as the mode of extraction (batch or continuous), temperature, extraction time, storage of extract, and detection method (Leong et al., 2018). The results obtained demonstrate the effectiveness of the extraction method in obtaining betacyanins compared to other extractive methods, which can be considered a sustainable approach to the production of betacyanins from pitaya peel.

Figure 4. Kinetic performance of betacyanin and phenolic compounds obtained from the semi-continous high-pressure hydrothermal process of pitaya peel. (a) betacyanin; (b) betacyanin accumulated; (c) betacyanin yield; (d) TPC; (e) TPC accumulated; (f) TPC yield. Label: Different letters indicate significant differences by Tukey's test at $p \leq 0.05$.



3.5. Phenolic compounds

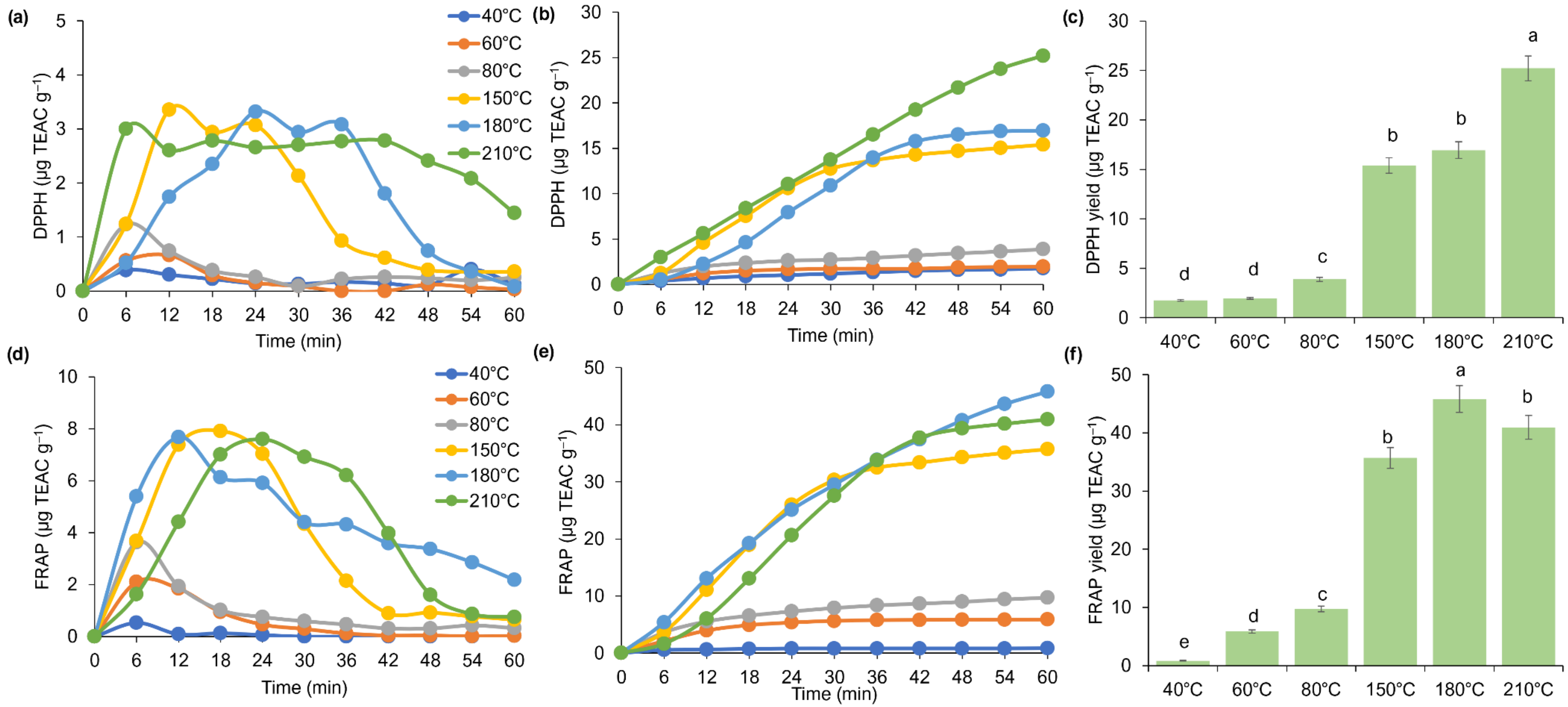
Phenolic compounds are secondary metabolites widely obtained in plants and their by-products (Alara et al., 2021). Phenolic compounds act in several metabolic processes against several types of adversities, such as ultraviolet rays, predators, and pathogens, and are also responsible for the organoleptic characteristics of vegetables (Alara et al., 2021). Studies on the extraction of phenolic compounds are increasing due to their biological potential, such as antioxidant, anti-inflammatory, and antimicrobial activities. The results obtained during the semi-continuous high-pressure hydrothermal process of pitaya peel demonstrate that the content of phenolic compounds varied proportionally with increasing temperature (**Figure 4**), ranging from 2.75 to 41.71 mg g^{-1}. The concentration of phenolic compounds was proportional to the increase in temperature. The kinetic behavior of phenolic compounds revealed that the first 30 min of the extraction was responsible for the recovery of approximately 70% at 210 °C and 90% at 150 °C. Similar parameters were reported by Alasalvar et al. (2023) when extracting phenolic compounds from lemon peel using semi-continous high-pressure hydrothermal process, which reported an optimal process time of 30 min and 160 °C. Extraction with a high-pressure hydrothermal process was more efficient than conventional methods, which can be explained by the better diffusion and penetration effects of the extracting solvent in the plant matrix (Rampelotto de Azevedo et al., 2022). Notwithstanding, high temperatures favored the extraction of phenolic compounds, especially because these compounds are covalently linked to sugars or structural components of the plant wall, requiring hydrolysis conditions for the solvent to extract the phenolic compounds (Tang et al., 2021). When comparing the results obtained with other studies, the use of high-pressure hydrothermal allowed better recovery of phenolic compounds than acidic, alkaline, ultrasound-assisted, and methanol hydrolysis, obtaining values up to 50-fold lower (Can-Cauich et al., 2017; Uslu and Özcan, 2021; Zhong et al., 2022).

