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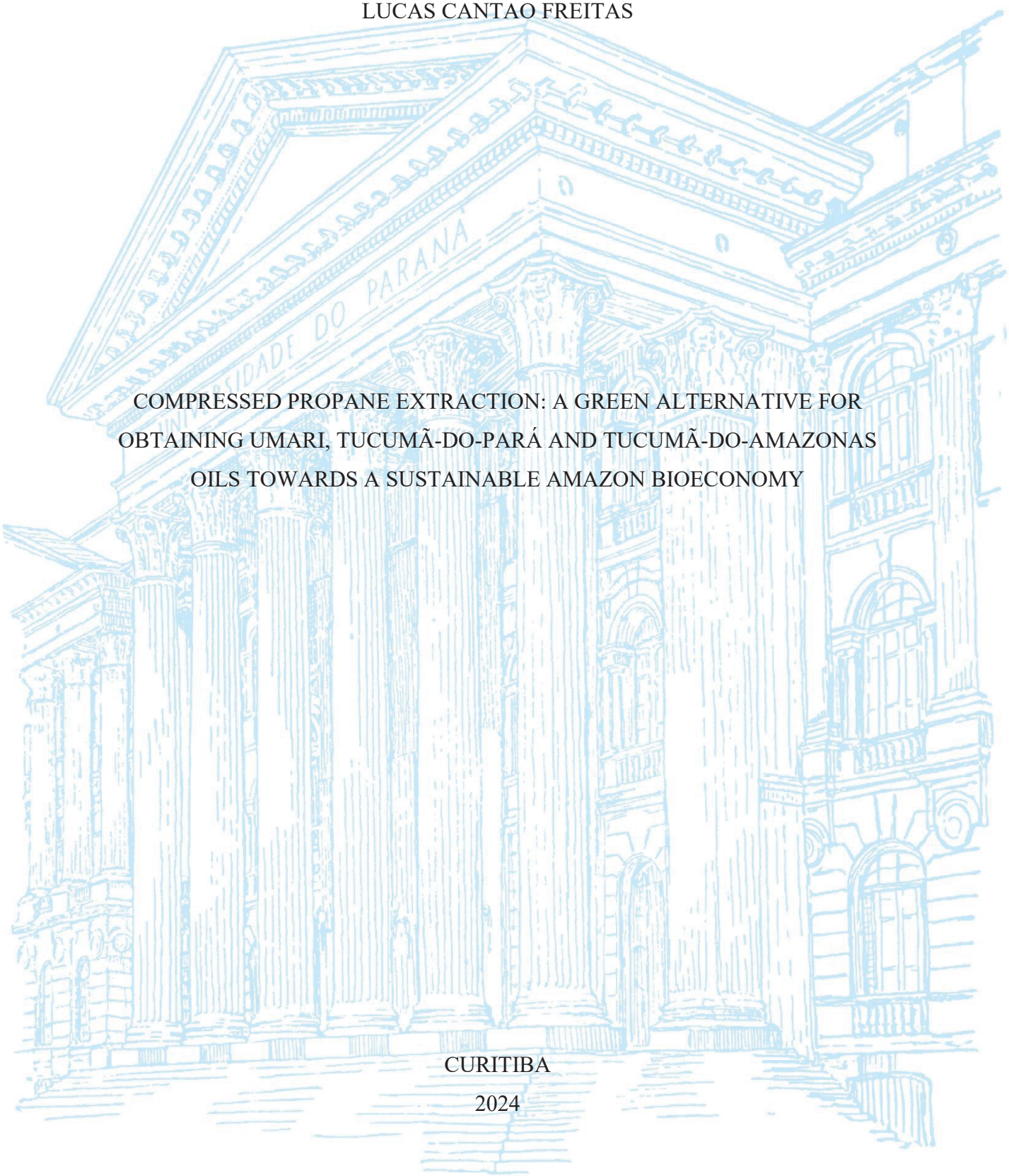

COMPRESSED PROPANE EXTRACTION: A GREEN ALTERNATIVE FOR OBTAINING UMARI, TUCUMÃ-DO-PARÁ AND TUCUMÃ-DO-AMAZONAS OILS TOWARDS A SUSTAINABLE AMAZON BIOECONOMY

CURITIBA

2024

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Tese apresentada ao curso de Pós-Graduação em Engenharia de Alimentos, Setor de Tecnologia, Universidade Federal do Paraná, como requisito para a obtenção do título de Doutor em Engenharia de Alimentos.

Orientadora: Profª. Drª. Maria Lucia Masson
Coorientador: Prof. Dr. Marcos Lúcio Corazza

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RESUMO

Esta pesquisa apresenta uma abordagem da atual conjuntura da bioeconomia na Amazônia, destacando as principais tecnologias de extração verde que podem impulsionar o desenvolvimento da bioeconomia regional. Paralelamente, a pesquisa aplica uma tecnologia de extração verde, a extração com propano pressurizado (ou comprimido), para a obtenção de óleos ricos em compostos de alto valor agregado de três espécies frutíferas da Amazônia, destacando suas potencialidades bioeconômicas. No capítulo I, é realizada uma abordagem crítica por meio de um artigo de revisão que discute o atual cenário da bioeconomia na Amazônia, trazendo dados importantes que podem nortear decisões governamentais e orientar os empreendedores que desejam investir na bioeconomia Amazônica. Este artigo de revisão traz uma visão geral e atualizada do uso de tecnologias de extração verde em cinco matérias primas amazônicas, discutindo os seus aspectos técnicos e econômicos, assim como as tendências futuras. O Capítulo II é um artigo de pesquisa que aplica uma tecnologia de extração verde (extração com propano pressurizado) para a obtenção de óleos da polpa de umari (*Poraqueiba sericea*), uma fruta amazônica pouco explorada e subvalorizada. Os resultados revelaram rendimentos de extração de até 29.2% em óleo rico em β-caroteno e ômega-9, com potencial para diversas aplicações industriais. O capítulo III aborda um artigo de pesquisa que aplica o propano pressurizado para obtenção de óleos da polpa de tucumã-do-Pará (*Astrocaryum vulgare*), que é uma fruta mais popular e conhecida na região amazônica. Os resultados também apontaram um rendimento de extração de até 33.9% em óleo rico em compostos bioativos, como o β-caroteno e ômega-9, apresentando um perfil de ácidos graxos bastante semelhante ao do óleo de umari. Por fim, o capítulo IV apresenta um artigo de pesquisa que utiliza um subproduto agroindustrial, a amêndoa do tucumã-do-Amazonas (*Astrocaryum aculeatum*), para a extração de óleo. O propano mostrou-se altamente eficiente para a obtenção do óleo, proporcionando altos rendimentos (até 35.9%) em um curto tempo, extraindo óleos ricos em ácidos láurico e mirístico. Portanto, os óleos das três espécies estudadas nessa pesquisa apresentam compostos de valor agregado com potencial para serem inseridos no cenário bioeconômico amazônico, podendo gerar renda e desenvolvimento regional.

Palavras-chave: Bioeconomia; Amazônia; Oleaginosas; Extração verde; Propano.

ABSTRACT

This research provides an in-depth analysis of the current state of the bioeconomy in the Amazon, emphasizing the key green extraction technologies that have the potential to drive regional bioeconomic development. At the same time, the study applies a green extraction technology (compressed propane extraction), to obtain oils rich in high-value compounds from three Amazonian fruit species, highlighting their bioeconomic potential. In Chapter I, a critical approach is carried out through a review article that discusses the current scenario of the bioeconomy in the Amazon, bringing important data that can inform government decisions and assist entrepreneurs who wish to invest in the Amazon bioeconomy. This review article provides an updated overview of the use of green extraction technologies in five Amazonian raw materials, discussing their technical and economic aspects, as well as future trends. Chapter II is a research article that applies a green extraction technology (compressed propane extraction) to obtain oils from the umari (*Poraqueiba sericea)* pulp, an underexplored and undervalued Amazonian fruit. The results revealed extraction yields of up to 29.2 wt% in oil rich in β-carotene and omega-9, with potential for several industrial applications. Chapter III addresses a research article that applies compressed propane to obtain oils from tucumã-do-Pará (*Astrocaryum vulgare*) pulp, which is a more popular and well-known fruit in the Amazon region. The results showed an extraction yield of up to 33.9 wt% in oil rich in bioactive compounds, such as β-carotene and omega-9, presenting a fatty acid profile quite similar to that of umari oil. Finally, Chapter IV presents a research article that uses an agro-industrial byproduct, the tucumã-do-Amazonas (*Astrocaryum aculeatum*) almonds, for oil extraction. Compressed propane proved to be highly efficient for obtaining the oil, providing high yields (up to 35.9 wt%) in a short time, extracting oils rich in myristic and lauric acids. Therefore, the oils from the three species studied in this research contain value-added compounds with the potential to be integrated into the Amazonian bioeconomic scenario, potentially generating income and fostering regional development.

Keywords: Bioeconomy; Amazon; Oilseeds; Green extraction; Propane.

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INTRODUCTION

The Amazon is home to a unique biodiversity, with an immense variety of species, many of which are still unknown or little explored industrially. However, with the development of the bioeconomy in the region, it is crucial to rise new alternatives that meet both socioeconomic and environmental demands. In this context, the sustainable exploitation of Amazonian species, combining traditional and scientific knowledge, and encouraging the participation of local communities in the production chains, becomes a promising path for regional development (Freitas *et al*., 2024b).

Some Amazonian species have already transcended regional borders, such as açaí (*Euterpe oleracea*) and cupuassu (*Theobroma grandiflorum*), which are currently consumed worldwide (Barbosa; Carvalho Junior, 2022; Marasca *et al*., 2022). These raw materials have been the subject of a significant amount of research and published studies, in addition to being among the main Amazonian species that generate income for the region. However, other species still lack studies and information that could attract the interest of investors in the bioeconomy sector. Among these species, the umari, tucumã-do-Pará, and tucumã-do-Amazonas stand out. These native Amazonian fruits are rich in oils containing high-value bioactive compounds (Freitas *et al*., 2024a; Machado *et al*., 2022).

Umari (*Poraqueiba sericea*) is an Amazonian fruit that is practically unexplored economically and scientifically, being sold at very low prices. Its pulp is very oily, with a fatty acid composition rich in oleic acid, a monounsaturated fatty acid known as omega-9 that has several health benefits. In addition, umari is rich in β-carotene, which is a precursor of vitamin A, with potential for use in several industrial segments (Freitas *et al*., 2024a). On the other hand, tucumã-do-Pará (*Astrocaryum vulgare*) is an Amazonian fruit that is better known and more widely consumed than umari. It is also an excellent source of oil rich in β-carotene and oleic acid, with relevant published studies proving the presence of these bioactive compounds. However, few alternatives have already been raised for extracting its oil in a sustainable way. Finally, the tucumã-do-Amazonas (*Astrocaryum aculeatum*), which is the main ingredient of the typical food known as "X-caboquinho", in the city of Manaus, Amazonas, Brazil, is a fruit highly valued in this region. However, once the pulp is extracted, the tucumã almond remains and is classified

as an agro-industrial byproduct, generating an environmental problem (Carvalho *et al*., 2024; Machado *et al*., 2022).

In this sense, aiming to combine the exploitation of natural resources with sustainable development, the application of green extraction technologies emerges as an environmentally friendly alternative for obtaining value-added components from Amazonian raw materials. Among these technologies, compressed propane extraction (CPE) has been widely considered for obtaining oils from vegetable matrices, as this solvent has a high affinity for lipid components. Considering that β-carotene is a fat-soluble compound, this method ends up being highly efficient for obtaining it. In addition, CPE has high extraction efficiency, providing high yields in less time when compared to conventional techniques (Teixeira *et al*., 2018; Zanqui *et al*., 2020).

Thus, to propose a green alternative for the extraction of Amazonian oils rich in high-value compounds, this research aims to apply extraction with compressed propane to obtain oils from the pulps of umari and tucumã-do-Pará, as well as from the tucumã-do-Amazonas almonds. The research seeks to elucidate the impacts of the process parameters on the extraction yields and characterize the oils, highlighting their benefits and possible applications in the Amazonian bioeconomic scenario. Furthermore, this thesis presents a critical approach to the current bioeconomic scenario in the Amazon, highlighting its main limitations, suggestions for improvement, and future trends, serving as an important information source for those who wish to invest in the Amazonian bioeconomy.

OBJECTIVES

GENERAL OBJECTIVE

Apply the compressed propane extraction method to obtain high-quality Amazonian oils from umari and tucumã, highlighting their bioeconomic potential.

SPECIFIC OBJECTIVES

Chapter I - Green extraction technologies: A path to the Amazon bioeconomy development.

- Discuss the current scenario of the Amazon bioeconomy;
- Highlight the green extraction technologies that can boost the Amazon bioeconomy;
- Review and highlight the main Amazonian raw materials with significant bioeconomic potential;
- Prospect future trends for the Amazon bioeconomy.

Chapter II - Compressed propane extraction of umari pulp oil: A rich Amazon source of β-carotene and omega-9.

- Obtain umari pulp oil using compressed propane as a solvent, investigating the effects of temperature and flow rate on the extraction oil yields;
- Compare the compressed propane extraction to conventional extraction technique;
- Analyze and characterize the oils obtained by compressed propane for the first time in the literature.
- Discuss the potential applications of β-carotene and omega-9 rich oils within the bioeconomic context.

Chapter III - Green extraction of β-carotene-rich oil from tucumã (*Astrocaryum vulgare* Mart.) pulp using compressed propane.

- Investigate the effects of temperature and flow rate on the oil extraction yields from tucumã pulp;
- Compare the different extraction methods in terms of yield;
- Analyze the extraction kinetics, identifying the best extraction conditions;
- Characterize the oils obtained and highlight their potential for industrial applications.

Chapter IV - Extraction of tucumã-do-Amazonas (*Astrocaryum aculeatum*) almond oil using compressed propane as solvent.

- Investigate the impact of process parameter variation on extraction yield;
- Evaluate the extraction kinetics to identify the most effective extraction conditions;
- Characterize the oil obtained by different extraction methods;
- Raise the potential of this agro-industrial byproduct for the Amazon bioeconomy.

CHAPTER I

GREEN EXTRACTION TECHNOLOGIES: A PATH TO THE AMAZON BIOECONOMY DEVELOPMENT.

This chapter is part of the article entitled “Green extraction technologies: a path to the amazon bioeconomy development” **PUBLISHED** in *Trends in Food Science & Technology*.

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GREEN EXTRACTION TECHNOLOGIES: A PATH TO THE AMAZON BIOECONOMY DEVELOPMENT

ABSTRACT

The conservation of the Amazon is necessary to mitigate two planetary crises: climate change and the loss of biodiversity that threatens different species. The current economic model practiced in the region is directly linked to the exploitation of natural resources without much concern for the environment. Therefore, there is an urgent need for incentives aimed at the full development of the bioeconomy in the region, with the application of green technologies to obtain sustainable added-value products. This review will highlight some green technologies to develop the bioeconomy in the Amazon region. Additionally, five Amazonian raw materials will be explored as potential boosters of the bioeconomy. Moreover, this study brings a panoramic approach to the current scenario, trends, challenges, and business opportunities for those who want to invest in the Amazon, in addition to supporting government public policies aimed at the region's sustainable development. Green technologies related to the bioeconomy should provide significant advancements in revenue creation and sustainable development of the Amazon, preserving the forest, adding value to local raw resources, and supporting the socio-bioeconomy development. However, for this to become a reality, the public and private sectors must work together toward a common goal: preserving Amazon biodiversity and using it sustainably and consciously, uniting science with the traditional knowledge of the local people.

Keywords: bioeconomy, amazon, green technology, sustainability, business opportunity.

INTRODUCTION

In an era when the notion of bioeconomy has been widely studied and disseminated worldwide, the Amazon stands out as one of the primary regions with enormous potential for the bioeconomy's emergence and development. Because of its immense biodiversity and abundance, the Amazon provides a diverse range of raw materials with bioeconomic potential that can provide a source of income and development to the region. Several species of plants and fruits from this region have added-value components that can be extracted sustainably by using green and environmentally friendly technologies (Barbosa & Carvalho Junior, 2022; Bergamo et al., 2022; Freitas et al., 2021; Valli et al., 2018).

In this framework, several studies indicate that numerous Amazonian species have secondary metabolites with active principles, which have important activities such as anti-inflammatory, anticancer, antidiabetic, antimicrobial, antiglicant, allelopathic, and so on. In addition, such species have high concentrations of antioxidants, vitamins, fibers, and other added-value compounds that can be used in different industrial sectors (Curimbaba et al., 2020; Peixoto Araujo et al., 2021).

Among the green extraction technologies capable of extracting components with high added-value from Amazonian species, we can highlight Supercritical Fluid Extraction (SFE), Ultrasound-assisted Extraction (UAE), Microwave-assisted Extraction (MAE), Pressurized Liquid Extraction (PLE) and Deep Eutectic Solvents (DES) extraction (Pagano et al., 2021; Zannou & Koca, 2022). All these extraction techniques are environmentally friendly and produce high-quality extracts. Some studies point to the technical feasibility of these extraction methods applied to Amazonian species (Barbi et al., 2019; Fetzer et al., 2021; Menezes Silva et al., 2023; Monteiro et al., 2019; Pinto et al., 2018; Pires et al., 2019). However, scale-up and economic feasibility studies are rare and limited to a few species.

Despite the great potential, Amazon is still little explored in terms of extraction of added-value components from its raw materials using green technologies. Even açaí, currently widespread and consumed worldwide, does not have a consolidated exploitation where all its fractions are used. Most of its agro-industrial by-products, such as seeds and fibers, are still discarded in the environment as waste. Even with a lot of scientific evidence that these by-products have molecules with relevant value (Barbosa & Carvalho

Junior, 2022), the scarcity of investments and government incentives for entrepreneurship in the area is still limiting the development of the bioeconomy in the region. However, this scenario tends to change in the coming years due to new strategies that local governments have recently applied.

Additionally, Belém, the capital of Pará state, was chosen to host the 30th edition of the United Nations Conference on Climate Change (COP-30) in 2025. Hosting this significant event in a city in the heart of the Brazilian Amazon should draw global attention to the region and, as a result, leverage the discussion on bioeconomy and sustainability.

Among the Amazonian species with relevant bioeconomic potential, we can highlight açaí (*Euterpe oleracea*), cupuassu (*Theobroma grandiflorum*), Brazil nut (*Bertholletia excelsa*), buriti (*Mauritia flexuosa*), peach palm (*Bactris gasipaes*) and their by-products. Because the food, cosmetics, and pharmaceutical industries are interested in these high-quality active principles, such species are significant sources of secondary metabolites that can be marketed at attractive pricing when extracted using green methods.