The profile of individual phenolic compounds was analyzed by LC–MS/MS in the extract obtained at 60 °C. In this condition, the phenolic compounds are associated with polyphenols extracted from the matrix.

3.6. Antioxidant activity

Figure 5 shows the antioxidant activity by the FRAP and DPPH assays of the extracts and hydrolysates obtained from the semi-continuous high-pressure hydrothermal process of pitaya peel. The antioxidant activity was directly associated with the temperature used in the process. It is possible that hydrolysis conditions (150, 180, and 210 °C) promoted the highest antioxidant activity due to the presence of total phenolic compounds. The antioxidant activity of the conventional extracts was higher than that of the extracts obtained by the semi-continuous high-pressure hydrothermal process. This result is directly related to the content of bioactive compounds in the extracts. Under low temperatures, betacyanins are responsible for the high antioxidant activity. When comparing the results of the DPPH and FRAP assays, the FRAP method showed higher results, which can be attributed to the reduction of iron ions compared to the inhibition of DPPH free radicals (Angonese et al., 2021).

Figure 5. Antioxidant activity of the extracts and hydrolysates obtained from the semi-continuous high-pressure hydrothermal process of pitaya peel. (a) DPPH; (b) DPPH accumulated; (c) DPPH yield; (d) FRAP; (e) FRAP accumulated; (f) FRAP yield. Label: Different letters indicate significant differences by Tukey's test at $p \leq 0.05$.



In this study, 180 °C was the best temperature to recover phenolic compounds, which presented the highest antioxidant activity. This result confirms that the main mechanism of antioxidant activity of these compounds is accomplished through electron transference (Jimenez-Garcia et al., 2022b). The relationship between phenolic compounds and antioxidant activity has been observed in previous studies (Arruda et al., 2018; Tang et al., 2021). Another factor that may have influenced the antioxidant activity is the formation of Maillard reaction products, which have excellent antioxidant activity in different mechanisms of action (Feng et al., 2022; Shakoor et al., 2022). Compared with the literature, the antioxidant activity of the extracts obtained by the semi-continuous high-pressure hydrothermal process of pitaya peel presented similar results with alkaline hydrolysis (25.19 µg TEAC g^{-1}) (Tang et al., 2021). In addition, the use of acid hydrolysis (1.84 µg TEAC g^{-1}), enzymatic (1.08 µg TEAC g^{-1}), methanol (7.01 µg TEAC g^{-1}) (Tang et al., 2021), and ultrasound-assisted extraction (14.14 µg TEAC g^{-1}) (Zhong et al., 2022) showed lower results than those obtained in this study. Finally, the use of temperatures between 150 and 210 °C in the high-pressure hydrothermal process promoted the recovery of antioxidant hydrolysates, which is associated with the presence of phenolic compounds generated from Maillard reaction products.

3.7. Organic acids and pH

Table 4 shows the organic acids obtained during the semi-continuous high-pressure hydrothermal process of pitaya peel. Citric and malic acids were obtained at all temperatures (from 40 to 210 °C). The highest concentrations of citric (25.98 mg g^{-1}) and malic acids (25.6 mg g^{-1}) were obtained at 60 °C. This result was expected because citric and malic acids are the main organic acids reported in the composition of pitaya fruit (Hua et al., 2018). In the case of citric acid, it was possible to verify that the use of extraction temperatures (40, 60, and 80 °C) favored recovery, while under hydrolysis temperatures (150, 180, and 200 °C), there was a decrease in the content of malic and citric acids. The reduction in these organic acids can be explained by the formation of degradation products at high temperatures during the hydrolysis of pitaya peel. Malic acid can be degraded to fumaric acid at temperatures above 150 °C (Iyyappan et al., 2019), and citric acid can be decomposed at 175 °C (Behera, 2020). Both organic acids were detected in the conventional extracts by maceration and ultrasound-assisted extraction. For the semi-continuous high-pressure hydrothermal process, the malic acid values recovered were lower than those of the conventional extracts (**Table 3**). The concentrations of malic, citric, oxalic, and succinic acids are in agreement with the literature, demonstrating the presence of these organic acids in the peel and pulp of pitaya (Hua et al., 2018; Lee et al., 2014; Roriz et al., 2022).

Table 4. Production of organic acids obtained from the semi-continuous high-pressure hydrothermal process of pitaya peel at a solvent-to-feed ratio of 40 g water g^{-1} pitaya peel.

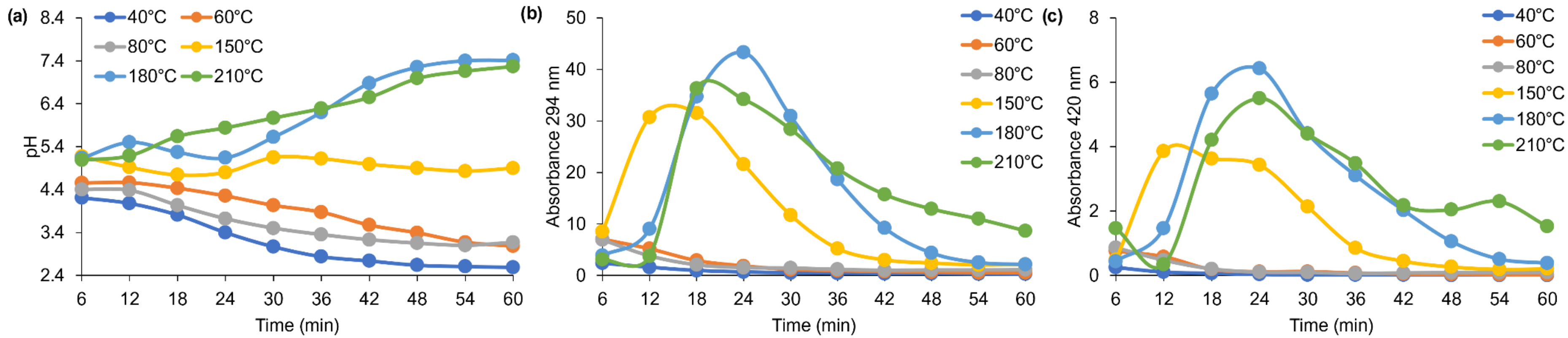
Parameters	**40 °C**	**60 °C**	**80 °C**	**150 °C**	**180 °C**	**210 °C**	**Unit**
Oxalic acid	n.d	11.72 ± 0.02[a]	25.32 ± 1.23[b]	n.d	n.d	n.d	mg g^{-1}
Citric acid	19.34 ± 0.20[a]	25.98 ± 0.81[b]	25.44 ± 1.23[b]	7.96 ± 0.97[c]	4.03 ± 0.01[c]	4.82 ± 1.17[c]	mg g^{-1}
Malic acid	8.96 ± 0.37[c]	25.60 ± 0.25[a]	19.50 ± 0.27[b]	10.59 ± 0.05[e]	10.52 ± 0.37[e]	5.15 ± 0.02[d]	mg g^{-1}
Succinic acid	n.d	n.d	n.d	n.d	n.d	5.02 ± 0.07	mg g^{-1}
Formic acid	n.d	n.d	n.d	n.d	n.d	53.78 ± 0.07	mg g^{-1}
Acetic acid	n.d	n.d	n.d	n.d	n.d	11.96 ± 0.82	mg g^{-1}
Total (mg g^{-1})	28.3 ± 0.16[c]	63.30 ± 0.56[c]	70.26 ± 2.71[b]	25.04 ± 0.37[c]	14.55 ± 0.38[d]	84.29 ± 2.04[a]	mg g^{-1}