As there is a lack of studies linking green extraction methods and the bioeconomy in the Amazon, this review aims to show the current scenario on the application of green technologies for the development of the bioeconomy in the Amazon, highlighting the green extraction technologies that can boost the bioeconomy in this region. Furthermore, this review highlights the primary Amazonian raw materials with bioeconomic potentials. It also addresses an analysis of investment trends and business opportunities in this area, intending to provide relevant information to encourage governments, the private sector, civil society, and the international community to invest and implement in the Amazon region sustainably.

CURRENT BIOECONOMY SCENARIO IN THE AMAZON

Due to its great biodiversity, the Amazon has been the focus of debates on bioeconomy, owing to its diverse range of raw resources with bioeconomy potential. The possibility of successful implementation of businesses in the bioeconomy segment in this region has attracted the attention of investors from all over the world. In the same way,

the local governments see it as a chance for sustainable economic growth and income generation in the region, through valorizing the production chain of some crops, such as açaí and cupuassu, by applying environmentally friendly technologies. As a result, incentive schemes for investment in this sector have been included to the goals of local governments (Pamplona et al., 2021; Valli et al., 2018).

Furthermore, global concern about the consequences of climate change has led to the development of more sustainable extraction technologies. The current scenario on the application of green extraction technologies addressing the development of the Amazon bioeconomy is still at an initial stage, where most of the technical feasibility information still comes from research performed on a laboratory scale. However, some research has already been carried out in terms of scale-up, making it possible to prospect the implementation of processes on an industrial scale (Menezes et al., 2022, 2023). There is still a restricted scope of economic feasibility studies, focusing on only a handful of species. This underscores the requirement for further research in this field.

Currently, we can observe that there has been a narrowing in the relationship between research institutions with public and private companies located in the Amazon region. This symbiotic relationship between research institutions (mainly universities) and private companies is very well explored in developed countries, with positive results. The main objective is to articulate the application of scientific and technological knowledge to transform natural resources into productive and financial assets supported by industrial structures of sustainable production.

The food, pharmaceutical, and cosmetics industries are the most connected with the growth of the bioeconomy in the Amazon. Some industries in the region have been adapting to the new global trends and gradually replacing their old conventional processes with environmentally friendly ones that take full advantage of the raw material, thus approaching the concept of biorefinery and circular economy. Natura and Amazonian SikinFood, both in the cosmetic sector, and Manioca, in the food sector, are examples of companies located in the Amazon that have already been developing some product lines using green technologies, involving local communities while seeking to cause less impact on the environment. This change in the industrial production profile has been providing more credibility and respect to the consumers, as they know that the company is concerned about the impacts they can have to the environment. These industrial practices

are usually conveyed in the marketing/branding of products, causing positive effect from both economic and socio-environmental points of view.

The adoption of sustainable technology targeted at adding value to Amazon Forest products must be the key to making the region's bioeconomy viable. According to Felin & Feltran-Barbieri (2023), the current GDP of the bioeconomy in the Legal Amazon is at least R$12 billion per year. This value considering only 13 products, for which there are statistical records. In terms of Brazil, the GDP of the bioeconomy (GDP-Bio) totaled R$1.447 trillion in 2019, representing 19.6% of Brazilian GDP in 2019. This value is made up of activities of plant origin (R$ 357,749.0 million, 24.7% of the total GDP-Bio); activities of animal origin (R$ 115,758.4 million, 8.0% of the total GDP-Bio); activities of extractive origin (R$ 41,144.8 million, 2.8% of the total GDP-Bio); bioindustrial activities 100% bio-based (R$ 777,585.6 million, 53.7% of the total GDP-Bio); and the activities of the bio-based bioindustry (R$ 154,514.8 million, 10.7% of the total GDP-Bio) (Lima & Pinto, 2022). Through these statistical data, it is possible to observe that the percentage of participation of the Amazon in the Brazilian bioeconomy is still small, thus requiring solid investments and actions for the development of the bioeconomy in this region.

Among the new actions related to the bioeconomy, the “Amazônia 4.0” project stands out, intending to leverage the development of the bioeconomy in the Amazon. Amazônia 4.0 is an institute that develops advanced technologies and methods to transform Amazon inputs into products with high added value, form a powerful bioindustry, train the local people, and create urgently needed alternatives to deforestation. Furthermore, combining traditional knowledge with modern science and Industry 4.0 within its mobile biofactories, referred to as the Creative Laboratories of the Amazon (LCAs) (Nobre & Nobre, 2019). The Amazônia 4.0 team is comprised of highly skilled individuals with a unique blend of scientific and technological knowledge, as well as management specialists. In addition to Amazônia 4.0, there is also the “Amazônia 2030” project, an initiative by Brazilian researchers to develop an action plan for the Brazilian Amazon. The project's objective is for the region to reach a higher level of economic and human development and achieve the sustainable use of natural resources in 2030 (Coslovsky, 2021).

On the other hand, it is important to highlight that projects of this magnitude, such as "Amazônia 4.0" and "Amazônia 2030", have some risks and limitations. For example,

factors such as region isolation, difficult access to equipment and training, lack of infrastructure, production volume, quality assurance, logistical difficulties, access to markets and lack of experience in selling are some of the challenges that hinder the development of these projects in the Amazon region. In many cases, the distances to transport production by river or land are enormous, raising problems with perishable raw materials such as açaí and cupuassu. A possible solution to this problem would be the prior drying of these fruits by means of cost-effective drying methods, as their volumes and weights would be reduced and their nutritional quality would be maintained, thus facilitating the logistics. Solar and vacuum based drying methods are promising in this sense (Reis et al., 2022). A future logistical alternative that has been quite studied is the air transport by unmanned vehicles. A logistics system involving the use of drones to transport value-added products in difficult-to-access regions have been seen as a promising alternative (Nobre & Nobre, 2019; Moshref-Javadi & Winkenbach, 2021; Costa et al., 2022).

In addition to the risks and constraints already mentioned, the potential reinforcement of exploitative practices generating paths to a greenwashing practice concerns many scientists. The strategy of local governments to facilitate, through government incentives, the installation of bio enterprises in the Amazon region can have a disastrous effect, since these companies may not actually be committed to the practice of the bioeconomy, merely just using this to receive government benefits and link a sustainable practice to their brands to sell more products. In other words, there is a risk that companies will simply apply the “bio” label to a green practice that is not necessarily reflected in the practice. This can be used for ‘greenwashing’, that is, using only sustainability rhetoric without substantial commitment. There is also a critical current that fears the use of bioeconomy as the “neoliberalization of nature” (Birner, 2018; Gawel et al., 2019; Garrett et al., 2023). In view of this, it is essential that local governments adopt a policy of in-depth assessment of companies capable of implementing businesses focused on the Amazon bioeconomy, aiming at the supervision and auditing of these companies effectively, considering the economic, environmental, and social effects of bioeconomy strategies and initiatives.

In addition to the programs already mentioned, there are some other initiatives, such as the UN 2030 agenda, which is a program where the Amazon is fully immersed, with implications for many global sectors. Therefore, achieving the UN's 2030 Agenda

in Latin America, especially the Sustainable Development Goals 7 (Affordable and Clean Energy), 9 (Industry, Innovation and Infrastructure), 12 (Responsible Consumption and Production), and 13 (Climate Action), is a priority not only for all countries that make up the Amazon region but also for the entire world. In this context, the development of the Amazon bioeconomy can largely contribute to achieving some of these goals.

Recently, the government of the state of Pará, in Brazil, launched the "State Bioeconomy Plan" during the 27th ONU Conference on Climate Change (COP-27), in Sharm el-Sheikh, Egypt. The three axes present in the Bioeconomy plan are: Research, development, and innovation; Genetic and cultural heritage and traditional knowledge; and Sustainable value chains and businesses, which bring together the 92 planned actions. According to the plan, this sustainable economy model should generate revenue of BRL 129 billion by 2040, which is equivalent to the state's current GDP. In this scenario, Pará stands out as the first state in Brazil to have a bioeconomy plan (Pará, 2022). **Fig.1** summarizes the current scenario and future challenges for the Amazon region.

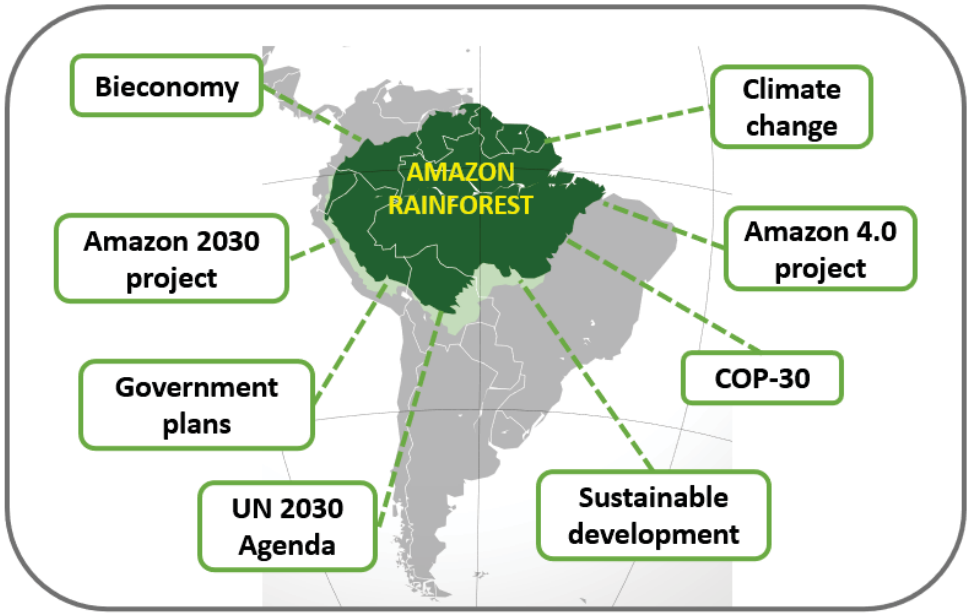

Figure 1 - Current scenario and future challenges for the Amazon region.

Finally, there is an incorrect belief that there are no skilled specialists in the Amazon region to meet the needs of potential industrial demands related to bioeconomic production; nevertheless, this is not the case. Several prominent research institutes in the Amazon annually graduate a substantial number of persons capable of working in these industries in terms of human resources and qualified labor. The vast majority of them already have expertise with local raw materials, as they have carried out in-depth research

at master's and doctoral levels applying green and sustainable techniques for extracting added-value substances from Amazonian matrices. Courses in Food Engineering, Chemical Engineering, Industrial Chemistry, Bioprocess engineering and Biotechnology, in addition to their respective postgraduate programs, are part of the list of courses at several renowned universities located in the Amazon region, such as the Federal University of Amazonas (UFAM) and the Federal University of Pará (UFPA) that provide hundreds of professionals to the market every year. Due to a lack of possibilities and professional development in the region, many of these highly qualified individuals go to other regions and even other countries. This dynamic is changing as the Amazon bioeconomy develops, and this qualified workforce can meet the needs of bio-enterprises that come to reside in the region.

Still in this context, there is a need to insert the local population into the Amazon bioeconomy scenario, taking advantage of their traditional knowledge to stimulate the socio-bioeconomy development, keeping the forest preserved and alive. Costa et al. (2022) also corroborate that support for indigenous peoples and local rural and urban communities is essential for the growth of their local economies through the valorization of their different forms of production and economic integration. Furthermore, Amazonian cities play an important role in promoting this bioeconomy, promoting mediation between society and nature. Urban areas located in the Amazon have the capacity to articulate, intensify, expand and create trends of economic diversification, which is strategic for maintaining and enabling a bio-ecology bioeconomy in the Amazon. To this end, it is necessary to implement a sustainable telecommunications, logistics and energy infrastructure compatible with the conservation of the region's terrestrial and aquatic ecosystems, respecting territorial peculiarities and meeting the priority interests and needs of the substantive economies of *quilombolas*, indigenous people, family farmers and traditional communities, establishing a harmonious relationship between the productive sector and sustainable development. It is essential that bio companies carry out sustainable practices in an honest and real way, without making misleading or unsubstantiated claims about the organization’s environmental performance (Garrett et al., 2023; Costa et al., 2023; Ollinaho & Kröger, 2023; Costa et al., 2022).

NEW EXTRACTION TECHNOLOGIES THAT CAN BOOST THE BIOECONOMY IN THE AMAZON

In this section, we will present an approach regarding the primary environmentally friendly extraction methods possessing bioeconomic potential. This approach aims to contribute to the advancement of the Amazon region, along with discussing their current state of the art in terms of application and combinations with other extraction techniques.

SUPERCRITICAL FLUID EXTRACTION (SFE)

Supercritical fluid extraction (SFE) is a technique that uses substances at temperatures and pressures above their critical points. These substances are referred to as supercritical fluids (SF), and they have remarkable features that combine the advantages of the liquid and gas phases., i.e., the SF can diffuse as a gas having a solvating power of a liquid. Due to its high penetrability power, it is possible to extract target compounds more efficiently. Pressure and temperature are the most studied variables in supercritical fluid extraction since they directly influence solvent density, leading to enhance selectivity of chemicals to be extracted. In addition to pressure and temperature, other process variables are being extensively studied, such as solvent flow, type and concentration of co-solvent, particle size, porosity, density, particle diameter, and bed height. SFE also enables its combination with other emerging technologies, such as pressurized liquid extraction (PLE) and ultrasound-assisted extraction (UAE), optimizing extract yields and quality, becoming an excellent extraction tool in terms of biorefinery (Carreira-Casais et al., 2021; Oliveira et al., 2023; Preetam et al., 2023).

Carbon dioxide is the fluid most used in SFE because it has several advantages, such as its characteristics under critical conditions of pressure and temperature (T_c = 31.1°C, P_c = 7.38 MPa), in addition to having high selectivity, making it suitable for extracting bioactive components. CO_2, in this scenario, is considered non-toxic, relatively inexpensive, non-explosive, readily available on the market, easy to remove from the final product, and has good extraction capacity due to its high penetration power. Yet, it is generally recognized as safe (GRAS), not hazardous to health, and therefore approved for use in food. Notably, CO_2 has low polarity and hence is inefficient for extracting polar

components such as phenolic compounds. However, using co-solvents makes it feasible to modify the polarity and broaden the spectrum of polar molecules that can be extracted (Fetzer et al., 2022; Freitas et al., 2021; Zoccali et al., 2019).

Many improvements and innovations have been implemented in the SFE to optimize the results in quantitative and qualitative terms. For example, there is the extraction in several steps, in which the pressure and temperature variables are gradually increased, obtaining fractions with different characteristics in each stage. In summary, non-polar and highly soluble chemicals are extracted using co-solvents at lower pressures, followed by moderately soluble compounds at intermediate pressures, and finally, polar and weakly soluble compounds at higher pressures. Likewise, in this same context, sequential supercritical extraction (SSE) has also been gaining attention from researchers. In this method, two or more extraction techniques, such as SFE and PLE, are combined, where the non-polar fraction is extracted with supercritical CO_2 or subcritical propane, and the predominantly polar fraction is extracted with PLE using polar solvents such as ethanol or water. These multi-step biorefining processes can increase the overall economic viability of extractions by adding an extra-value product with different compositions and properties (Avilés et al., 2023; Marillán & Uquiche, 2023; Teixeira et al., 2021).

Despite being a technology with positive characteristics and having had a considerable scientific advance in recent years, there is still a deficit of information about supercritical extraction processes' scalability. There is still a scarcity of data in the literature that may be used to support the implementation of large-scale extraction systems. Furthermore, it is vital to note that each raw material has its own unique characteristics; it is not possible to fully recreate the process parameters of one raw material in another, therefore each one must be studied separately. Several Amazonian natural matrices have been explored using SFE in this context. However, the rate of progress remains constrained due to the lack of researchers engaged in exploring this domain, coupled with the absence of robust incentives from both the public and private sectors. There is a discernible shift in this landscape, characterized by an emerging trajectory of heightened impetus within this sphere. This trajectory is primarily attributed to governmental interests converging with private initiatives to establish ecologically conscientious industries that harmonize with the forest ecosystem. This evolving landscape aligns seamlessly with the global imperative for the sustainable utilization of

Amazonian resources, thereby catalyzing the advancement of the bioeconomy across the region.

COMPRESSED PROPANE EXTRACTION (CPE)

Compressed propane extraction is an environmentally friendly method increasingly used for obtaining oils from plant matrices. This nonpolar solvent has a high affinity for lipid compounds, making it particularly suitable for extracting lipophilic bioactive compounds such as β-carotene. Typically, the same equipment used for supercritical CO_2 extraction can be employed, with only the solvent supply needing to be changed. Additionally, equipment for compressed propane extraction generally operates at lower pressures than supercritical CO_2, potentially reducing both capital and operational costs. Propane can be efficiently recovered and recycled during the extraction process, minimizing waste and lowering overall solvent consumption. This extraction method presents an alternative for use in biorefineries of oilseeds, often employed in the initial degreasing stage. Following this, the biomass can proceed to other extraction processes such as supercritical CO_2 with co-solvents or pressurized liquid extraction (PLE). Several studies have reported that compressed propane extraction is highly efficient, achieving yields comparable to or even exceeding those of other environmentally friendly extraction techniques (Zanqui et al., 2020; Trentini et al., 2017; Correa et al., 2016; Pederssetti et al., 2011).

ULTRASOUND-ASSISTED EXTRACTION (UAE)

UAE is considered an environmentally friendly technique for the food industry, mainly due to the characteristics attributed to ultrasound. In extraction processes involving ultrasound, mechanical waves propagating through the medium in cycles of compression and rarefaction at frequencies between 20 kHz and 100 kHz, promote the phenomenon called cavitation. In cavitation, the nucleation, growth, and collapse of microbubbles in liquids cause a sudden increase in temperature and pressure in the medium through which the waves propagate, as well as the creation of microjets. The microjets are responsible for the erosion and/or rupture of the solid particles in the

medium, providing a good extraction efficiency. By optimizing parameters such as ultrasound frequency, power, and time, it is possible to obtain extractions of chemical compounds of interest with recoveries similar to those of classical methods in less time (Beaudor et al., 2023; Heleno et al., 2016; Menezes Silva et al., 2023; Pereira et al., 2017; Wang et al., 2023; Xu et al., 2022).

UAE has already been applied to some Amazonian matrices, obtaining promising results in yield, quality of extracts, and environmental aspects (Menezes et al., 2023; Silva et al., 2022). However, scale-up studies are scarce, and more research is needed to elucidate process parameters and economic viability. Due to the gradual increase in the number of scientific works in the field and the private sector interest, the trend is that this scenario faces a positive change in the coming years, mainly in terms of its application aimed at transferring technology from the laboratory to the industrial scale, providing the advancement of the Amazon bioeconomy.