The results are expressed as the mean ± standard deviation. Analysis conducted in triplicate (n=3). n.d., not detected. Different letters in each line indicate significant differences by Tukey's test at $p \leq 0.05$.

In the hydrolysates obtained at high temperature, formic acid was detected at a higher concentration (53.78 mg g^{-1}) and was detected only at 210 °C. The detection of this acid is directly related to glucose degradation, demonstrating that the hydrolysis reactions are directly associated with the temperature (Chen et al., 2019; Muharja et al., 2018). In addition, acetic acid was detected at 210 °C (11.96 ± 0.82 mg g^{-1}), demonstrating the degradation of hemicellulose products (Oliveira et al., 2020).

Figure 6 shows the pH of the extracts and hydrolysates obtained from the semi-continuous high-pressure hydrothermal process of pitaya peel. The pH varied between 4.21 and 5.17, similar to the concentrated extracts. The treatments at 40, 60, and 80 °C presented acidification during semi-continuous extraction, reaching a pH of 2.59 at the end of the process. These results can be associated with the extraction of organic acids, promoting a reduction in pH. Otherwise, the treatments with high temperatures increased pH values, where values of approximately 7.5 were obtained for 180 and 210 °C. This increase can be associated with the neutralization of organic acids and the presence of Maillard reaction products under hydrolysis conditions (Barroso et al., 2022). The possible explanation can be associated with the autoionization reaction of water and the degradation of amino acids at high temperatures and pressures, promoting the release of hydroxyl radicals into the medium and the formation of amines from the decomposition of glycine into methylamine and other amines (Ahmed and Chun, 2018; Jiang et al., 2021; Park et al., 2015).

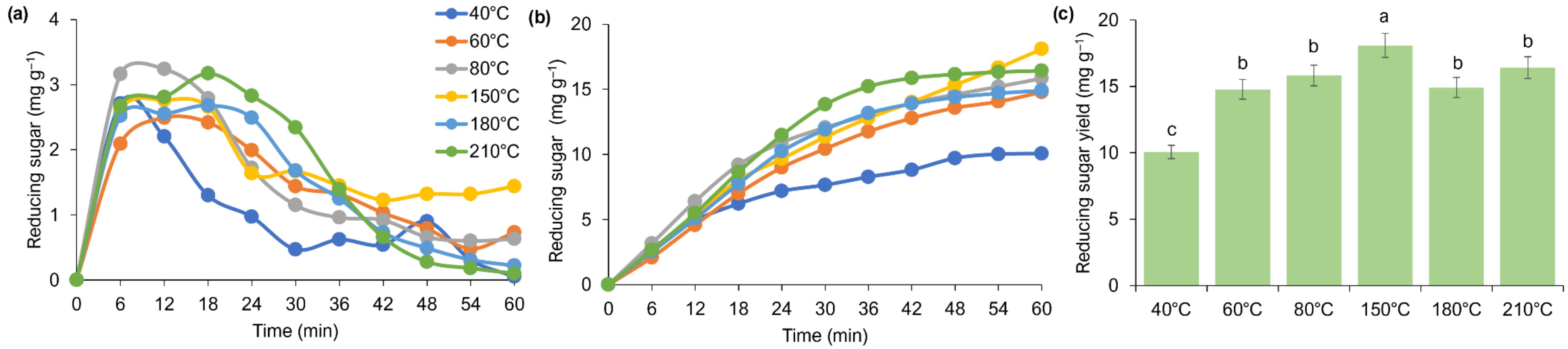
Figure 6. pH and Maillard reaction products of the extracts and hydrolysates obtained from the semi-continous high-pressure hydrothermal process of pitaya peel. (a) pH; (b) absorbance at 294 nm; (c) absorbance at 420 nm.



3.8. Sugar and Maillard reaction products

Figure 7 shows the total reducing sugars obtained from the semi-continuous high-pressure hydrothermal process of pitaya peel. The highest yields were obtained at the beginning of the process, indicating the possible presence of soluble sugars. The conventional extractions presented values between 12.34 and 14.36 mg g^{-1}, similar to the concentration of the extracts obtained at low temperatures. The highest yields of reducing sugars occurred in 12 min at 80 °C (3.24 mg g^{-1}) and 18 min at 210 °C (3.17 mg g^{-1}). The reducing sugar concentration continued to increase until reaching 18.09 mg g^{-1} at 150 °C and then decreased at 180 °C, increasing again at 210 °C. The behavior obtained for reducing sugars is related to the generation of Maillard reaction products (**Figure 6**). The Maillard reactions were indirectly quantified by measuring the absorbance at 294 (intermediate Maillard products) and 429 nm (final Maillard products). The results demonstrated that the Maillard reaction products were obtained only under hydrolysis conditions. The highest concentration was obtained at 24 min, indicating that the sugars produced were converted into inhibitors and Maillard reaction products. However, it was impossible to identify the formation of Maillard reaction intermediates, furfural compounds, using HPLC-RID, possibly due to the advanced stage of the Maillard reaction. This result was already expected since this reaction requires the presence of reducing sugars, and they decreased with increasing extraction time (Shakoor et al., 2022). The extractions carried out at 40, 60, and 80 °C showed much lower absorbances than those presented by the extracts at 150, 180, and 210 °C, demonstrating that temperature is one of the factors that significantly influence the occurrence of the Maillard reaction. Similar results were reported during the subcritical water hydrolysis of crab shells, where the absorbance of the hydrolysates increased with the temperature, indicating the formation of Maillard reaction products (Rodrigues et al., 2021). Finally, the use of temperatures above 180 °C promoted the reduction of the reducing sugar content and the increase in absorbances, demonstrating that these temperatures favored the occurrence of Maillard reaction products. This fact can be affirmed by comparing the conventional extracts and the extraction performed at low temperatures, where a lower absorbance was obtained, demonstrating the low concentration of Maillard reaction products.

Figure 7. Production of reducing sugars obtained from the semi-continuous semi-continuous high-pressure hydrothermal process of pitaya peel. (a) reducing sugar; (b) accumulated reducing sugar; (c) reducing sugar yield.



Label: Different letters indicate significant differences by Tukey's test at $p \leq 0.05$.

4. Conclusion

Semi-continous high-pressure hydrothermal process is an effective and sustainable approach for the recovery of value-added products from pitaya peel. The use of 60 °C in the semi-continuous process favored the recovery of malic acid (25.6 mg g^{-1}), citric acid (25.98 mg g^{-1}), and betacyanins (1.52 mg g^{-1}). The major phenolic compounds obtained at 60 °C were *p*-coumaric acid (1.44 ± 0.04 10^{-1} mg g^{-1}), protocatechuic acid (9.14 ± 0,03 10^{-2} mg g^{-1}), and piperonylic acid (7.42 ± 0.3 10^{-2} mg g^{-1}). The highest yield of reducing sugars (18.09 mg g^{-1}) was achieved at 150 °C. Under hydrolysis conditions, formic and acetic acids were produced due to the degradation of hemicellulosic sugars. The content of phenolic compounds increased with temperature, reaching the highest concentration at 210 °C (41.61 mg g^{-1}). The phenolic compounds at hydrolysis temperature above 150 °C resulted from the decomposition of sugars into Maillard reaction products, and this can be directly associated with the high antioxidant activity by FRAP (45.80 µg TEAC g^{-1}) and DPPH (25.22 µg TEAC g^{-1}) assays at temperatures above 180 °C. Finally, the use of semi-continous high-pressure hydrothermal process mode can be a sustainable and promising approach for the recovery of value-added compounds from pitaya peel, advocating a circular economy approach in the agri-food industry.