MICROWAVE-ASSISTED EXTRACTION (MAE)

Microwaves are electromagnetic radiation whose frequency ranges from 300 MHz to 300 GHz, corresponding to wavelengths from 1 mm to 1 m. Electromagnetic radiation is non-ionizing energy, meaning it does not disrupt chemical bonds or produce molecular changes owing to electron removal. The electromagnetic radiation of microwave energy results from the action of two perpendicular fields: electric field and magnetic field. Microwaves, in particular, act on the molecules of the material exposed to them through dipole rotation and ionic conduction. These mechanisms are responsible for heating the material by converting electromagnetic energy into heat. According to the theory, dipole rotation happens when an electric field is applied, which promotes the alignment of molecules having dipole moments, and when these molecules return to their disordered state, the energy gained during their alignment is lost in the form of heat. This process of alternating alignment and relaxation of molecules occurs thousands of times per second, generating heat by molecular friction. The ionic conduction mechanism is developed through the electromagnetic field that promotes the migration of ions that are attracted by the poles of opposite signs. The alternation of the poles occurs thousands of times per second, resulting in the collision of the ions with each other and with non-ionized

molecules, generating heat by friction. Thus, in microwave heating, or dielectric heating as it is also known, heat is generated uniformly inside the matrix, directing itself to its surface (Ganorkar et al., 2023; Kim Tran et al., 2023; Letellier & Budzinski, 1999; Liew et al., 2016).

In microwave-assisted extraction, the electromagnetic energy converted into heat increases the internal temperature of the matrix cells due to heating and evaporation of the moisture present, causing an increase in internal pressure, rupture of membranes, and release of the target compound. Among the microwave-assisted extraction process variables, the equipment power, extraction time, type of solvent, ratio between solid and solvent, and the condition of the plant matrix stand out. MAE has been used for the extraction of several components, such as pectins, polyphenols, flavonols, anthocyanins and polysaccharides(Gunalan et al., 2023; Jovanović et al., 2023; Wei et al., 2023). Therefore, there is a growing interest in this technology for extracting compounds with high added value from Amazonian food matrices and their by-products. Like the other extraction technologies presented here, the MAE also has few studies of process scalability, suggesting the need for more studies to elucidate the process.

PRESSURIZED LIQUID EXTRACTION (PLE)

PLE technology is efficient and produces little waste during the extraction process, which can reduce costs and save time. In the PLE process, the solvent is below the critical point to maintain the liquid phase during extraction. The pressure and temperature conditions are chosen to increase the mass transfer rate, reducing the surface tension and solvent viscosity and increasing the solubility of the components, which makes the solvent more permeable in the extracted solid matrix (Silva et al., 2020; Moret et al., 2013; Otero et al., 2019).

In general, the extraction process can be explained in two steps; first, it starts to be solubility-controlled, followed by a diffusion-controlled phase. In dispersion-controlled sample matrices, there are often strong interactions between the matrix and analytes or long diffusion paths for analytes to pass through the sample matrix. In this case, the solvent's temperature and the particles' size can be critical factors to increase the extraction efficiency. Increasing the temperature and/or reducing the particle size will

likely avoid extraordinarily long extraction times. In solubility-controlled sample matrices, the analyte-matrix interactions are quite weak, and the extraction rate depends mainly on the analyte partition between the matrix and the extraction fluid. In this case, the yield is increased by using more frequent replacement with fresh extraction solvent (Ho et al., 2022; Mustafa & Turner, 2011; Turner et al., 2001).

In PLE, a wide range of extraction temperatures can be used, ranging from ambient temperature to 200 °C, and pressures ranging from 3,5 to 20 MPa. Elevated temperatures also improve extraction efficiency, as it helps to disrupt analyte-sample matrix interactions caused by van der Waals forces, hydrogen bonding, and dipolar attraction. The main advantage of applying pressure during extraction is that a temperature above the boiling point can be used while the solvent maintains its liquid state. Moreover, PLE is suitable for a wide range of solutes, polar to non-polar, with the factors affecting extraction being: solvent type, extraction time, temperature, particle size, and water content of the sample. Solvents such as ethyl acetate, ethanol, and water are used in PLE, and mixing some of these solvents can improve the extraction efficiency (Fetzer et al., 2022; Möckel et al., 1987; Mustafa & Turner, 2011; Richter et al., 1997; Rostagno et al., 2009).

In this context, PLE is considered a green extraction method that can recover high amounts of phenolic compounds, lignins, carotenoids, lipids, essential oils and other compounds, representing an essential alternative for the pharmaceutical, chemical and food industry, and can be applied to obtain added-value components from various Amazonian matrices (Maciel-Silva et al., 2022a; Miranda et al., 2021; Rudke et al., 2019a; Viganó et al., 2022).

DEEP EUTECTIC SOLVENTS (DES)

Due to pollution and toxicity related to synthetic organic solvents used by the petrochemical, pharmaceutical, cosmetic, and food industries, there is environmental awareness and a worldwide demand for replacing these chemicals. In this sense, researchers have applied efforts to seek economically viable alternatives to the use of synthetic organic solvents, which, annually, are consumed around 20,000,000 metric tons (MMT) worldwide (Liu et al., 2022). In this scenario, Deep Eutectic Solvents (DESs)

have emerged as an ecologically correct alternative to conventional organic solvents (methanol, chloroform, toluene, and others). DESs differ from other green solvents in that they are low cost, less toxic, biocompatible, and easier to make. They are also sustainable solvents with high solubility, non-flammable, and difficult to volatilize.

DESs are mixtures obtained by mixing a quaternary ammonium or metal salt with a simple hydrogen donor (such as alcohols, acids, amines and amides), where hydrogen bonds are formed between the halide anion and the donor moiety of hydrogen. Compared to the original compounds before mixing, there is a considerable reduction in the melting point in the formation of the liquid. In this configuration, it was possible to observe an equilibrium in the melting point for two phases (solid-liquid), which comprise two different phases of solid and liquid melting of two constituents. Furthermore, most combinations used to form DESs are widely common and inexpensive (economic advantage). Natural eutectic solvents (NADESs) are gaining popularity due to their chemical variety, industry-acceptable toxicity, and biodegradability (Singh et al., 2021).

Grinding, evaporation, lyophilization, and assisted heating of other techniques (ultrasound and microwave) are the main methods of synthesis of DESs and NADESs. The grinding method allows mixing (room temperature) through simple grinding using a mortar and pestle. The heating method (usually between 50 and 100°C), the mixture is placed under constant agitation until a homogeneous liquid is formed. Both the grinding and heating process enable a process yield of 100% (Płotka-Wasylka et al., 2020; Saini et al., 2022).

DESs are used in different areas of industry and research, and their applicability mainly depends on their physicochemical properties. Due to their relatively high conductivity, DESs have numerous applications. For instance, DESs can be applied as absorbents for CO_2 removal and adsorption of toxic gases responsible for environmental pollution, extracting metals from marine, extraction of food proteins, removing phenolic compounds, extractive desulfurization of fuel oils, fractionation of waste lignocellulosic biomass and its conversion to added-value bio-based chemicals, extraction of fatty acids from microalgae biomass (Smith et al., 2014).

In terms of sustainability, the use of green technologies has been well regarded by consumers, increasing the value of their products. This has a decisive impact when consumers purchase products, who tend to prefer products from companies committed to

the environment. For instance, consumers tend to give preference to eco-friendly packaging, as they are easy to recycle and made from materials manufactured using minimal impact on energy consumption or natural resources. Another example is active packaging produced by supercritical solvent impregnation of natural extracts, a green technology which is an innovative approach for developing films with antioxidant capacity, promoting packaging that interacts with the product in a desirable way. In this same context, the "clean label" also emerges as a new and widely used term in the food industry, which refers to challenges that include the health and safety of food products from the perspective of maintaining environmental sustainability, intertwining with the bioeconomy concept (Siddiqui et al., 2022; Siegrist & Hartmann, 2020; Bastante et al., 2017).

Thus, with the knowledge obtained in this section, it is now possible to understand and correlate the technologies mentioned above with the reality of the Amazon and its prospects, specifying state of the art, and bringing scientific approaches to five Amazonian species that can leverage the bioeconomy in the region. Therefore, the next section will provide a source of knowledge and statistics to assist investors and government policymakers in the field of bioeconomy.

MAIN AMAZONIAN RAW MATERIALS WITH BIOECONOMIC POTENTIAL

Açaí (*Euterpe oleracea*), cupuassu (*Theobroma grandiflorum*), Brazil nut (*Bertholletia excelsa*), buriti (*Mauritia flexuosa*) and peach palm (*Bactris gasipaes*) were chosen to be covered in this work since they are the Amazonian raw materials with significant production and commercialization statistical data. Furthermore, they have scientific data that demonstrate the presence of value-added components that, if inserted into the bioeconomic scenario, can be potential accelerators of regional development, bringing income and improving the lives of local populations.

AÇAÍ (*Euterpe oleracea*)

General aspects

The açaí palm (*Euterpe oleracea* Mart.) is indigenous to the Brazilian Amazon, with the state of Pará having its primary distribution. The largest and densest populations of açaí palm are found in the estuary of the Amazon River. From the 1990s onward, the açaí palm began to be cultivated on a commercial scale (Nogueira et al., 2005). Açaí berries are edible and have been used to produce a wide range of foodstuffs. In year 2021, almost 1.4 million t of açaí berries were produced, amounting to about US$1 billion (Brasil, 2023).

The term “açaí”, followed by the total soluble solids content, is used to describe the drink obtained from the edible part of the fruit extracted with addition of water and filtration, preserving distinctive color, aroma and flavor, and minimal anthocyanin content. A prerequisite is that it displays a minimum of 8° Brix. The regulations in Brazil also address consumables such as "clarified açaí" and "dehydrated açaí" (Brasil, 2018). However, “açaí” is the main product made from the açaí berries, being used to produce most of the açaí foodstuffs.

Açaí berries contain several health-promoting (functional) compounds. Such compounds may be extracted from açaí pulp or seeds applying green extraction technologies. Açaí berries' health-promoting substances contain phenolic compounds, including anthocyanins, fatty acids, mannose, inulin, and cellulose.

Açaí as a raw material for multiple purposes

Various products can be made from açaí berries. For example, it is well known that these fruits contain remarkable amounts of anthocyanins, specially cyanidin 3-glucoside and cyanidin 3-rutinoside (Heck et al., 2023). Given the anthocyanins health-promoting properties, their extraction from açaí berries is a promising application for stimulating bioeconomy. In this context, conventional extraction approaches have been used to extract anthocyanins from açaí berries and prepare anthocyanin-rich extracts. For example, acidified methanol has been used for extracting anthocyanins from açaí berries (Gouvêa et al., 2012), clarified açaí pulp (Pacheco-Palencia & Talcott, 2010), and açaí

products in the USA (Lee & Finn, 2007). Regarding açaí extracts, an anthocyanin-rich dry extract was prepared from açaí berries by using ethanol and acetic acid (Silva et al., 2019). In another report, solid-phase extraction (SPE) with methanol has been used to produce an anthocyanin-rich açaí extract (Santos et al., 2014). In addition, a commercially available açaí extract is used as an anti-aging product (Clariant, 2023b). Açaí berries anthocyanins' stability was higher when they were stored at lower temperatures (20 °C) and in the dark (Paula et al., 2019).

Açaí berries can also be used for producing açaí berry oil, a dark green viscous product with antioxidant capacity and açaí aroma. Açaí berry oil is made from fruit pulp and being specially used by the pharmaceutical and cosmetics industries. The traditional methods used for açaí berry oil extraction comprise drying of açaí pulp followed by mechanical pressing (Lira et al., 2021) or by extraction with organic solvents like hexane (Castro et al., 2021). Açaí berry oil presents an acid value of 2.79 mg KOH/g, iodine value of 84.62 g I_2/100g, refractive index of 1.46, and density of 0.89 (Pereira et al., 2017). Its fatty acid profile is mainly composed of about 52% oleic acid, 26% palmitic acid, and 8% linoleic acid (Nascimento et al., 2008).

The seeds of açaí berries are used for different purposes. For example, products for skin exfoliation based on açaí seeds are commercially available (Clariant, 2023a). In addition, açaí seeds are a source of inulin, a dietary fiber, which can be extracted using aqueous extraction (Lima et al., 2021). Extraction of cellulose nanocrystals and lignin from açaí seeds using organic solvents, namely cyclohexane, and ethanol, is also reported (Linan et al., 2021). Cellulose can also be extracted from açaí seeds using only ethanol (Barros et al., 2021). Mannose, an aldohexose, can be obtained from açaí seeds employing water/ethanol extraction and acid hydrolysis of the mannan contained in them (Monteiro et al., 2019). Phenolic compounds like proanthocyanidins and resveratrol have been extracted from açaí seeds through extraction with cold methanol (Rodrigues et al., 2006), water (Barros et al., 2015), ethanol (Melo et al., 2016), methanol at 40°C and ethanol/water (Martins et al., 2020). Finally, oil can be extracted from açaí seeds by using n-hexane. Açaí seeds oil presents antimicrobial against *Staphylococcus aureus*, possibly useful for producing novel drugs for treating the diseases caused by these bacteria (Melhorança Filho & Pereira, 2012).

In the Belém Metropolitan Region of the Brazilian state of Pará, approximately 1200 t of açaí fibrous tissue is rejected after juice processing. According Mesquita (2013),

açaí fiber has the potential to be employed as a raw material in the fabrication of particle ecopanels (MDP) for commercial application in the civil construction and furnishing industries (Mesquita, 2013). **Fig. 2** shows the main products obtained from the açaí fruit that can boost its use in the bioeconomic scenario.

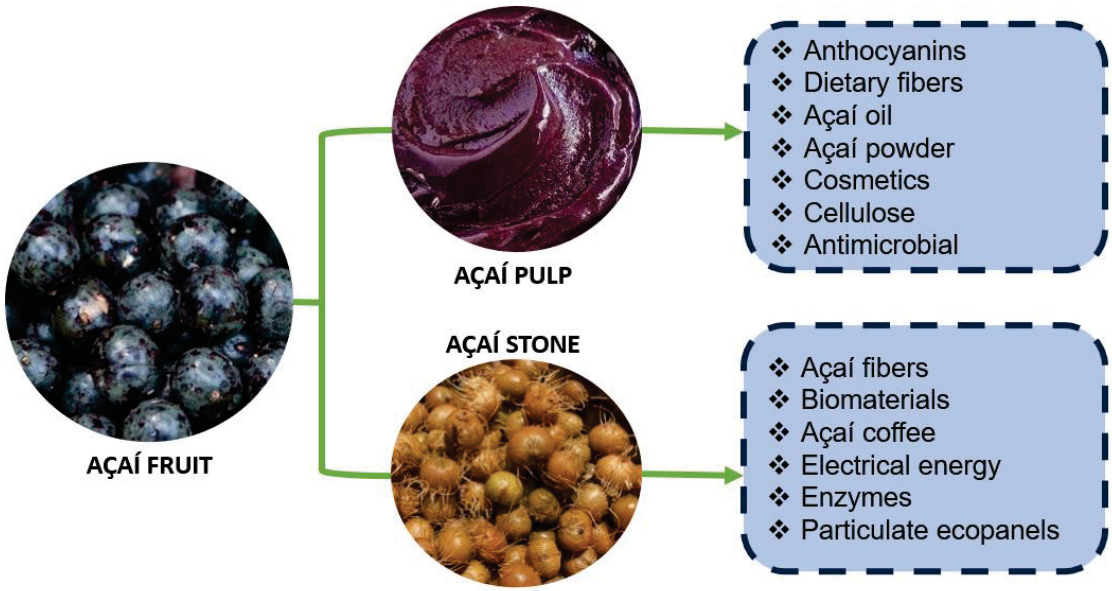

Figure 2 - Products obtained from the açaí fruit.

The full use of açaí berries is thus feasible, which fits the idea of biorefinery. A biorefinery can be defined as a framework in which biomass is used optimally for producing various products. Biorefineries attempt to be self-sustaining and environmentally friendly (Saral et al., 2022). According to Hingsamer & Jungmeier (2019) the main features of biorefineries are: 1) the coupled generation of energy (vaporous or liquid biofuels) and materials (such as chemicals, feed, and food); 2) the use of various processing steps (mechanical, thermochemical); and 3) the use of different raw materials from both virgin and remaining (residual) sources.

Green technologies applied to the açaí and its biorefinery prospects

Green biorefineries can operate as long as green technologies are used. Green extraction techniques require less energy, solvent, and time, thereby in line with sustainable development approaches (Picot-Allain et al., 2021). In this sense, green extraction technologies have been used for processing açaí berries, yielding several

products. Açaí berry oil, for example, has been extracted via supercritical CO_2 extraction at varied density, temperature, and pressure (Batista et al., 2015). These authors found that the highest global yield (45.4 ± 0.58%) was obtained for a CO_2 density of 900 kg/m^3, a temperature of 70 °C, and a pressure of 490 bar. Additionally, açaí berry oil presented a low saturated/unsaturated ratio with the highest concentration of monounsaturated fatty acids than polyunsaturated fatty acids, which is a positive feature. Açaí berry oil also showed a phytotoxic effect (inhibition of seed germination) on plants of the species *Mimosa pudica*, which suggests that it could be used for weeding control (Batista et al., 2015). Bioactive compounds were found at high concentrations in the extracted oil as well. Thus, supercritical extraction showed a feasible way to produce açaí berry oil with high nutritional value.

In another study, a phenolic compound rich extract was obtained from açaí pulp, seed, and slurry utilizing pressurized microwave-assisted extraction (Buratto et al., 2019). Silica aerogels were produced with the extract. The solvent used was ethanol/water (1:1). Results showed that phenolic compounds extracted from all açaí fractions effectively impregnated the aerogels, protecting them from the deleterious effect of high temperature. Additionally, phenolic acids were identified in the aerogels. Using the same green extraction technology, it was obtained açaí extracts from seeds and pulp, which were encapsulated with biopolymers by means of supercritical anti-solvent process. They discovered that using ethanol as solvent and polyvinylpyrrolidone (PVP) as biopolymer resulted in systems with the highest total phenolic compounds content and best microscopic features. In summary, pressurized microwave-assisted extraction was determined as a viable method for generating açaí extracts that effectively permeated and encapsulated matrices capable of safeguarding and releasing bioactive compounds for applications in food and cosmetics (Buratto et al., 2021).

A green extraction of phenolic compounds from açaí berries toward phenolic compounds concentration and antioxidant activity was optimized by Hanula et al. (2020). They discovered that the highest total phenolic and flavonoid content was obtained using ultrasound extraction at 37 kHz frequency, 100% amplitude, and 45°C for 25 or 45 min. On the other hand, the highest anthocyanin content was observed for ultrasound extraction at 25 °C for 25 min and microwave extraction (360W) at 25° C for 2.00, 3.16, or 4.33 min. They concluded that green extraction technologies produce extracts with higher bioactive content in shorter time and expending lower energy.

Pressurized liquid extraction (PLE) coupled with SPE was employed for extracting anthocyanins from semi-deffated açaí pulp (Maciel-Silva et al., 2022). Firstly, the pulp was deffated by supercritical fluid extraction (CO_2) to yield açaí berry oil as a co-product and improve PLE yield. Results showed that PLE was more efficient for extracting anthocyanins when a temperature of 71 °C and water at pH 2.0 (solvent) were used. Summarizing, PLE was found to yield an anthocyanin-rich açaí extract using acidified water as a green solvent. When PLE was coupled to SPE, the extract presented a higher concentration.

In brief, açaí berries are an outstanding source of compounds of interest to the food, pharmaceutical, cosmetic, and other industries. The comprehensive utilization of açaí berries has become a tangible reality, suggesting that they can be used as biomass in biorefineries. The use of green extraction technologies for processing açaí has already been reported, being an approach suggested for stimulating the Amazon bioeconomy. Being the place where the most açaí is produced in the world, the Amazon region can stop just exporting açaí and start industrializing it using sustainable technologies to add-value to the product. Instead of selling açaí pulp, it would be more interesting to sell its isolated bioactive compounds, such as anthocyanin. Such approach would change the economic scenario of the region, bringing development and income to the local population.

CUPUASSU (*Theobroma grandiflorum*)

General aspects

The cupuassu tree is native to the Amazon rainforest, specifically to the states of Pará, in Brazil. It grows to between 4 and 10 meters in height and yields drupes weighing an average of 1.500 grams. (Souza et al., 1999). In 2017, around 21.2 thousand t of cupuassu fruits were produced, amounting to about US$10.5 million (Brasil, 2017), highlighting that this value must be deducted from the production cost, which varies according to the region, the technology applied, logistics, among other factors.

Cupuassu has been widely used in many industrial segments in Brazil and abroad due to its pleasant flavor, making it one of the most popular Amazonian fruits worldwide. Such growth in the diversification of cupuassu consumption has made its production chain much more interesting economically, making the cultivation of the species a profitable alternative for local populations.

A review of the health advantages of Amazonian fruits emphasized the health promoting components of cupuassu and their benefits. Theobromine, volatile chemicals (aldehydes, ketones, and alcohols, ethyl butanoate, ethyl hexanoate, and linalool), unsaturated fatty acids, and flavonoids were identified in the fruit as being responsible for antioxidant and probiotic effects, as well as hypertriglyceridemia reduction (Avila-Sosa et al., 2019). Another work highlighted the antidiabetic and intestinal anti-inflammatory properties of cupuassu pulp (Assmann et al., 2021). Indeed, one of the studies cited in the review indicates that a proanthocyanidin-rich extract derived from cupuassu pulp can reduce diabetes problems caused by nitrosative stress and kidney inflammation (Punaro et al., 2019).

Another report suggests that a diet based on cupuassu pulp promotes intestinal anti-inflammatory activity (Curimbaba et al., 2020). Additionally, in vitro testing revealed that methanolic extracts of cupuassu pulp have inhibitory effect against uropathogenic *Escherichia coli* (Alcântara et al., 2021). Furthermore, an ethanolic extract prepared from cupuassu pulp presented a remarkable concentration of flavonoids with high antioxidant activity, suggesting that its consumption could help to prevent cardiovascular diseases, neurodegenerative disorders, and cancer (Carmona-Hernandez et al., 2021). Cupuassu juice was found to inhibit the enzyme CYP3A4, suggesting that its consumption could increase the bioavailability of drugs metabolized by this enzyme, ultimately improving the systemic exposure of orally administrated drugs (Costa et al., 2022). Flours prepared from cupuassu peel and seeds inhibited the enzyme α-amylase, which hydrolyzes starch, implying that their consumption may limit the flow of glucose into the blood, preventing diabetes (Andrade et al., 2022).

Cupuassu as a raw material for multiple purposes

Cupuassu fruits are specially used for producing cupuassu pulp, a remarkable product for the cupuassu production chain. The potential of cupuassu pulp has therefore been assessed. Several studies, for example, have identified pulp fragrance as distinct and pleasant (Augusto et al., 2000; Boulanger & Crouzet, 2000a, 2000b; Quijano & Pino, 2007). The compounds mainly responsible for cupuassu aroma are esters. Pasteurization somehow degrades them, producing cooked notes (Silva et al., 2000). Yet, pasteurization is considered a suitable way to assure pulp preservation at room temperature (Silva et al., 2003). Heating decreases the apparent viscosity of cupuassu pulp, which presents a shear-thinning behavior (Ferreira et al., 2008). The carbohydrates responsible for pulp viscosity are starch (Vriesmann et al., 2009), and pectin (Vriesmann & Petkowicz, 2009), which can act synergistically to produce gels (Vriesmann et al., 2010). According to (Rogez et al., 2004), cupuassu pulp has a pH of 3.4 and a dry matter content of 12.1%, with the primary components being sucrose (34.6%), dietary fiber (14.3%), and lipids (12.7%). The acidic pH predominantly results from citric acid (Hernandez et al., 2010). Cupuassu pulp also present bioactive compounds like flavonoids and ascorbic acid, which are somehow lost during frozen storage (Pugliese et al., 2013). The end of the shelf-life of frozen cupuassu pulp is attributable to enzymatic browning (Martim et al., 2013), although its microbiological quality is usually good (Sousa et al., 2020).

Several studies in which cupuassu pulp was used for producing food and pharmaceutical products were carried out over the last years. For example, there are reports on the processing of puree (Freire et al., 2016; Silva & Silva, 1999), nectars (Vieira et al., 2000, 2001, 2002), powders (Silva et al., 2008; Pombo et al., 2020), dried slices (Giraldo-Zuniga et al., 2010), juices (Teixeira et al., 2011), yoghurt (Costa et al., 2015), phenolic extracts (Barros et al., 2016), food emulsions (Teixeira et al., 2016), probiotic beverages (Pereira et al., 2017), fruit bars (Vallejo et al., 2017), functional beverages (Hernandez et al., 2018), healing biofilms (Matos et al., 2018), food packages (Melo et al., 2019), dairy beverages (Paula et al., 2021), and bread (Amorim et al., 2022).

Cupuassu seeds, a by-product from pulp extraction, contain phenolic compounds with antioxidant activity (Yang et al., 2003), essential metals (Naozuka & Oliveira, 2007), essential amino acids (Carvalho et al., 2008) and bioactive peptides (Cruz et al., 2016). They have been used for preparing products like cupuaçu chocolate (cupulate) (Caetano

et al., 2002); Cucaita et al., 2014), edible fats (Gilabert-Escrivá et al., 2002; Lannes et al., 2004), flour, protein concentrate, protein isolate (Carvalho et al., 2006), chocolate-likebeverages (Lannes & Medeiros, 2008), skin butter (Colomé et al., 2010), biodiesel (Pantoja et al., 2013), beta-carotene-loaded lipid nanoparticles (Gomes et al., 2017), cardioprotective aqueous extracts of fermented seeds (Fantinelli et al., 2017), nanoparticles for resveratrol release (Soldati et al., 2018) and for feminine hygiene products (Pohlmann et al., 2018), ingredients for feed (Silveira et al., 2019), phenolic extracts (Costa et al., 2020), catalysts for biodiesel production (Mendonça et al., 2019), ingredients for functional foods (Costa et al., 2020) and regenerative skin products (Barbalho et al., 2022).

Cupuassu liquor, i.e., crushed seeds after fermentation, sun drying, and roasting, present sensory characteristics that resemble cocoa liquor. However, their consumption results in superior health benefits (Oliveira et al., 2015; Oliveira & Genovese, 2013). Cupuassu seeds are now largely consumed as cupulate, a chocolate produced from them. Since 2015, the Brazilian Agricultural Research Corporation can use the term “cupulate” exclusively (Braga, 2014). **Fig. 3** illustrates the main steps to produce cupulate. Reports on this topic showed that seeds fermentation should be carried out preserving pulp that naturally involves the seeds, which was important for forming volatile compounds in cupulate (Ramos et al., 2016). Yeasts like *Pichia* and *Hanseniaspora* along with bacteria like *Acetobacter*, *Lactobacillus*, and *Bacillus* are the responsible for producing flavor compounds during fermentation (Ramos et al., 2020). A study on seeds roasting suggested that the temperature and time for producing the best aroma and preserving macronutrients are 115 ° C and 25 minutes (Criollo-Nuñez et al., 2020). Cupulate also contains cupuassu vegetable butter, obtained by pressing seeds (Nazaré et al., 1990). Cupuassu butter can be also used for producing cosmetics. It is composed mainly by oleic (38.8%), stearic (38.1%), palmitic (11.2%) and arachidic (7.9%) acids and could succeed in replacing fatty acids in the skin (Dourado et al., 2015). It is important to note that a few years ago, most cupuassu seeds were discarded as waste. Currently, the use of its seeds in the production of cupulate has made it as important as the pulp, making cupuassu a potential protagonist in the Amazon’s bioeconomy scenario.

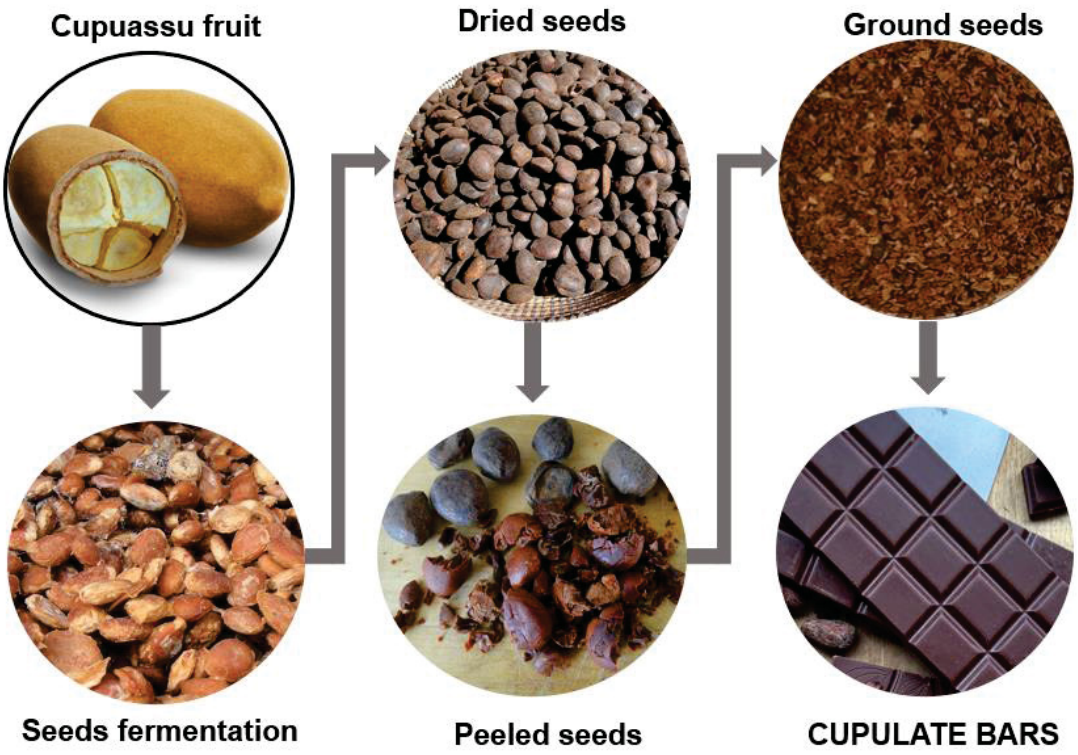

Figure 3 - Main cupulate production steps.

Other by-products resultant from cupuassu processing have been used for many purposes. The by-product from cupuassu pulp processing, which is composed of husks, pomace, and seeds, contains remarkable amounts of lipids (3.69%) and proteins (1.65%) (Séfora et al., 2011). A use proposed for this by-product was as a substrate to produce phenolic acids, invertases, and transferases by *Aspergillus carbonarius* (Barros et al., 2022). Cupuassu pomace was used to produce flour, which presented high phenolic content (403 mg GAE/100 g) and high antioxidant activity (146.9%; DPPH method) (Freitas et al., 2017).

The by-product resultant from cupuassu oil extraction from the seeds by mechanical pressing (cupuassu cake) was used as an ingredient of feed for buffaloes in the study conducted by Silva et al. (2021). The results demonstrated that cupuassu cake had no deleterious impact on the rumen or the mammary glands of the animals. Additionally, the quantity and type of unsaturated fatty acids contained in the milk produced by buffaloes fed with cupuassu cake suggests that it is a suitable ingredient for feed for such animals. Cupuassu cake is also called cupuassu seed by-product. It has also been used for preparing ethanolic extracts, which were found to contain mainly terpenes, steroids, and phenolic compounds, especially flavonoids (Costa et al., 2022).

The cupuassu shell, until some time ago, used to be discarded as waste; however, it is currently being used for several purposes in various industrial sectors. As it makes up about 43% of the total weight of the cupuassu fruit, the shell has also been generating interest from researchers and companies that wish to undertake in the bioeconomy sector in the Amazon region. **Table 1** shows some of the applications of the cupuassu fruit shell that can add value to the productive chain of this fruit.

Table 2 - Application of cupuassu fruit shell for multiple purposes.

Cupuassu shell form	Application	References
Biosorbent	Removal of textile dyes.	(Cardoso et al., 2011)
Flour	Bread preparation with good nutritional and sensory properties.	(Salgado et al., 2011)
Substrate	Production of edible mushrooms.	(Machado et al., 2016)
Substrate	Endoglucanase production	(Falcão et al., 2022)
Acid hydrolysis material	Obtaining added-value products like furfural, levulinic acid and hydroxymethylfurfural.	(Marasca et al., 2022)
Pyrolysis raw material	Production of useful products like alcohols, aldehydes, ketones, organic acids, aromatic and aliphatic hydrocarbons	(Alves et al., 2022)

Green technologies applied to the cupuassu and its biorefinery prospects

Biorefinery using cupuassu as biomass has been proposed by Cerón et al. (2015). Results showed that cupuassu pulp could produce pasteurized pulp, antioxidant extract obtained by supercritical extraction, and essential oil extracted by steam distillation. In addition, seeds could be used to produce oil and ethanol. Finally, by-products resultant

from pulp and seeds processing could be used to produce poly-3-hydroxybutyrate, a biopolymer.

Other reports on the application of green extraction technologies to cupuassu are available. For example, using a supercritical solvent, fat was recovered from fermented cupuassu seeds (De Azevedo et al., 2003). Results showed that ethane was superior to CO_2 as solvent due to its higher affinity for fats. In addition, Mosquera et al. (2014) extracted fat from cupuassu seeds through microwave-assisted extraction, finding that extracts presented a better fat quality (higher PUFAs content) when compared to conventional solvent extraction methods. Finally, Herrera et al. (2014) extracted antioxidant compounds from dry cupuassu seeds using microwave-assisted extraction, discovering that a higher yield was obtained when using solvent extraction ratio of 1:15, power of 300 W, extraction times of more than 10 min and more than three extraction cycles. The latter presented the highest effect on bioactive compounds extraction.

BURITI (*Mauritia flexuosa*)

General aspects

Among the Aracaceae family, *Mauritia flexuosa L*. is considered one of the most abundant Brazilian palms. It is known in Brazil as “Buriti” and can be found in different states, mainly in the Amazon states such as Pará, Amazonas, Amapá, and Rondônia. This palm can reach about 40 m in height and its fruits, measuring 5-7 cm in length and 4-5 cm in width, can weigh up to 85 g. Its edible pulp is orange-brown (it has a sweet and earthy flavor), and its inner endocarp is white and fibrous (Fontes et al., 2006). According to the Brazilian Institute of Geography and Statistics (IBGE), the buriti production in 2022 reached 422 tons (IBGE, 2022), and the pulp of the fruit costs an average of R$ 6.00 in a survey at regional fairs in Belém, Pará state. An adult palm can produce up to 200 kg of fruit, which can be processed into 30 kg of flour or 5 to 6 liters of finally extracted oil (the flour contains 22% oil). With 60 buriti palms per hectare, 300 to 360 liters of oil can be recovered per hectare (Morais & Gutjahr, 2012).

On average, despite the variation in the origin of the fruit (Rudke et al., 2021) buriti is composed by 39.99% seed, 38.31% pulp and 21.70% shell. The industrial applications are more related to the fruit pulp, which has a predominance of moisture (50.5 to 79.35%) followed by lipids (9.03 to 22.17%) and carbohydrates (2.10 to 26.20 %). This fruit is rich in antioxidant compounds and fibers, which may vary in chemical composition according to the region or climatic conditions in which it is found (Cândido & Silva, 2017; Milanez et al., 2018).

Buriti, due to its antioxidant compounds in its composition, presents itself as a potential raw material to combat pathologies related to reactive oxygen species. Liposoluble bioactive compounds (for example, carotenoids, being β-carotene and tocopherols) and unsaturated fatty acids, such as oleic acid, are the main compounds of interest present in Buriti mesocarp oil (Pereira et al., 2022). The oil extracted from the fruit is mainly composed of oleic and palmitic fatty acids, which can help prevent cardiovascular diseases (Milanez et al., 2018). β-carotene is the main bioactive compound found in Buriti (~70% of carotenoids), playing an essential role in the human diet as a pro-vitamin A to fight reactive oxygen species and decrease skin cancer (Barreto et al., 2021). Some studies point to anti-inflammatory and antimicrobial activity due to the presence of compounds such as phenolic compounds (Nobre et al., 2018).

Buriti as a raw material for multiple purposes

The impact of cellular oxidative stress on human health caused by reactive oxygen species (ROS) has led the consumer market to seek new natural products rich in antioxidant compounds. Despite the potential of micronutrients and helpful active compounds to improve consumer health and application in the manufacturing of added-value products, buriti is an Amazonian fruit that has received little industrial attention in comparison to other regional commodities (Barboza et al., 2022; Rudke et al., 2021).

In the food industry, the primary use of the Buriti fruit is in the form of juice, sweets, ice cream, popsicles, jellies, and wines, and its by-products can be used as additives to improve the nutritional value and properties of processed food (Resende et al., 2019). In the cosmetics industry, buriti oil can be used as a sunscreen formulator due

to its antioxidant capacity to prevent and eliminate alterations caused by free radicals produced by solar radiation (Mansur et al., 2020).

Buriti, due to its functionalities promoted by its antioxidant compounds, is an Amazonian matrix with potential application for the development of new technological industrial products in the food, pharmaceutical and cosmetic sectors, as shown **in Fig. 4.** To reach this level of application, studies are being developed to optimize its extraction method and, thus, maintain its functional properties and increase its range of industrial application. An example is the case of the research developed by Pereira et al. (2022), where they verified that the carotenoid content in oleosomes emulsions was more stable than the carotenoids in bulk Buriti oil during 28 days of storage. Another study developed by Silva et al. (2022) found that high-intensity ultrasound combined with a chemometric approach was up to 2x more effective for extracting carotenoids from buriti pulp than conventional methods. These and other research show an interesting alternative for the conventional extraction of buriti oils for new pharmaceuticals, cosmetics and functional foods, as they allow greater stability and availability of bioactive compounds of interest (Leite et al., 2021a; Leite et al., 2021).