CRediT authorship contribution statement

Vanessa Cosme Ferreira: Methodology, Investigation, Analysis, Validation, Writing - original draft. **William Gustavo Sganzerla:** Conceptualization, Methodology, Investigation, Analysis, Validation, Writing - original draft, Writing - review & editing. **Tiago Linhares Cruz Tabosa Barroso:** Methodology, Investigation, Validation, Writing - review & editing **Luiz Eduardo Nochi Castro:** Investigation, Analysis, Writing - review & editing. **Leda Maria Saragiotto Colpini:** Conceptualization, Supervision, Resources, Project administration, Funding acquisition, Writing - review & editing. **Tânia Forster-Carneiro:** Supervision, Resources, Project administration, Funding acquisition, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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CAPÍTULO 4 – Discussão Geral

A valorização final e ambientalmente adequada de resíduos sólidos gerados (cascas) a partir do processamento de pitaia foi estudada nesta dissertação de mestrado. O tema está diretamente relacionado à necessidade do desenvolvimento de um processo eficaz de tratamento de resíduo sólido combinando a tecnologia de extração/hidrólise em água subcrítica. É importante ressaltar que ainda não existem pesquisas e aplicações industriais desenvolvidas a partir desta integração de sistemas de extração/hidrólise em água subcrítica sendo de grande interesse para promover a inovação frente às pesquisas brasileiras e mundiais. A seguir será apresentada uma discussão mais detalhada de cada capítulo.

4.1. Discussão sobre o Capítulo 2 - An updated review of recent applications and future perspectives on the sustainable valorization of pitaya (Hylocereus spp.) by-products

Uma análise bibliométrica foi realizada para verficar o desenvolvimento sustentável na indústria alimentar e possíveis implementação de biorrefinarias, defendendo uma bioeconomia circular relacionados com resíduos procedentes do processamento de pitaia. A revisão bibliométrica sobre o andamento da pesquisa, atualizações e tendências futuras de recuperação de produtos com maior valor agregado das cascas de pitaia no conceito de biorrefinaria foi realizada.

Os resultados indicam que, as pesquisas relacionadas à pitaia aumentaram significativamente ao longo dos anos. No período estudado, o maior número de publicações foi em 2021, com 159 artigos. Com o aumento das pesquisas, houve também mudanças nas áreas de interesse, evidenciando uma variação dos temas e a exploração de novas aplicações. As principais palavras-chave utilizadas incluem variações dos nomes da fruta e de seus compostos bioativos, indicando que muitos estudos publicados focaram na caracterização da fruta, sua composição e a extração de compostos de interesse. No diagrama gerado com os dados da pesquisa, temas relacionados à casca de pitaia e pectina foram identificados como emergentes, ganhando cada vez mais reconhecimento e sendo mais estudados. A maioria dos artigos mais citados aborda os compostos bioativos presentes na pitaia, como betalaínas, betacianinas e compostos antioxidantes, destacando a fruta como uma fonte promissora desses compostos.

A casca da pitaia pode representar até 40% do peso do fruto, sendo um dos principais subprodutos do seu processamento. A valorização da casca é de grande interesse, pois já foi comprovado que ela apresenta maiores teores de compostos fenólicos em comparação com a polpa, além de conter compostos como terpenoides, alcaloides, betanina, isobetanina, vitaminas, proteínas e fibras. Com a caracterização tanto da polpa quanto da casca, o interesse em extrair compostos de alto valor agregado tem crescido.

Os métodos de extração podem ser classificados como clássicos e modernos. Os métodos clássicos, amplamente validados, utilizam grandes volumes de solventes, muitas vezes tóxicos, demandando mais tempo e custos. Em contrapartida, os métodos modernos são eficientes e ecológicos. Ambos os tipos são usados para a extração de compostos valiosos, com a escolha do método dependendo do produto de interesse.Dentre os compostos extraídos da pitaia por métodos modernos estão pectina, betacianina, polifenóis e flavonoides, entre outros. Os extratos obtidos apresentam propriedades funcionais, antioxidantes, antimicrobianas e anticancerígenas. Esses extratos já encontram aplicação na indústria de alimentos, sendo incorporados em produtos como panificados e laticínios, além de serem utilizados em embalagens alimentares, como filmes e revestimentos comestíveis.

Aplicar o conceito de biorrefinaria à pitaia pode maximizar a utilização de todas as partes do fruto, reduzindo resíduos e aumentando o valor econômico dos subprodutos. A valorização integral da pitaia, por meio de métodos de extração modernos e sustentáveis, e a aplicação de seus compostos na indústria de alimentos, como ingredientes funcionais e embalagens comestíveis, exemplificam o potencial da biorrefinaria para criar cadeias de valor sustentáveis. Portanto, a integração das pesquisas atuais com o conceito de biorrefinaria pode contribuir significativamente para o desenvolvimento de soluções inovadoras e sustentáveis na utilização da pitaia e seus subprodutos.