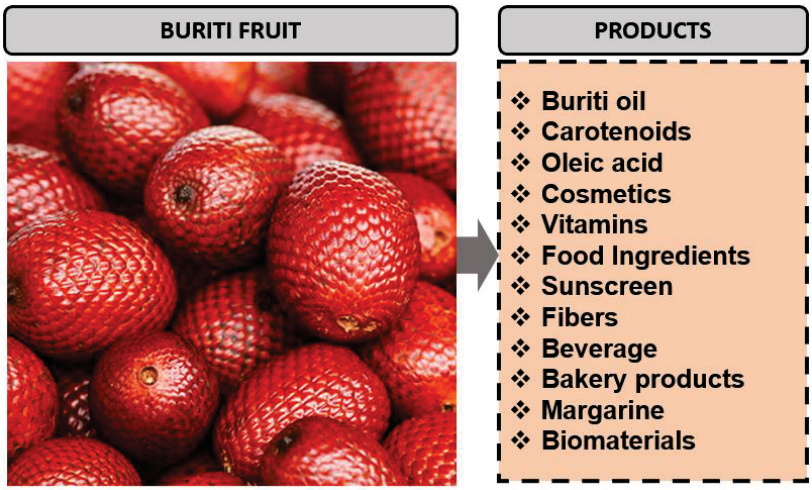

Figure 4 - Buriti fruit and its products.

In addition to the previously listed Buriti applications, additional uses for the fruit are reported. Borgonovi et al. (2021) identified that Buriti pulp was able to increase the antioxidant activity (6 to 8%) and probiotic potential of fermented milk. Buriti is commonly identified by its high availability of fibers that assist in the function of the intestinal microbiota, which can directly influence chronic diseases such as diabetes and obesity (Barboza et al., 2022). In the same beverage segment, Bovi et al. (2017) developed

an isotonic sports drink by adding emulsions extracted from Buriti to replace synthetic dyes with carotenoids present in buriti oil, promoting the same pigmentation action and incorporating antioxidant activity.

Seeking to develop products with low saturated fat content, Mesquita et al. (2022) produced meat products with Buriti oil with potential application for hamburgers, sausage, meatballs, and others. The researchers observed that partial substitution modified the fatty acid profile's composition, increasing the monounsaturated fatty acids' content and decreasing the total saturated fatty acids. Braga-Souto et al. (2022) and Aquino et al. (2016) developed bakery products based on Buriti flour and oil to increase the injection of Vitamin A, dietary fiber, and minerals. Both studies identified improvements in the composition and sensory characteristics of the products. Buriti seeds have a wide range of minerals, including K (809.88 ± 22.87 to 1042.92 ± 33.60 mg 100 g^{-1}), Na (80.25 ± 2.08 to 231.48 ± 39.14 mg 100 g^{-1}), Mg (52.37 ± 5.46 to 67.65 ± 5.99 mg 100 g^{-1}), Ca (27.19 ± 0.84) (Vásquez-Ocmín et al., 2010) and others. An interesting application is observed as an enricher of flours, cereals, cookies, and other similar products.

Buriti oil possesses a noteworthy application within the margarine industry and in heat-treated foods, owing to its rich oleic acid content, which enhances the product's nutritional value. Additionally, the inherent thermal stability of oleic acid, coupled with its synergy with antioxidants, imparts higher resistance to oxidation to the oil (Cruz et al., 2020).

Green technologies applied to buriti and its biorefinery prospects

Traditional refining processes are known to be inefficient for increasing the amounts of compounds of industrial interest in Buriti, as they reduce the levels of vitamin A, unsaturated fatty acids, and antioxidant activity in comparison to crude buriti oil (Aquino et al., 2012). As a result, the viability of adopting green extraction techniques is real and may be improved. When comparing green extraction methods or the usage of biorefinery applied to Buriti in comparison to other matrices, there is much to be investigated in terms of the extraction of chemicals of industrial interest.

Supercritical CO_2 was used in buriti fruit, where the combined action of pressure (20 to 30MPa) and temperature (313 to 328K) were able to extract about 80% of the initial carotene content (França et al.,1999). Barreto et al. (2021) also studied an ecologically correct system with supramolecular solvent (SUPRAS) formed by aggregates of octanoic acid, which obtained extraction of β-carotene and phenolic compounds (gallic acid, quercetin, and catechin). Furthermore, when combined with the antibiotics norfloxacin and gentamicin, the extracts showed synergistic action against multidrug-resistant strains of *Staphylococcus aureus, Pseudomonas aeruginosa* and *Escherichia coli.*

Given the recovery of antioxidant compounds from buriti bark by green methods, Rudke et al. (2019) evaluated the antioxidant potential of buriti bark extracts obtained by pressurized liquid extraction (PLE) with ethanol/water mixtures. The yields with the best results ranged from 16.82 to 25.16%, total carotenoids from 23.38 to 1056.59 μg equivalent of β-carotene g^{-1}, total phenolic content from 143.37 to 172.02 mg gallic acid equivalent g^{-1}, DPPH from 31.04 to 48.62 μg mL^{-1} and ABTS from 1.87 to 2.70 mmol TEAC g^{-1}.

Assis et al. (2021) evaluated choline chloride-based deep eutectic solvents (DESs) as co-solvents for the ethanolic extraction of total carotenoids from buriti fruits. It was identified that using DESs did not increase the yield of ethanolic carotenoid extraction. However, it was observed that the buriti bark could be a rich source of carotenoids with yields comparable to the fruit. Thus, green solvents were suggested to increase the recovery of total carotenoids from the buriti fruit by an ecologically correct process.

In this framework, it is possible to observe the potential for extracting compounds of industrial interest present in the bark, seed, and pulp of buriti by green techniques, generating possibilities for biorefinery application. Thus, it is suggested that new green or ecologically correct methods be evaluated to increase the extraction or recovery of these compounds. In what is raised, biorefinery platforms based on supercritical and/or pressurized fluids are presented as good alternatives for exploring this matrix and encourage the bioeconomy of the Amazon.

BRAZIL NUT (*Bertholletia excelsa*)

General aspects

Brazil nuts (BNs) are nuts extracted from *Bertholletia excelsa*, a tropical tree belonging to the *lecythis* family (*Lecythidaceae*) that can reach over 150 feet. Therefore, it is one of the largest trees found in South America, where it grows and bears fruit, almost specifically in dense natural areas with low concentrations of individuals (Baldoni et al., 2020).

In addition to NBs being excellent natural sources of Selenium, they also have high nutritional value and antioxidant and anti-inflammatory properties due to the presence of unsaturated fatty acids, high-value proteins, dietary fiber, minerals, phenolic compounds, tocopherols and others (Hou et al., 2021; Vasquez et al., 2021). The protein content of BN is also remarkable, with 15-17% protein by fresh weight. BN 2S albumin, called Ber e 1, comprises about 30% of the total protein and is exceptionally rich in sulfur amino acids (De la Cruz et al., 2013).

Among the benefits to human health, BN promotes a decrease in cardiovascular risks in obese people (positively influencing the lipid profile and nutritive microvascular reactivity) by decreasing triglyceride and cholesterol levels (Cominetti et al., 2012; Hou et al., 2021; Maranhão et al., 2011), oxidative stress (Stockler-Pinto et al., 2010), cancer prevention (Ip & Lisk, 2009; Yang, 2009) and improved cognitive functions (Rita Cardoso et al., 2015)

Its significance is also attributed to its extraction and exportation, which offer a substantial source of income for the local population. According to data on extractivism of natural plant resources (IBGE, 2020), BN had a production value growth of 3.7% in 2019, resulting in R$ 135.8 million. The State of Amazonas remains in the national leadership, with 12.2 thousand tons of the product, approximately 37% of the total produced in the country. In 2019, the price of Brazil nuts in shell had an average price of R$ 4.12.

Brazil nut as a raw material for multiple purposes

As BN is a food rich in bioactive compounds and micronutrients, there is a high potential as a raw material for functional products. Nonetheless, similar to other sources of antioxidant and anti-inflammatory compounds, a concern arises regarding the extraction of these compounds and the preservation of their functional quality for practical application. In this sense, Gomes & Torres (2016) optimized the extraction of antioxidant phenolic compounds from Brazil nut cake and, thus, it was achieved under the following conditions: ethanol-water (40:60; v/v); 2.5 min homogenization; and 1 h of extraction at 60°C. According to these researchers, the concentrations of phenolic compounds ranged from 70.0 to 421 $mg.kg^{-1}$. Gomes et al. (2019) evaluated the microencapsulation of Brazil nut extract by spray-drying, using starch modified with octenyl succinic anhydride (OSA starch) and inulin. The authors developed a functional powder with stability for up to 120 days, providing an interesting ingredient for functional foods.

In the food industry the main goal is to use BN to produce different products, such as cereals, chocolate bars, ice creams, beverages, meals, breads and others (De la Cruz et al., 2013). In order to generate functional products without the need for advanced technologies, Lima et al. (2021) developed nutritional bars based on Brazil nuts and baru almonds as raw materials. In addition to maintaining nutritional characteristics and good sensory acceptability, the production technology of nutritional bars can be transferred to cooperatives and can be carried out by small industries. Additionally, to reduce the unpleasant or rancid taste of soy-based products, Barbosa et al. (2020) elaborated mixed formulations of fermented beverages with different combinations of water-soluble soy extract and water-soluble extract of Brazil nut. All formulations developed were considered excellent substrates for the growth of lactic acid cultures. The formulation containing 75.0% SHE and 25.0% BNHE proved to be the most viable as it presented the best technological properties.

In the bioenergy field, Colpani et al. (2022) et al. evaluated kinetic and thermodynamic parameters of BN (shell and husk) production residues. The utilization of Brazil nut residues as raw materials for renewable energy sources has demonstrated suitability, and the prospect of their widespread application holds significant potential to contribute to the circular bioeconomy.

Green technologies applied to brazil nut and its biorefinery prospects

In terms of biorefinery and green extraction methods applied to BN culture, it is possible to observe a dearth of research. Nutritionally, Brazil nuts are source of nutrients, including protein, fiber, selenium (Se), magnesium, phosphorus, and thiamine. They also contain niacin, vitamin E, vitamin B 6, calcium, iron, potassium, zinc, and copper. Nuts have high nutritional value, containing 60-70% oil and 17% protein. Furthermore, the oily endosperm contains about 70% unsaturated fats, which can also lead to rancidity problems (Yang, 2009). Thus, it is clear that due to its particular composition, mainly with Se availability and oil content, green methods in the BN matrix can be used to extract compounds of industrial interest.

Among the few approaches, Santos et al. (2013) studied the supercritical extraction of Brazil nut oil. The researchers evaluated the global yield isotherm, which presented optimal conditions at 60 °C and 300 bar, and composition analysis showed a predominance of unsaturated fatty acids, especially ω-3, 6, and 9 fatty acids.

The extraction of oil from the by-product of the production of BN beverages by supercritical carbon dioxide (SC-CO_2) was developed by Vasquez et al. (2021). These authors identified high yields (about 100% oil recovery) under conditions of 400 bar and 60°C. In the condition of 200 bar and 60 °C, it was possible to obtain better levels of squalene and β-sitosterol (590.8 and 283.9 mg/g of extract). Santos et al. (2013) analyzed the yield, nutritional quality, and thermal-oxidative stability of BN oil through supercritical extraction with CO_2 and similar conditions of pressure and temperature (300 bar and 60 °C) generated optimal conditions for extracting the oil (a predominance of unsaturated fatty acids, specially ω-3, 6, and 9 fatty acids). Thus, the applied method was presented as an alternative for recovering compounds of interest for application in food, cosmetics, or nutraceuticals.

Zanqui et al. (2020) evaluated the extraction of BN oil using subcritical propane compared to conventional extraction methods. The researchers concluded that the green method was able to bring advantages to the extraction process, such as the non-use of toxic solvents, reduction of extraction time, and maintenance of the main chemical constituents (in terms of fatty acids, triacylglycerols and phytosterols) of the oils to maintain the main nutritional properties.

The BN oil extraction method by ultrasound was used by Schons et al. (2017). It was possible to obtain a high-quality oil with a predominance of linoleic (36.5%), oleic (32.1%), and palmitic (15.3%) fatty acids. The researchers concluded that the proposed method for obtaining BN oil using ultrasound-assisted extraction proved to be simple, fast, and inexpensive.

Other emerging methods of green extraction have yet to be explored, such as microwave and enzymatic extractions. These emerging methods stand as new means to replace conventional approaches to obtain extracts with higher nutritional quality in parallel with the reduction of environmental impacts.

PEACH PALM (*Bactris gasipaes*)

General aspects

The palm tree of the *Arecaceae* family, known as Peach palm (*Bactris gasipaes Kunth*) (Brazilian name: pupunha), is one of the most widespread trees of this family in the Amazon region. Therefore, the states of northern Brazil are the largest producers of its fruits. These fruits are related to traditional extractivism and have several varieties: red, yellow, orange, green, and even white, as illustrated in **Fig. 5** (Chisté et al., 2021; Santos et al., 2022).

Figure 5 - Different varieties of peach palm.

Despite the peach palm being native to the Amazon region, the world's largest explorers of products derived from this species are the Brazilian states of São Paulo, Paraná, Santa Catarina, and Bahia. The fruit pulp usually has about 72% of the fruit weight, followed by the seeds (21%) and peel (6%) (Chisté et al., 2021). According to Felisberto et al. (2020), it is only known that fruit production has a balance of 5 to 10 bunches per year/plant, and each bunch can contain up to 12 kg of fruit. Thus, the harvest of 1 hectare can reach up to 10 tons of fruit/year. According to IBGE (2017), Brazil produced 8,873 tons of peach palm in 2017, with a sales price of approximately BRL 3.12/kg.

Regarding its nutritional value in the mesocarp of the fruit, it is known to have energy values (273.5 cal/100 g), niacin (0.81 mg/100 g), vitamins C (18.7 mg/100 g), B1 (thiamine) (0.045 mg/100 g), B2 (0.135 mg/100 g) and A (1.1 mg/100 g) (Felisberto et al., 2020). Furthermore, like other fruits of its family, the peach palm is a source of carotenoids, of which 20 to 24% correspond to β-carotene, and 14% are related to lutein, and phenolic compounds (flavonoids, 34% of which correspond to schaftoside) (Chisté et al., 2021).

Peach palm as a raw material for multiple purposes

In the Amazon region, the fruit is sold at community fairs and regional shops, and it is primarily consumed by heating it in salted water, fermenting it in a thick drink, or drying and transforming it into flour. Its palm heart is also used as a source of natural fiber. Regarding its industrial application, due to its important nutritional and energy values, peach palm is useful for the production of extruded foods (Carvalho et al., 2010), formulation of different cookies, cakes, and meat products, breaded and even for specific consumers as it is a healthy food matrix (Pires et al., 2019).

Due to its beneficial properties to the human, as it consists of carbohydrates, lipids, proteins, minerals, and compounds with antioxidant properties, the fruit and its by-products, such as its seeds, have been the subject of research in several sectors for its use in pharmaceuticals and cosmetics (Pinheiro et al., 2022). Concerning peach palm residues, they present a substantial economic potential due to a large amount of other natural compounds of interest, such as flavonoids, nanocellulose, fibers, and fatty acids, which present substantial benefits to the health of consumers (Radice et al., 2014). Furthermore, biomass from these residues could be converted into fuel, electricity, heat, high-value chemicals, and other bio-based products to support green policies (Kee et al., 2022).

In this context, Franco et al. (2019) extracted nanocellulose from peach palm waste. For these authors, based on the results obtained, it is possible to state that agricultural residues from peach palm extraction can be processed to obtain nanocellulosic material with morphological, chemical, and physical properties that allow its application in several areas, including the formation of stable emulsions for use in foods, medicines, and cosmetics, as well as physical reinforcement in the formulation of biopolymer compounds.

Green technologies applied to the peach palm and its biorefinery prospects

Espinosa-Pardo et al. (2014) applied supercritical fluid technology to obtain extracts rich in carotenoids from peach palm, and the best operating extraction condition

was 300 bar and 40 °C, which presented antioxidant activity comparable to commercial caffeic acid. The authors suggest that this product can be used as an active ingredient in pharmaceuticals as a precursor to vitamin A for people who are deficient in this vitamin, in the control of oxidation processes in food processing acting as an antioxidant, and in the cosmetic industry for photoprotection to reduce severe photosensitivity in patients with some dermatological diseases.

Giombelli et al. (2020) evaluated the valuation of residual material, a portion of the peach palm stem, through the subcritical extraction of soluble sugars and phenolic compounds in water. The authors identified that under maximized conditions (100 bar, 130 °C and 90 min), high concentration of phenolics and monosaccharides, mainly gallic, hydroxybenzoic and chlorogenic acids, were extracted.

Ultrasound-assisted extraction of total carotenoids from peach palm bark was evaluated by Ordóñez-Santos et al. (2015). The method applied to the by-product proved to be efficient in extracting carotenoids (163.47 mg/100 g) under ultrasonic intensity of 1528 W/m^2, temperature of 35 °C and time of 30 min. In addition to enabling high extraction of compounds of industrial interest, the authors highlighted the method's applicability due to its low cost compared to other more complex green methods.

In addition to using green methods for extracting bioactive compounds of industrial interest, there is an emphasis on converting peach palm crop residues into bioenergy, biofuel, and biomaterials using biorefinery concept. This possibility lies in the fact that these residues have a high composition of lignocellulosic material (Franco et al., 2019). According to Philippini et al. (2020), agro-industrial by-products can be used as raw material in biorefineries for the release of sugars, proteins, oils, and other micronutrients, which can be employed for the development of different media compositions.

FUTURE TRENDS AND BUSINESS OPPORTUNITIES

The investment scenario in the bioeconomy sector in the Amazon is encouraging due to government incentives, the wide diversity of species, the availability of raw materials in the region and the improvement of logistics infrastructure. According to Nobre & Nobre (2019) , more than 240 plant species are used as the basis for products

from various industrial segments. Still, the potential is infinitely superior since the Amazon holds the most extraordinary biodiversity in the world. The bioeconomy can create additional value for the local economy through the utilization of standing forests. Strand et al. (2018) indicate a potential value of around US$ 7 trillion for the Amazon Forest keep standing, with rational use of its biological and ecosystem resources. Coslovsky (2021) points out that about sixty products compatible with the standing forest included in the Brazilian export basket moved a total of US$ 298 million, representing less than 0.2% of the calculated market for them, which demonstrates the magnitude of space to advance in the development of productive chains of socio-biodiversity.

For example, the profitability per hectare of a biodiverse environment with managed açaí production is estimated to be over US$ 1500, compared to approximately US$ 200 for a hectare of soy cultivation (Nobre & Nobre, 2019), without considering the increase in job creation and related ecosystem services. With the advent of new studies, logistics improvement and discoveries of new technologies, this value tends to increase even more, as the high-added-value compounds extracted from this fruit and its by-products are capable of substantially adding value to the productive chain of this species.

According to Nobre (2021), the basic premises are: innovation of processes and technologies of the 4th industrial revolution, environmental conservation, appreciation of local culture and traditional knowledge of local populations. For example, cocoa as a commodity is sold for 3 dollars per kilogram, but when transformed into chocolate, it can reach 60 dollars for the same amount. It is worth highlighting that there is a production and logistics cost, which must be taken into consideration. A transformation based on innovation enables complex processes to take place in the unlikely conditions of the interior of the Amazon.

The interest in national and international products with high added-value obtained from Amazonian raw materials demonstrates an excellent business opportunity in the region. Applying new extraction technologies, these components can be extracted and sold separately for the national and international markets. This reduces logistics costs and promotes the valorization of the production chain. Açaí is an example of a raw material that fits into this context, since its main bioactive molecule, anthocyanin, can be extracted in the Amazon through clean technologies and sold in isolated form in the internal and external markets. Buriti (*Mauritia flexuosa*), considered one of the greatest sources of

beta-carotene in the world, is also an example of raw material with investment potential in the region and all other raw materials detailed in section 3 of this work.

Techno-economic evaluation of the extraction of value-added components from some raw materials reveals that the use of green extraction technologies presents encouraging data, providing higher quality and lower production costs when compared to extracts obtained using conventional techniques. For example, Ochoa et al. (2020) demonstrated that the production cost for extracting anthocyanins from purple yam (*Dioscorea alata*) by ultrasound-assisted extraction (UAE) was US$ 124.08 kg^{-1}, while for low-pressure solvent extraction (LPSE) it was US$ 263.65 kg^{-1} and for soxhlet extraction it was US$ 765.13 kg^{-1}. According to the sensitivity study, the extraction of anthocyanins from purple yam by LPSE and Soxhlet is not viable in the range of sales prices evaluated in the work. However, the extraction of anthocyanins from purple yam by UAE is economically attractive when the selling price is higher than US$170 kg^{-1}. It is worth mentioning that this production price obtained by UAE was estimated through the scale-up study, where the production price fell from US$ 950.52 kg^{-1} to US$ 124.08 kg^{-1} when the extractor capacity increased from 5 to 500 liters, demonstrating that increasing the scale of green extraction processes significantly reduces the cost of production.

Best et al. (2021) carried out a techno-economic evaluation for the production of oil and phenolic-rich extracts from buriti fruit using sequential supercritical (SFE+CSE) and conventional solvent extraction (CSE). The authors demonstrated an approximately 2-fold reduction in cost of manufacturing in the best scenarios studied, showing that SFE+CSE is economically more advantageous for obtaining bioactive compounds from buriti pulp on an industrial scale when compared to the conventional extraction technique. In a similar way, Hasanov et al. (2023) carried out the techno-economic evaluation of supercritical fluid extraction of flaxseed oil by computational software Super Pro Designer®, demonstrating that high scales of SFE systems are feasible due to the high productivity. Although there is a need for a high investment for large-scale extraction systems, it is possible to obtain low cost of manufacturing and a short payback time. Through the works mentioned, it is possible to infer that increasing the scale of production can cause a reduction in manufacturing costs, making investments more attractive.

Additionally, in recent years, government agencies such as the BNDES (Brazilian Development Bank) have been making increasing efforts in favor of sustainability,

encouraging investments that contribute to this agenda and encouraging companies and other partners to adopt better socio-environmental practices that promote the socio-bioeconomy of the Amazon region (Pamplona et al., 2021). The relevant number of proposals submitted to funding notices and the companies' initiative to finance innovation reveal a somewhat encouraging scenario for the coming years. In addition, the approximation of companies to the universities has been generating good results in developing and applying new technologies.

Therefore, the advantages of investing in an industry focused on sustainable green processes in the Amazon are diverse. First, logistics are facilitated, as the raw materials are acquired in the region, reducing conservation and transport costs from where the raw material is obtained to the factory where it will be processed. Second, as previously mentioned in this work, there is qualified labor in the region that can be recruited to assume strategic positions in these biofactories, in addition to having a population with a lot of traditional knowledge that can be used symbiotically with scientific knowledge, promoting the socio-bioeconomy. Consequently, this will lead to recognizing individuals who were born and raised in these areas and are already acclimated to the region. Furthermore, there is a rising influx of incentives provided by government agencies to stimulate fresh investments in the bioeconomy sector within the Amazon. This can facilitate the implantation and establishment of industries in this sector through incentives of different modalities. Finally, the Amazon has an immense biodiversity of species, which makes it possible to process several raw materials at the same time and, with this, to obtain several products that can serve several different market niches, thus increasing the scope of the bioindustry.

Yet, it is important to highlight that companies need to be committed to building a green future, avoiding the practice of greenwashing, which can have many negative consequences for businesses, such as the loss of consumer trust, generating decline in sales, damage to reputation and regulatory scrutiny. There must be a commitment from all actors involved in the bioeconomic scenario, valuing the real sustainable development.

CONCLUSION

The application of green extraction technologies aimed at developing the bioeconomy in the Amazon is a promising way to improve the current scenario in the region since they are environmentally friendly technologies that meet social, economic, and environmental demands. Prospects for sustainable business creations involving açaí, cupuassu, brazil nut, buriti, and peach palm fruits are encouraging since they are raw materials that generate different products, enabling the application of the biorefinery model. For this, it is essential to establish an institutional arrangement that makes it possible to address all limitations, whether regulatory, infrastructural, or technical, in an articulated and synergistic manner with the public and private sectors. Thus, the Amazon will become more attractive to investors who wish to undertake sustainably in the region, adding value to the production chain of local raw materials, keeping the forest standing, valuing the knowledges of traditional communities, generating income, regional development, and consequently improving the population lives.

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CHAPTER II

COMPRESSED PROPANE EXTRACTION OF UMARI PULP OIL: A RICH AMAZON SOURCE OF β-CAROTENE AND OMEGA-9

This chapter is part of the article entitled “Compressed propane extraction of umari pulp oil: a rich amazon source of β-carotene and omega-9” **PUBLISHED** in *The Journal of Supercritical Fluids*.

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COMPRESSED PROPANE EXTRACTION OF UMARI PULP OIL: A RICH AMAZON SOURCE OF β-CAROTENE AND OMEGA-9

ABSTRACT

Compressed propane extraction (CPE) was applied to umari fruit (*Poraqueiba sericea* Tul.) pulp to obtain an oil rich in high added-value components. CPE was performed at different temperatures and flow rate, which was compared to conventional extraction (Soxhlet). The oils were analyzed for global yields, fatty acid composition, β-carotene content, antioxidant activity, total phenolics content (TPC), total flavonoids (TF), and thermal behavior. The highest extraction yield (29.2 wt%) was obtained with CPE at 80 °C and 3 mL min^{-1}, being higher than that obtained by Soxhlet (27.8 wt%). The flow rate variation had a significant effect ($p < 0.05$) on the oil extraction yield, while the temperature had a negative effect. The umari oil obtained by CPE emerges as a rich source of oleic acid (omega-9) and β-carotene (vitamin A precursor), with potential to be applied in various industrial segments and inserted into the Amazon bioeconomy scenario.

Keywords: Oil, Bioeconomy, Green extraction, Kinetics, Amazon.

INTRODUCTION

The Amazon region holds the greatest biodiversity in the world, representing around 60% of the Brazilian territory [1]. However, many of its species are still unexplored in the scientific and industrial sphere. In this context, the umari fruit (*Poraqueiba sericea* Tul.) appears as a species with good technological potential as it presents components that favor its use at industrial levels, such as its high oil content and the presence of value-added secondary metabolites. Umari fruit, belonging to the *Icacinaceae* family, is an edible fruit, very aromatic and composed of an oil-rich pulp containing approximately 65% unsaturated fatty acids, being widely consumed by the Amazonian population, mainly in its fresh form [2,3]. However, it still does not have comprehensive technological use capable of expanding and enhancing its production chain, being considered an undervalued fruit, which is sold at a very low cost.

In the current bioeconomy scenario, the application of green technologies emerges as an important tool for the development of the Amazon region and, consequently, the valorization of the production chain of several regional species that have economic-industrial potential [4,5]. Among these technologies, extraction techniques using high pressures stand out, which have been applied to replace conventional methods to optimize extraction, reducing process time and preserving thermolabile compounds using mild temperatures [6–8]. These technologies associated with selective solvents enhance the extraction of high-quality components. Extractions under compressed conditions are also associated with a reduction in energy consumption and the elimination of post-processing steps [9,10].

In this sense, compressed propane is a solvent that has been widely tested for extractions of natural compounds due to its solubility in vegetable oils. Extractions using compressed (or pressurized) propane have achieved effective results, using a small amount of solvent in a short period of time. The propane is a solvent that does not leave residues in food, which is one of its advantages for use in the extraction of natural products. As a gas, propane quickly evaporates and does not remain in the oil obtained after extraction, which is why it has been widely studied as an alternative green solvent for obtaining natural and edible products [9–12].

High-pressure extraction technologies typically provide high-quality extracts, preserving value-added components such as antioxidants, vitamins and pigments [7,13]. Despite few studies available in the literature, there are reports that the umari fruit has

relevant levels of total phenolic compounds and β-carotene (provitamin A), presenting potential antioxidant activity [3,14]. Umari oils obtained by conventional extraction methods presented a composition of fatty acids with a high content of oleic acid (~65%), a monounsaturated fatty acid (omega-9), which has several benefits for human health, helping to reduce risks of cardiovascular diseases and diabetes control [2,3].

Thus, considering the lack of studies in the literature addressing the use of high-pressure extraction technologies, mainly applying compressed propane as solvent to obtain value-added components from umari, and considering its biological, nutritional, and industrial potential, it is necessary to investigate this fruit with a view to its application in the bioeconomic context. Consequently, this study aimed to obtain umari pulp oil using compressed propane as a solvent, investigating the effects of temperature and flow rate on the extraction oil yields, in addition to compare to conventional extraction technique. Besides, this work aimed to analyze and characterize the oils obtained by compressed propane for the first time in the literature, providing important data for valuing the umari production chain.

MATERIALS AND METHODS

UMARI FRUIT SAMPLES

Sample preparation

The umari fruits (*Poraqueiba sericea* Tul.) used in this work were collected in the municipality of Cametá, state of Pará, Brazil (2° 15′ 15″ S, 49° 30′ 44″ W) (SisGen: AE72BF1). The fruits were sanitized using a sodium hypochlorite solution (200 ppm). Then, the pulp fraction was removed from the seeds and subjected to freeze-drying (Liotop L101, Liobras, Brasil). The freeze-dried umari pulp was ground in a domestic food processor and then classified using different Tyler series sieves (8, 12, 20, 24, 32 meshes) coupled to a mechanical shaker (Produtest, São Paulo, Brazil). The average particle diameter was obtained by the method described by Souza et al. [15]. Finally, all fractions were blended and packed in low-density polyethylene bags, sealed under vacuum, and stored in a freezer at -5 °C until the analysis.

Proximate composition

The proximate composition of the freeze-dried umari pulp was determined by analyzing the moisture, lipid, protein, ash, and carbohydrate contents, according to the AOAC [16] methodologies. The moisture content of the fresh pulp was obtained by drying the sample in an oven at 105 °C, until constant weight. The ash content was determined by incineration of the samples in a muffle furnace heated to 600 °C. The lipid content was obtained by the Soxhlet method, using petroleum ether as a solvent. The protein content was quantified by the Kjeldhal method, using 6.25 as the conversion factor for nitrogen. The carbohydrate content was obtained from the difference between 100 and the sum of the moisture, ash, lipid, and protein contents.

UMARI PULP OIL EXTRACTION

Soxhlet extraction

Soxhlet extraction yields were determined in triplicate, using *n*-hexane (Neon, 99.95% purity) as solvent. Approximately 5 g of comminuted umari sample and 150 mL of solvent were used, being refluxed to its boiling point in a Soxhlet extractor apparatus (UNIGLAS, Campinas, São Paulo, Brazil) for 6 h. After extraction, the solvent was removed from the oil using a rotary vacuum evaporator (Model RV 10 digital, made by IKA) and then taken to the oven at 60 °C to remove remaining solvent residues. The oil was kept in an amber glass vessel and stored at -5 °C until further analysis. The percentage extraction yield was calculated in dry basis, according to Equation (1).

$$Y_{(\%db)} = \left(\frac{m_o}{m_s\left(1-\frac{U_s}{100}\right)} \right) 100 \quad (1)$$

Where: $Y_{(\%db)}$ is the yield in dry basis, m_o is the obtained extract mass, m_s is the sample mass used, and U_s is the percentage moisture of the freeze-dried umari pulp.

Compressed propane extraction (CPE)

Extractions were carried out using a laboratory-scale supercritical extraction apparatus provided with an extraction vessel of length (L) = 22 cm, diameter (ϕ) = 1.9 cm and internal volume (V) = 62.4 mL, described in detail in previous studies by our research group [17,18]. Briefly, the extraction system is composed of an extractor vessel connected to an ultra-thermostatic bath for temperature control, a syringe pump (ISCO, model 500D, Lincoln, NE, USA) to pressurize the propane, a needle valve to control the solvent flow rate inside the extractor, in addition to temperature and pressure sensors to control the process. The syringe pump temperature was set at 10 °C for all extractions using an ultra-thermostatic bath. The experiment temperature was maintained and controlled by another bath connected to the extractor jacket. The oils were collected at atmospheric pressure and room temperature. The propane with 99.5% purity (White Martins S.A., Araucária, PR, Brazil) was used in the experiments.

Approximately 13.2 g of raw material were used for each extraction. The static period (confinement) was set at 30 min, based on previous studies by our research group. The dynamic period was established at 70 min, and the extract mass was measured every 5 min. In this study, the pressure was set at 10 MPa, since the literature as well as the studies from our research group indicate that this variable has a minimal effect on the extraction yield of oil crops using compressed propane [8,10]. Therefore, the solvent flow and temperature parameters were chosen to be varied. Although less explored in the literature, the flow rate can provide relevant information in terms of engineering and process cost, in addition to being less explored when compared to other variables such as temperature, pressure and particle size. Therefore, extractions were carried out employing a two-level, two-factor experimental design to explore the best combination of two independent factors that affect the extraction process. The experimental design for compressed propane consisted of temperatures (40 - 80 °C) and solvent flow rates (1.0 - 3.0 mL min^{-1}), including triplicates at the central point. The extraction temperatures were measured from the extractor vessel connected to an ultra-thermostatic bath; and the solvent flow rates were measured through a needle valve using the operating conditions of the syringe pump set at 10 °C and a pressure of 10 MPa for all extractions. The overall extraction yield of crude oil was calculated using Equation (1), as described in Section 2.2.1.

OIL CHARACTERIZATION

Fatty acid composition

The fatty acid composition of umari extracts was determined by Gas chromatography with flame ionization detector (GC-FID). Samples were prepared according to the AOCS Official Method Ce 2–66 for converting oils into fatty acid methyl esters (FAMEs) [19]. FAMEs were analyzed using a Shimadzu chromatograph (GC 2010 Plus), a capillary column (SH-Rtx-Wax, Shimadzu, 30 m x 0.32 mm, 0.25 μm), flame ionization detector (FID) and split injection (1:10). The injector and detector temperatures were 240 °C and 250 °C, respectively. The oven temperature was set to start at 100 °C and remain at this temperature for 5 min, followed by an increase to 240 °C at a rate of 4 °C min^{-1} and maintained at this temperature for 5 min. The carrier gas was helium at 32.5 cm^3 min^{-1}. FAMEs were identified by comparison with the retention times of a standard mixture of FAMEs (Supelco, MIX FAME 37, St. Louis, MO 63103, USA). Quantification of fatty acids was performed using the area normalization procedure. The results were expressed as a percentage of each fatty acid present in the sample.

β-Carotene content

The analysis of the total β-carotene content was performed using the method proposed by Young & Britton [20], adapted by Cuco et al. [21]. Briefly, a 10 mg aliquot of each oil was diluted in 10 mL of *n*-hexane. Then, the solution was analyzed under absorbance of 450 nm using UV-vis spectrophotometer (Spectro 3000W, Marte Científica, Brazil). The total β-Carotene content was calculated using Equation 2. The results were obtained in triplicate and expressed as total mg of β-carotene per 100 g of oil.

$$Total\ \beta-carotene = \frac{(Abs\ Vol\ 10^3)}{E_{1cm}^{1\%}\ W} \quad (2)$$

Where *Abs* is absorbance at 450 nm; *Vol* is the dilution volume (mL) and $E_{1cm}^{1\%}$ is the extinction coefficient (2592) of β-carotene in hexane proposed by Ogawa et al. [22], and *W* is the mass of the sample (g).

Total phenolic content (TPC)

The total phenolic content (TPC) of the umari oil extracted with different methods were determined according to the Folin–Ciocalteu method [23]. Firstly, the samples were prepared by mixing 100 mg of oil with 1 mL of methanol:water (80:20, v/v), shaken and centrifuged for 10 min at 3000 rpm. To determine the TPC, 0.4 mL of the supernatant and 0.1 mL of methanol were mixed with 2.5 mL of Folin-Ciocalteu reagent (diluted 1:10 in distilled water). The mixture was kept in the darkness for 3 min. Afterward, 2 mL of 7.5 % (w/v) sodium carbonate aqueous solution was added to the mixture and incubated in the dark for 2 h. Then, the absorbance was measured at 760 nm in an UV/Vis Spectrophotometer (UV-1800 Shimadzu). All assays were performed in triplicates. The quantitative results were calculated using a gallic acid calibration curve and were expressed as mg of gallic acid equivalent (GAE) per 100 g of sample (mg GAE 100 g^{-1}).

Total flavonoids (TF)

Total flavonoids content of samples was determined based in the method proposed by Zhishen et al. [24], with some modifications. Aliquots (0.2 mL) of samples, prepared as previously described, and 1.8 mL of distilled water, and 0.12 mL of $NaNO_2$ (5%, w/v) were added to amber bottles and mixed. After 5 min, 0.12 mL of $AlCl_3$ (10%, w/v) was added; and after 6 min, 0.8 mL of NaOH (1 mol L^{-1}) and 0.96 mL of distilled water were added. Absorbance was measured at 510 nm in UV-vis spectrophotometer (UV-1800 Shimadzu). The catechin was used as the standard for a calibration curve and the results were expressed as mg of catechin equivalent (CE) per 100 g of sample.

Antioxidant capacity

The $DPPH^{\bullet}$ assay was performed based at method described by Brand-Williams et al. [25]. A 3.9 mL aliquot of a 6×10^{-5} mol L^{-1} $DPPH^{\bullet}$ methanolic solution was mixed with 100 μL of samples methanol:H_2O solutions, previously described. The $DPPH^{\bullet}$ absorbance was monitored at 515 nm after 1 h. The quantification was performed using

a Trolox analytical curve, and the results were expressed as μmol of Trolox equivalent antioxidant capacity (TEAC) per 100 g of sample (μmol TEAC.100 g^{-1}).