4.2. Discussão sobre o Capítulo 3 - Sustainable valorization of pitaya (Hylocereus spp.) peel in a semi-continuous high-pressure hydrothermal process to recover value-added products

Os experimentos foram realizados em reator com água subcrítica a 15 Mpa de pressãovazão de água de 2 mL/min, relação solvente/alimentação de 60 g de água/g de casca de pitaia e temperaturas variando de 40 a 210 °C. Os resultados indicaram na caracterização inicial da casca de pitaia valores de fibra (34,04 g/100 g), proteína (5,04 g/100 g), umidade (11,63 g/100 g), lipídeos (0,85 g/100 g), açúcares redutores (3,01 ± 0,11 g/100 g) e açúcares totais (14,04 ± 0,45 g/100 g) semelhantes aos encontrados na literatura. A acidez (14,57 ± 0,15 g de ácido málico/100 g) foi superior aos valores relatados em outros estudos, possivelmente devido a variações no local de cultivo, tempo de maturação e espécie da pitaia. Durante o processo hidrotérmico semicontínuo de alta pressão, a extração de betacianina foi mais eficiente a 60 °C, com recuperação de 1,52 mg/g, seguida de 80 °C (0,92 mg/g) e 40 °C (0,55 mg/g). A betacianina é um composto termosensível, degradando-se em altas temperaturas. Estudos anteriores com extração assistida por ultrassom e micro-ondas também identificaram 60 °C como a temperatura ideal. Fatores como a parte da fruta analisada, maturação, local de plantio e tipo de pitaia influenciam o teor de betalaína. O processo semicontínuo demonstrou ser uma abordagem promissora e sustentável para a produção de betacianinas.

No processo hidrotérmico semicontínuo de alta pressão da casca de pitaia, o teor de compostos fenólicos variou de 2,75 a 41,71 mg/g, aumentando com a temperatura. Em 30 minutos, 70% dos compostos foram recuperados a 210 °C e 90% a 150 °C. Este método superou os convencionais devido à melhor difusão e penetração do solvente (Rampelotto de Azevedo et al., 2022). Análises por LC-MS/MS do extrato a 60 °C identificaram ácidos p-cumárico, protocatecuico e piperonílico como principais compostos fenólicos, entre outros produtos de valor agregado. A extração desses compostos é de grande interesse devido ao seu potencial antioxidante, antiinflamatório e antimicrobiano. A atividade antioxidante aumentou com a temperatura de hidrólise (150, 180 e 210 °C), diretamente relacionada à presença de compostos fenólicos. Os extratos convencionais apresentaram atividade antioxidante superior aos obtidos pelo processo hidrotérmico semicontínuo de alta pressão, com o método FRAP mostrando melhores resultados que o DPPH. A 180 °C, a recuperação de compostos fenólicos foi mais eficaz, confirmando que a transferência de elétrons é o principal mecanismo de atividade antioxidante. A formação de produtos da reação de Maillard também contribuiu para a alta atividade antioxidante. Comparado a outros métodos, a hidrólise hidrotérmica produziu melhores resultados antioxidantes, especialmente entre 150 e 210 °C.

Os ácidos cítrico e málico foram encontrados em todas as temperaturas durante o processo hidrotérmico semicontínuo de alta pressão, com maiores concentrações a 60 °C (25,98 mg/g e 25,6 mg/g, respectivamente). A extração a temperaturas mais baixas (40, 60, 80 °C) favoreceu a recuperação desses ácidos, enquanto temperaturas mais altas (150, 180, 210 °C) resultaram na sua degradação. O ácido fórmico foi detectado em maior concentração (53,78 mg/g) a 210 °C, indicando degradação de glicose. O pH dos extratos variou de 4,21 a 5,17, com acidificação a baixas temperaturas e aumento do pH em altas temperaturas, associado à neutralização de ácidos orgânicos e produtos da reação de Maillard. Estes resultados destacam a influência da temperatura na recuperação e degradação de ácidos orgânicos durante a hidrólise da casca da pitaia. Os maiores rendimentos de açúcares redutores foram alcançados no início do processo, sugerindo a presença de açúcares solúveis. As extrações convencionais variaram entre 12,34 e 14,36 mg/g, semelhantes aos extratos de baixas temperaturas. O maior rendimento de açúcares redutores foi em 12 min a 80 °C (3,24 mg/g) e 18 min a 210 °C (3,17 mg/g). A concentração aumentou até 150 °C (18,09 mg/g), diminuiu a 180 °C e aumentou novamente a 210 °C, relacionada à geração dos produtos da reação de Maillard. A formação destes produtos foi observada por aumento de absorbância a 294 e 429 nm em temperaturas altas. Este comportamento indica que temperaturas acima de 180 °C favorecem a reação de Maillard, resultando em menor teor de açúcares redutores e maior concentração de produtos de Maillard, ao contrário das extrações a baixas temperaturas.