The ABTS method was performed based on a procedure described by Re et al. [26]. ABTS was dissolved in water to a 7 mmol L^{-1} final concentration. This solution (5 mL) was mixed with 88 μL potassium persulfate solution (140 mmol L^{-1}) and then incubated in the dark for 16 h at room temperature to produce a stock solution of the radical cation ($ABTS^{\bullet+}$). The $ABTS^{\bullet+}$ working solution was prepared by diluting the stock solution with absolute ethanol until reaching an absorbance of 0.700 ± 0.020 at 734 nm. For the samples analyses, aliquots of methanol:H_2O solutions prepared as previously described, and methanol up to 30 μL was added to amber bottles and mixed with 3 mL of the $ABTS^{\bullet+}$ radical cation working solution ($A_{734\ nm}$ = 0.700 ± 0.020). Absorbance readings were taken at 734 nm after 6 min. Quantification was conducted using a Trolox analytical curve, and the results were expressed as μmol of Trolox equivalent antioxidant capacity (TEAC) per 100 g of sample (μmol TEAC.100 g^{-1}).

Thermal behavior of umari oil

Thermal analyzes of umari oils obtained by the compressed propane and Soxhlet extraction methods were carried out to determine the impact of different extraction techniques on the oil's thermal behavior. For the thermogravimetric analysis (TGA) and differential scanning calorimeter (DSC) it was selected the operational condition with the highest yield to represent the extraction with compressed propane due to the trend of industrial interest in the higher yield process.

Thermogravimetric analysis (TGA)

The thermal stability of umari pulp oils obtained by different extraction methods was determined by thermogravimetric curves obtained with the TGA 4000 equipment (Perkin Elmer Inc. Waltham, MA, USA). For CPE, the condition with the highest oil yield was chosen. Approximately 10 mg samples of umari oil were placed in a platinum pan and then in an oven, where they were heated from 30 °C to 750 °C (10 °C min^{-1}) in a synthetic air atmosphere with a flow of 50 mL min^{-1}. The results obtained were processed

using Pyris^{TM}. Thermogravimetric (TG) and derivative thermogravimetric (DTG) curves were evaluated using Origin 8.6 software (OriginLab, Massachusetts, USA).

Differential scanning calorimeter (DSC)

The differential scanning calorimeter analyses were performed using a DSC (850 Perkin Elmer Inc. Waltham, USA) equipped with nitrogen (N_2, 99.999% purity) as purge gas. Umari oil samples obtained from different extraction methods were analyzed. For CPE, the condition with the highest oil yield was chosen. Approximately 4 mg of oil were weighed and sealed in an airtight aluminum pan, then the cooling and heating tests were carried out. The following time-temperature setting was used: equilibration at 20 °C for 5 min to ensure complete sample temperature homogenization; heating to 50 °C at 5 °C min^{-1} and holding for 5 min, followed by cooling to -80 °C at -5 °C min^{-1} to induce crystallization, keeping this temperature for 5 min. Finally, heating was carried out from -80 to 50 °C at 5°C min^{-1} to obtain the melting profile [27].

STATISTICAL DATA ANALYSIS

The experimental design was performed using Statistica 7.0 software (Statsoft Inc., Tulsa, OK, USA), where data were analyzed for variance (ANOVA), and Tukey's test analyzed significant differences at the 5% significance level. The results obtained from the chemical characterization of the extracts were expressed as mean ± standard deviation (SD). The graphs were created using Origin 8.6 (OriginLab, Northampton, MA, USA) software.

RESULTS AND DISCUSSION

RAW MATERIAL CHARACTERISTICS

The average particle diameter was 1.61 mm. The proximate composition of the freeze-dried umari pulp showed the following results: 7.81 ± 0.30 wt% moisture; 1.74 ± 0.09 wt% ash; 7.98 ± 0.08 wt% proteins; 25.25 ± 0.28 wt% lipids and 57.22 wt% total

carbohydrates. The results showed that umari pulp has a high lipid content, with similar contents to those found in peach palm (18.73 wt%) [28] and uxi (25.61 wt%) [13], placing this species among the richest Amazonian sources of this macronutrient.

YIELDS AND EXTRACTION KINETICS

The yields obtained from compressed propane extraction (CPE) are expressed in **Table 1**. The yield for Soxhlet extraction using hexane as solvent was 27.8 ± 0.3 wt% (dry basis) in a total extraction time of 360 min and 12 solvent cycles. For CPE, the highest extraction yield (29.2 wt%) was found under conditions of 80 ºC and a flow rate of 3 mL min^{-1}, while the lowest yield (25.0 wt%) was obtained at 80 °C and 1 mL min^{-1}. For comparison purposes, the yield obtained by Soxhlet using hexane as solvent was adopted as a reference to calculate the efficiency of extractions with compressed propane. The highest yield obtained with CPE was numerically higher than that obtained in conventional extraction (Soxhlet) using hexane as solvent. Furthermore, the extraction time with propane was 70 min, while in the Soxhlet extraction it was 360 min, indicating that similar yields can be obtained in a much shorter time using CPE, in addition to not requiring a second stage of separation.

Table 1 - Experimental conditions and results of umari pulp extracts obtained with compressed propane.[@]

Run	Pressure (MPa)	T (°C)	Flow rate (mL min^{-1})	Density[#] (kg/m^3)	Extraction yield**(wt%)	Extraction efficiency[##] (%)
1	10	40	1	495.50	26.6	95.7
2	10	80	1	435.25	25.0	90.0
3*	10	60	2	467.20	27.3 ± 0.5	98.2
4	10	40	3	495.50	28.9	104.1
5	10	80	3	435.25	29.2	104.9

[@]Experimental design (2^2).

*Center points with mean and standard deviation values from three extractions.

[#]propane density in the extraction condition, obtained from the NIST database.

**Yields calculated in dry basis.

[##] Mass of extract obtained with propane by mass of extract obtained with hexane in Soxhlet.

In the evaluated working range, the flow rate variation had a significant effect on the yield of umari pulp extraction, as shown in the Pareto chart (**Figure 1**), which shows the influence of the independent variables (flow rate and temperature) on the extraction's overall yield. The temperature had a negative effect on the overall yield of the extraction carried out with compressed propane.

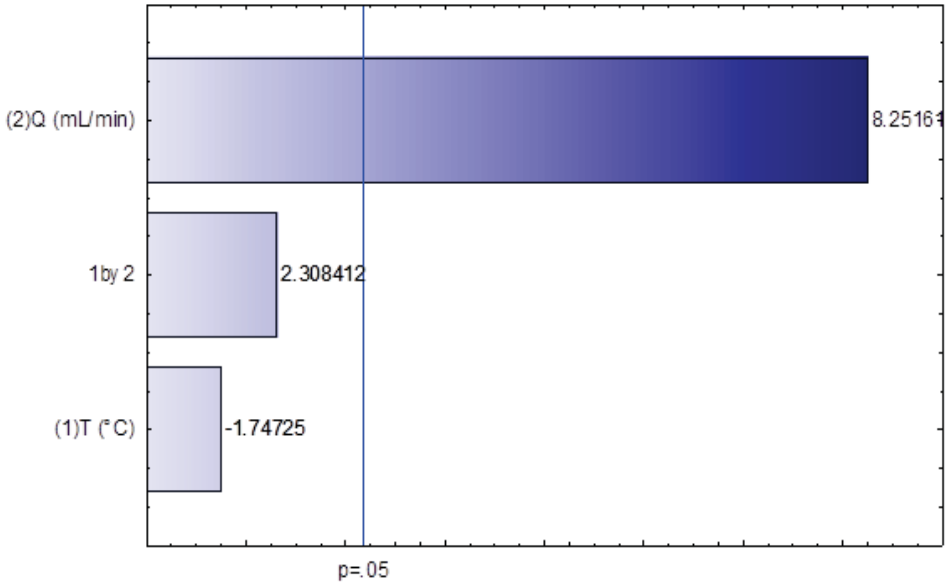

Figure 1 - Pareto chart showing the effects of process parameters on the extraction yield of umari pulp oil.

The overall extraction curves using compressed propane as solvent are shown in **Figure 2.** The conditions of 80 °C and 3 mL/min; 60 °C and 2 mL/min; and 40 °C and 3 mL/min showed overall extraction curves with similar profiles. In the first stage, from the beginning of dynamic extraction until approximately 15 min, constant extraction rate (CER) occurred due to the high availability of oil on the surface of the particles, where most of this easily accessible solute was extracted. At this stage, mass transfer occurs predominantly by convection [29]. The falling extraction rate (FER) was observed within the intermediate period of 20–35 min. At this stage, the solute layer surrounding the cell begins to acquire flaws, starting the diffusion of the solute inside the particle. Mass transfer by convection and diffusion occurs together at this stage. Thereafter, the extraction rate is reduced due to the depletion of readily available oil until the third stage, called diffusional constant rate (DCR), is observed. In this last stage, solubility is limited by the internal diffusion mechanism, where extraction occurs predominantly by the

diffusion of the solvent into the particle and by the diffusion of the solvent extract to the surface of the particle. This behavior was also observed in other studies [9,10,30,31].

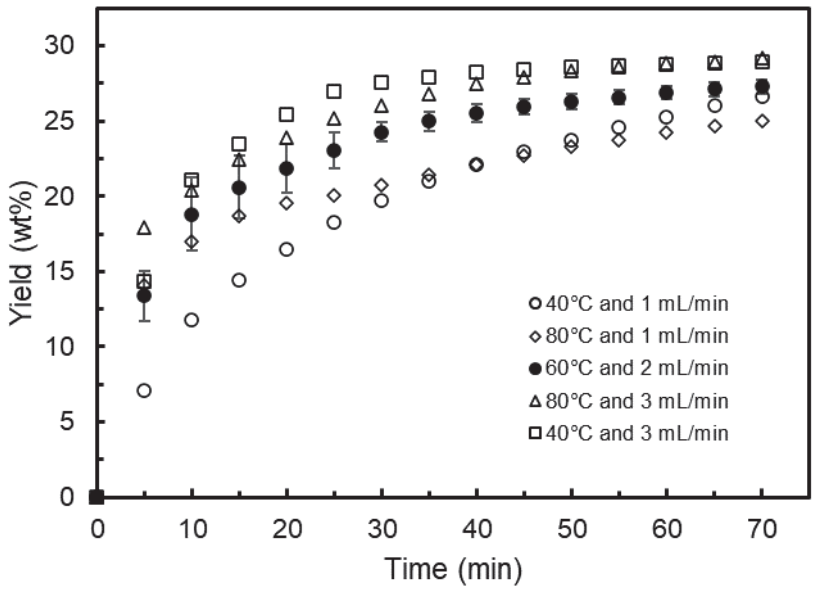

Figure 2 - Experimental overall extraction curves for umari pulp extraction using compressed propane as solvent.

The overall extraction curves of the two conditions in which the lowest solvent flow rate was used (40 °C and 1 mL/min; 80 °C and 1 mL/min) showed slightly different behavior from the others, which may be attributed to the lower solvent flow rate in the extraction bed. In the condition of 80 °C and 1 mL/min there was a high extraction rate in the first 15 min, reaching approximately 75 wt% of the final extraction yield. However, after this period the curve showed an almost linear behavior, indicating that under this condition it does not reach a plateau of stability, thus requiring more time and, consequently, more solvent consumption to achieve higher yields. This may have occurred due to the higher extraction temperature which favored the extraction of easily accessible oil available in the particles during the static extraction period, where the sample was in contact with propane for 30 min at 80 °C, resulting in a high oil yield in the initial minutes of dynamic extraction. After that, the extraction was controlled predominantly by diffusion, showing a linear trend. The same behavior was observed in the curve for the 40 °C and 1 mL/min condition, with the difference that it showed a slower extraction rate in the first 15 min, which may be attributed to the use of a milder temperature. After that, the curve exhibited an almost linear behavior until the end of

extraction, without reaching the stability plateau. It is worth noting that the use of higher temperatures improves extraction efficiency, as it helps to break analyte-sample interactions, decreasing the surface tension of the solvent, increasing wettability, and allowing the analytes to dissolve more quickly in the solvent. Furthermore, an increased temperature decreases the viscosity of a liquid solvent, thereby increasing its penetration into the matrix [32–34].

Considering only the extraction yield as a parameter of interest, the condition of 80 °C and 3 mL/min can be considered the best option, as it presented the highest extraction yield. However, the condition of 40 °C and 3 mL/min seems to be the most attractive, as it provides similar yields and works at a milder temperature (40 °C), which may favor the preservation of thermolabile compounds present in the extract and provide reduced operating costs. Furthermore, in this condition, it is possible to recover 95% of the total oil in the first 30 min of extraction.

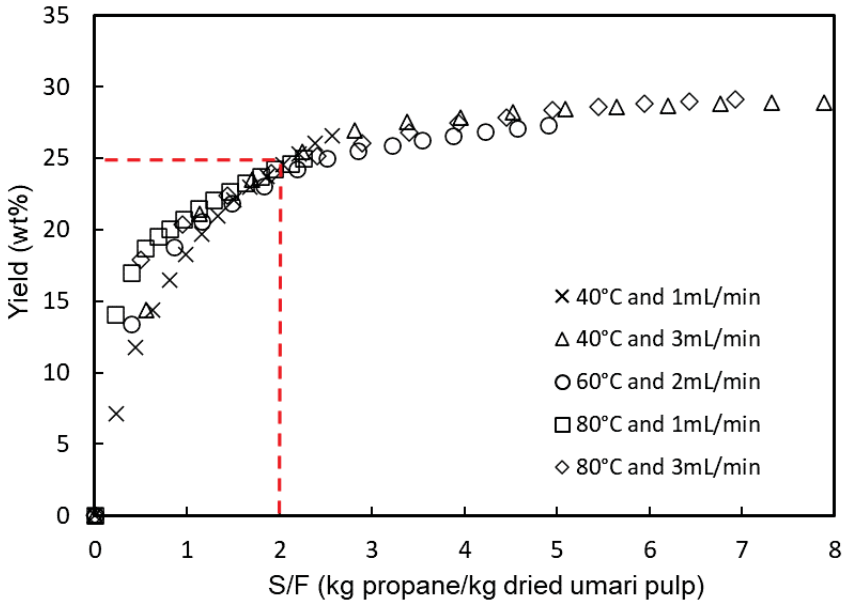

Figure 3 - Extraction curves with compressed propane at different temperatures and flow rates.

Figure 3 depicts the extraction yields to the mass ratio between solvent and feed S/F. The results indicate that in all process conditions analyzed, the S/F of 2 kg of propane/kg of dried umari pulp was able to provide a yield of 25.0 wt% oil, which is equivalent to almost 90% of the total umari oil, taking the Soxhlet extraction yield (27.8 wt%) as a reference and assuming that it was able to recover all the lipid fraction from

the umari pulp. In extractions carried out with the highest flow rates (3 mL/min) it is possible to observe that there is no longer meaningful increase in the extraction yield after S/F equal to 4. From that point on, the extraction yield increased slowly, as the extraction was controlled mostly by diffusional constant rate (DCR) until exhausting the available oil content in the sample. In this situation, it is suggested that the extraction be stopped at this point, thus avoiding unnecessary solvent and energy consumption. According to Goto et al. [35], when the curves follow a single trend line, it indicates that the extraction is predominantly controlled by the solubility of the easily accessible solute, with little influence from the internal diffusion mechanism. Han et al. [36] and Jesus et al. [37] found graphic profiles similar to those found in the present work.

OIL CHARACTERIZATION

Fatty acid composition of umari pulp oil

The fatty acid composition of umari pulp oil obtained by different methods is expressed in **Table 2**. The results show that the fatty acid profiles obtained are quite similar, regardless of the extraction technique used to obtain the oil. Five fatty acids were identified in umari pulp oils, of which oleic acids (65.09–66.09%) and palmitic acids (27.93–28.50%) were the majority. Berto et al. [2] found similar results for umari oil, with oleic acid predominating (~68%), followed by palmitic acid (~20%). Silva [3] had also previously revealed the predominance of oleic acid (~65%) and palmitic acid (~24%) in umari pulp oil obtained by conventional extraction methods. The umari pulp oil presented a profile with a predominance of unsaturated fatty acids (67.29–68.35%), whose concentration was much higher than the saturated fraction (31.66–32.71%). The presence of monounsaturated fatty acids (65.09–66.09%) was higher when compared to polyunsaturated fatty acids (1.83–2.65%) in all extractions performed.

Table 2 - Fatty acid composition (wt%) of umari pulp oil extracted by compressed propane and Soxhlet.

Fatty acids	**propane (40 °C/10 MPa/ 1 mL min^{-1})**	**propane (40 °C/10 MPa/ 3 mL min^{-1})**	**propane[a] (60 °C/10 MPa/ 2 mL min^{-1})**	**propane (80 °C/10 MPa/ 1 mL min^{-1})**	**propane (80 °C/10 MPa/ 3 mL min^{-1})**	**Soxhlet (Hexane)**
Palmitic (C16:0)	28.06	28.39	28.19	28.15	27.93	28.50
Stearic (C18:0)	3.80	3.74	3.72	3.75	3.73	4.21
Oleic (C18:1)	66.08	66.04	65.93	66.09	65.70	65.09
Linoleic (C18:2)	0.80	0.64	0.81	0.79	1.16	0.84
Linolenic (C18:3)	1.26	1.19	1.34	1.22	1.49	1.36
∑ SFA[1]	31.86	32.13	31.91	31.90	31.66	32.71
∑ MUFA[2]	66.08	66.04	65.93	66.09	65.70	65.09
∑ PUFA[3]	2.06	1.83	2.15	2.01	2.65	2.20
∑ PUFA + MUFA	68.14	67.87	68.08	68.10	68.35	67.29

The standard deviation for all fatty acids was lower than 0.8%.

[a] Center point for compressed propane extraction.

[1] Saturated fatty acids.

[2] Monounsaturated fatty acids.

[3] Polyunsaturated fatty acids.

The results showed that umari pulp oil is rich in oleic acid, which is a monounsaturated fatty acid known as omega-9 that participates in our metabolism, playing a fundamental role in the synthesis of hormones. Omega-9 can also help the body absorb vitamins more efficiently, in addition to being related to healthier triglyceride levels, helping to reduce levels of total blood cholesterol, LDL (bad cholesterol) and increasing HDL (good cholesterol), playing an important role in the prevention of cardiovascular diseases [38,39]. The high concentration of MUFAs present in umari oil also draws attention in the industrial context, as they have beneficial effects on controlling serum cholesterol, and the consumption of foods rich in MUFAs is recommended to replace foods rich in SFAs [40]. Therefore, due to the high concentration of unsaturated fatty acids (PUFA+MUFA), umari pulp oil extracted with compressed propane stands out as a food with high nutritional value with the potential to be used in various industrial segments, being an important raw material option for insertion into the Amazon bioeconomy scenario.