CAPÍTULO 5 – Conclusão Geral

5.1. Conclusão sobre o Capítulo 2 - An updated review of recent applications and future perspectives on the sustainable valorization of pitaya (Hylocereus spp.) by-products

- A análise bibliométrica mostrou que a aplicação da polpa de pitaia utilizada na produção de alimentos são produção de geléias, sorvetes, iogurtes, pães e vinhos, possui compostos bioativos com propriedades anti-inflamatórias, antioxidantes, antimicrobianas e anticancerígenas;
- Pesquisas indicaram que, cerca de 33% do fruto da pitaia é gerado como subproduto durante o processamento agroindustrial, os quais são descartados de forma não sustentável, como em aterros sanitários ou como alimentação animal;
- Técnicas apropriadas podem converter as cascas descartadas em produtos de alto valor, promovendo a bioeconomia circular;
- Extratos da casca da pitaia exibem atividades antioxidante, antimicrobiana e anticancerígena. Além disso, podem ser incorporados em embalagens de alimentos para mitigar reações oxidativas, monitorar o frescor através de variações no pH e estender a vida útil de produtos perecíveis;
- A extração de compostos bioativos ainda é limitada a métodos convencionais;
- Métodos de extração sustentáveis como micro-ondas, ultrassom, líquidos pressurizados e fluidos supercríticos devem ser mais estudados para valorizar a casca da pitaia;
- Concluiu-se que, utilizar a casca da pitaia como matéria-prima para produtos de valor agregado pode reduzir os impactos ambientais, promover a economia circular e pode diminuir a pegada de carbono da indústria alimentícia.

5.2. Discussão sobre o Capítulo 3 - Sustainable valorization of pitaya (Hylocereus spp.) peel in a semi-continuous high-pressure hydrothermal process to recover value-added products

- O experimento com reator semicontínuo mostrou-se promissor e sustentável para a produção de betacianinas, sendo a extração de betacianina mais eficiente a 60 °C de temperatura e com recuperação de 1,52 mg/g;
- O teor de compostos fenólicos analisado variou de 2,75 a 41,71 mg/g, aumentando com a temperatura;
- A atividade antioxidante aumentou com a temperatura de hidrólise (150, 180 e 210 °C);
- A formação de produtos da reação de Maillard contribuiu significativamente para a alta atividade antioxidante;
- A maior concentração de ácidos cítrico e málico foram encontrados na temperatura de 60 °C (25,98 mg/g e 25,6 mg/g, respectivamente), temperautras mais altas (150, 180, 210 °C) resultaram na degradação destes ácidos;
- O ácido fórmico foi detectado em maior concentração a 210 °C, devido a maior degradação de glicose;
- O pH dos extratos variou de 4,21 a 5,17, com acidificação a baixas temperaturas e aumento do pH em altas temperaturas. O aumento do pH em altas temperaturas está associado à neutralização de ácidos orgânicos e produtos da reação de Maillard;
- Maiores rendimentos de açúcares redutores foram alcançados no início do processo, sugerindo a presença de açúcares solúveis. O maior rendimento de açúcares redutores foi em 12 min a 80 °C (3,24 mg/g) e 18 min a 210 °C (3,17 mg/g);
- A concentração de açúcares redutores aumentou até 150 °C, diminuiu a 180 °C e aumentou novamente a 210 °C, relacionada à geração dos produtos da reação de Maillard;
- A formação de produtos da reação de Maillard foi observada por aumento de absorbância a 294 e 429 nm em temperaturas altas;
- Temperaturas acima de 180 °C favoreceram a reação de Maillard, resultando em menor teor de açúcares redutores e maior concentração de produtos de Maillard;

- Conclui-se que, este método superou os convencionais devido à melhor difusão e penetração do solvente devido ao uso de processo hidrotérmico semicontínuo de alta pressão, sendo uma abordagem sustentável e promissora para recuperar compostos de alto valor da casca da pitaia, promovendo uma economia circular na indústria agroalimentar.

CAPÍTULO 6 – Referência Bibliográficas

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ANEXO 1

DECLARAÇÃO DE AUTORIZAÇÃO DE USO DE CONTEÚDO

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