β-Carotene content

The umari oil obtained with compressed propane showed high levels of β-carotene (BC) in all conditions investigated (**Table 3**), varying from 129.79 ± 2.33 to 149.36 ± 4.43 mg/100 g oil. The highest BC content was found in the lowest temperature and lowest flow rate condition (40°C; 1 mL min^{-1}), while the lowest BC value was found in the highest temperature and highest flow rate condition (80°C; 3 mL min^{-1}). The fact that BC is a compound that degrades at high temperatures may justify the higher BC values at lower temperature conditions. Furthermore, the oils obtained with compressed propane had higher BC contents when compared to the oil obtained by Soxhlet extraction, demonstrating that compressed propane is capable of extracting oils with more BC in less time, in addition to using a green technology with no need for subsequent organic solvent separation steps. This result can be justified due to the high penetrating ability of the pressurized fluid, solubilizing carotenoids by convective and diffusive mechanisms. Barbi et al. [9] and Zanqui et al. [41] found lower BC contents (89.55 mg/100 g and 70.95 mg/100 g oil) for inajá pulp oil and blends of edible oil with carrots using compressed propane, respectively. On the other hand, Trentini et al. [42] found a higher value of BC (356.05 mg/100 g oil) in macauba pulp oil obtained with compressed

propane. Therefore, as carotenoids are lipophilic compounds of low polarity, propane ends up being a great solvent for obtaining this important compound from the umari fruit pulp, showing potential for use in various industrial segments, such as the pharmaceutical industry.

In this context, pre-clinical and clinical studies have demonstrated that among carotenoids, β-carotene (BC) has the potential to modulate the genesis, progression and worsening of chronic diseases, such as cardiovascular, metabolic, neurodegenerative diseases and cancer. This occurs because carotenoids are phytochemical compounds with intense biological antioxidant, anti-inflammatory and immunomodulatory activity [43–45]. In fact, it has already been demonstrated that in experimental models, dietary BC supplementation slows the progression of atherosclerosis, reducing vascular inflammation and hepatic cholesterol secretion [46–48]. Higher BC levels have also been associated with lower risk of cardiovascular disease (CD), where individuals with higher baseline serum BC had reduced risk of mortality from CD, heart disease, stroke, and diabetes compared to individuals with lower concentrations [49,50]. Additionally, BC exerts protective effects on atrophy and muscle loss in cancer [51]. BC pretreatment inhibits neuroblastoma tumorigenesis by regulating cell differentiation and cancer stem cell markers *in vitro* and *in vivo* [52–54].

Beta-carotene treatment also attenuated the nephrotoxic effects of bromobenzene by reducing oxidative stress and lipid peroxidation, improving antioxidant status in an environmental poisoning model [55]. Furthermore, BC reduced oxidative stress and pro-inflammatory cytokines in mononuclear cells from Alzheimer's patients, reducing the production of ROS, improving cellular antioxidant capabilities and modifying inflammation induced by cytokines [56]. Interestingly, BC treatment led to neuroprotection by inhibiting microglial activation, excitotoxic pathway, modulation of autophagy, attenuation of oxidative damage and activation of defensive antioxidant enzymes in animal models [57]. The pharmacological potential of BC in the treatment of diseases that have a high prevalence and high social impact on the quality of life of thousands of individuals around the world is undeniable, but there is still much debate about important therapeutic factors regarding this application. Many of them still need elucidation and more comprehensive studies such as safe dosage, forms of administration (food, matrix or supplement), duration of treatment, combination with other nutrients and individual factors that are important therapeutic modifiers [58–60]. Thus, umari oil

obtained with compressed propane is rich in BC, with great potential to be inserted into the bioeconomy scenario, generating income and development for the Amazon region.

Table 3 - β-Carotene, total phenolic contents, total flavonoids content and antioxidant capacity for umari pulp oil obtained from compressed propane and Soxhlet extraction.

Run	Extraction conditions	Solvent	β-Carotene (mg 100 g^{-1})	TPC (mg GAE 100g^{-1})	TF (mg CE 100 g^{-1})	DPPH (µmol TEAC 100 g^{-1})	ABTS (µmol TEAC 100 g^{-1})
1	40 °C; 1 mL min^{-1}	Propane	149.36 ± 4.43	3.42±0.38	670.08±12.19	21.75±0.96	293.97±1.37
2	40 °C; 3 mL min^{-1}	Propane	146.08 ± 7.87	3.66±0.40	630.47±8.04	20.30±1.61	251.12±8.16
3*	60 °C; 2 mL min^{-1}	Propane	133.43 ± 2.81	3.36±0.34	661.35±11.32	18.97±1.08	266.18±3.48
4	80 °C; 1 mL min^{-1}	Propane	145.82 ± 1.66	3.09±0.20	723.32±4.28	17.79±1.56	279.13±10.14
5	80 °C; 3 mL min^{-1}	Propane	129.79 ± 22.33	2.62±0.24	591.60±13.87	16.26±1.03	262.32±5.86
6	Soxhlet; 68 °C	Hexane	132.12 ± 8.60	5.18±0.40	1010.29±33.29	22.91±1.25	247.02±3.81

* Triplicate at central point for compressed propane extraction.

Total phenolic content (TPC) and total flavonoids (TF)

The oils extracted with compressed propane exhibited TPC values ranging from 2.62 ± 0.24 to 3.66 ± 0.40 mg GAE/100 g oil. The highest values were observed for Run 1 (3.42 ± 0.38 mg GAE/100 g oil) and Run 2 (3.66 ± 0.40 mg GAE/100 g oil) at lower extraction temperatures (40 °C) (**Table 3**). In contrast, these values were lower than those obtained using Soxhlet extraction with hexane, which yielded 5.18 ± 0.40 mg GAE/100 g oil. This trend is consistent with findings reported in the literature. Fetzer et al. [10] observed that Soxhlet extraction resulted in higher TPC values (22 ± 3 mg GAE/100 g oil) in cumaru oils compared to extractions using pressurized propane under various conditions (4.2 ± 0.3 to 10.5 ± 0.5 mg GAE/100 g oil). Similarly, Azevedo et al. [61] found that Soxhlet extraction produced higher TPC values (26.64 mg GAE/100 g oil) in corn germ oil, whereas extractions using pressurized propane yielded lower values (21.11 to 23.07 ± 0.03 mg GAE/100 g oil).

Total flavonoids (TF) are also presented in **Table 3**. It was observed that TF values ranged from 591.60 ± 13.87 to 723.32±4.28 mg CE 100 g^{-1}. Among these results, it can be noted that the highest temperature with the lowest flow rate used (propane at 80° C and 1 mL min^{-1}) resulted in higher TF values. Furthermore, like the TPC behavior, the TF results for Soxhlet extraction (1010.29 ± 33.29 mg CE 100 g^{-1}) were higher than those for propane extraction. Comparing with data obtained from the extraction of macaúba pulp oil using compressed propane by Trentini et al. [42], the results presented in **Table 3** were higher than the reported range (11.03 ± 0.07 to 13.12 ± 0.10 mg CE 100 g^{-1}). The different TPC and TF results between the compressed propane and Soxhlet methods can be explained by the high temperatures used in CPE (80 °C; 3 mL min^{-1}) and, primarily, by the greater solubility (higher affinity) of certain bioactive compounds, such as flavonoids, in nonpolar solvents such as hexane [31,42].

Antioxidant capacity

Table 3 shows the results obtained for two antioxidant assays (DPPH and ABTS) for oils extracted by different methods. Both antioxidant assays are extensively employed for assessing the antioxidant capacity of various compounds, including plant extracts, foods, and vegetable oils [31]. The DPPH radical scavenging capacity of the samples

extracted using compressed propane exhibited a range from 16.26 ± 1.03 to 21.75 ± 0.96 μmol TEAC 100 g^{-1}, with the highest value (21.75 ± 0.96 μmol TEAC 100 g^{-1}) in the conditions of 40 °C and 1 $mL.min^{-1}$, which was a little lower than the result obtained by the Soxhlet method (22.91 ± 1.25 μmol TEAC 100 g^{-1}). Regarding the results obtained for ABTS, there was a variation from 251.12 ± 8.16 to 293.97±1.37 μmol TEAC 100 g^{-1}, with the highest value observed in the condition of 40 °C and 1 mL min^{-1}. For ABTS essay, run 1 (40 °C; 1 mL min^{-1}) presented the highest values of antioxidant capacity, even higher than those obtained by Soxhlet (247.02 ± 3.81 μmol TEAC 100 g^{-1}), suggesting that the use of lower temperatures may have contributed to the maintenance of some thermosensitive bioactive compounds. This corroborates the higher data obtained for β-carotene (149.36 ± 4.43 mg 100 g^{-1}) in the conditions with lower temperature, which is frequently reported in the literature as being responsible for considerable antioxidant activity against the DPPH radical [21].

Thermal behavior of umari oil

Thermogravimetric analysis (TGA)

The mass loss (TGA) and mass loss rate (DTG) curves of umari pulp oil extracted by different methods (compressed propane and Soxhlet) were obtained by thermogravimetric analysis (**Figures 4A and 4B**, respectively). Due to the very similar fatty acid composition, both solvents exhibited a thermal behavior that was also analogous, with the phases of mass degradation detailed in **Table 4**.

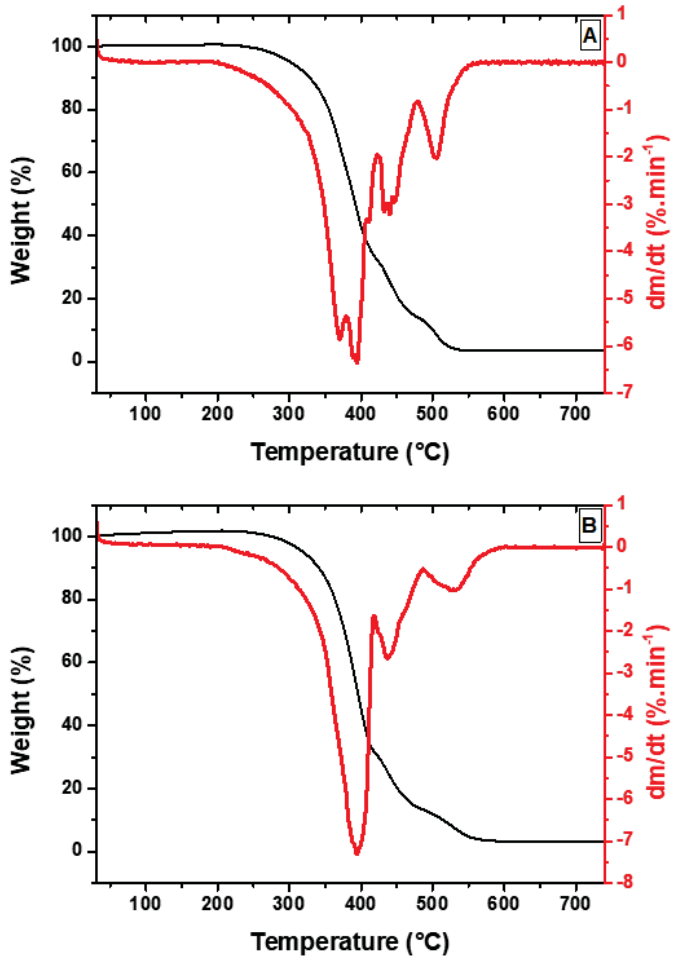

Figure 4 - TGA and DTG curves represented by the red and black lines, respectively, for umari pulp oil extracted using (A) hexane (Soxhlet) and (B) compressed propane (80 °C/10 MPa/3 mL min^{-1}).

As shown in **Figures 4A** and **4B**, mass degradation occurs in three main stages (**Table 4**), evidenced by three peaks. The first stage (I) could be associated with thermal decomposition and the onset of polyunsaturated fatty acid triglycerides degradation. The following steps (II and III) correspond to the decomposition of monounsaturated and saturated fatty acids, in addition to peroxides and hydroperoxides formed by the polymerization of fatty acids degradation products from the initial phase [9,62,63].

Table 4 - TGA and DTG curve data for the extraction of umari pulp oil using hexane and compressed propane.

Solvent	**Phase of thermal degradation**	**T_i (°C)**	**T_m (°C)**	**T_f (°C)**	**Δm (%)**

Hexane	Stability	30.91	-	276.61	-
	I	276.61	394.53	423.10	74.09
	II	423.10	440.38	476.99	12.26
	III	476.99	505.75	553.06	11.75
Compressed propane	Stability	31.10	-	237.17	-
	I	237.17	394.26	418.27	77.69
	II	418.27	438.05	484.95	11.44
	III	484.95	531.91	589.48	9.09

T_i, initial temperature; T_m, maximum mass loss temperature; T_f, final temperature; Δm, mass loss.

In this study, extraction with hexane provided slightly superior thermal stability of the oil, as mass loss initiates at 276.61 °C, while oil extracted with compressed propane begins to degrade at 237.17 °C. Barbi et al. [9] obtained a similar thermal degradation behavior with inajá pulp oil extracted with Soxhlet (hexane and petroleum ether) and compressed propane under different conditions. In the mentioned study, curves related to conventional extractions showed two degradation peaks, while curves related to propane extractions showed two or three peaks, depending on the condition applied. Furthermore, in the extraction with compressed propane (at 10 MPa, 20 °C, and a flow rate of 2 mL min^{-1}). mass loss began at 269 °C, with three peaks (at 368 °C, 424 °C, and 562 °C), indicating a similarity in fatty acid composition between inajá and umari oils.

Differential scanning calorimeter (DSC)

Differential Scanning Calorimetry (DSC) was applied to obtain the cooling curves (**Figure 5A**) and heating curves (**Figure 5B**) of umari pulp oil extracted by different methods (compressed propane and Soxhlet). It is possible to observe very similar thermal events when different solvents are compared, both in terms of the temperatures at which the events occur and their intensity, which was expected as the oils have practically the same composition of fatty acids.

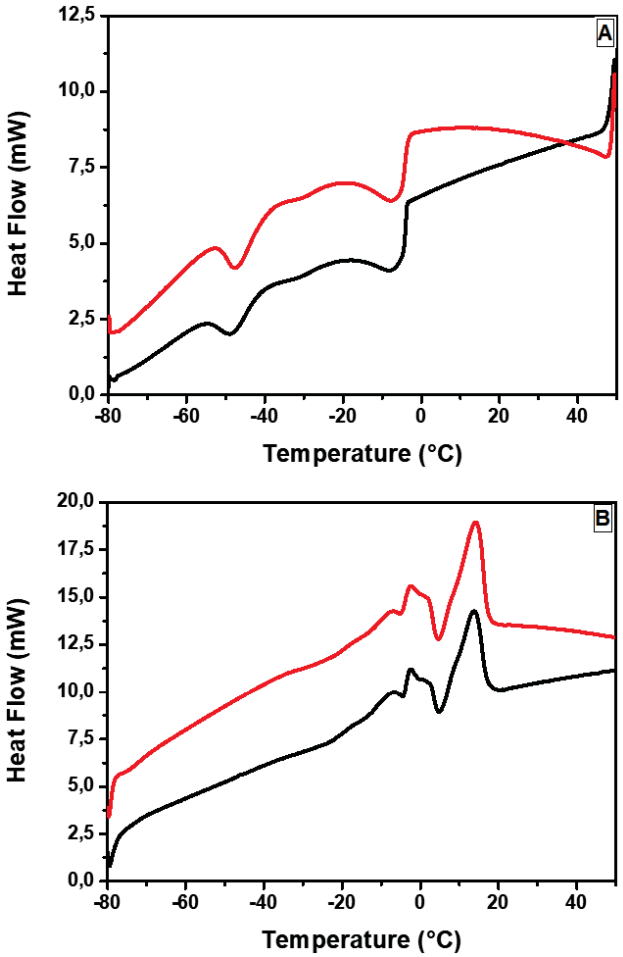

Figure 5 - DSC curves of umari pulp oil extracted using hexane (Soxhlet), represented by black lines, and compressed propane (80 °C/10 MPa/3 mL min^{-1}), represented by red lines. (A) crystallization (50 to -80 °C) and (B) melting (-80 to 50 °C) behavior.

Figure 5A indicates two main exothermic peaks. Nucleation begins at -3°C, with the first peak occurring at approximately -8 °C. Then, there is a second crystallization peak at -49°C. Total crystallization of the samples ends at approximately -55 °C. The different exothermic crystallization phases are likely due to the presence of different fractions of a specific type of triacylglycerol [64,65]. The initial crystal formation phase is related to molecule rearrangement due to the presence of highly saturated triacylglycerols, while the end of crystallization occurs as a consequence of molecule aggregation and compaction [66,67].

Figure 5B highlights two endothermic peaks related to sample melting. The first peak is not well delineated and can almost be interpreted as two peaks (at -8 °C and -3 °C), but due to their proximity they can be classified as a single thermal event, with maximum intensity at -3 °C. The second crystallization peak occurs at 14 °C. In the study

by Fetzer et al. [10], conducted with cumaru seed oil, a similar thermal behavior is observed, with the second peak indicating that the transition to the liquid state is completed at approximately 19 °C. In general, the higher the degree of unsaturation of fatty acids, the lower the melting point [68].

CONCLUSION

The extraction of umari oil using compressed propane showed promising results in terms of yield, even higher than that obtained with conventional extraction. Furthermore, the oil obtained by CPE proved to be rich in β-carotene (vitamin A precursor) and oleic acid (omega-9), presenting high values of these compounds, with contents higher than those found in many Amazonian species. Additionally, umari oil presented interesting values of antioxidant capacity and good thermal stability, making this product a great source of value-added compounds with potential for use in various industrial sectors, including the food, cosmetic and pharmaceutical industries. As umari is still a little explored and undervalued fruit, these discoveries can add value to this species, introducing it into the Amazonian bioeconomic scenario and promoting regional development.

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CHAPTER III

GREEN EXTRACTION OF β-CAROTENE-RICH OIL FROM TUCUMÃ (*Astrocaryum vulgare*) PULP USING COMPRESSED PROPANE

This chapter is part of the article entitled “Green extraction of β-carotene-rich oil from tucumã (*Astrocaryum vulgare*) pulp using compressed propane” **SUBMITTED** to *The Journal of Supercritical Fluids*.

GREEN EXTRACTION OF β-CAROTENE-RICH OIL FROM TUCUMÃ (*Astrocaryum vulgare*) PULP USING COMPRESSED PROPANE

ABSTRACT

Tucumã pulp oil was obtained using compressed propane extraction (CPE) and compared with the conventional Soxhlet extraction method. The oils were characterized for their fatty acid composition, β-carotene content, antioxidant capacity, total phenolics content (TPC), total flavonoids (TF), and thermal behavior. CPE extraction provided the highest extraction yield (33.9 wt%) with an efficiency of up to 91.4%. The extraction condition that resulted in the highest yield was 80 °C and 3 mL/min, with solvent flow rate showing a significant effect ($p < 0.05$) on the oil extraction yield. Tucumã pulp oil obtained via CPE proved to be an excellent source of β-carotene (21 times more than oils obtained by Soxhlet) and oleic acid, both of which are bioactive compounds of great interest in various industrial niches, with potential applications in cosmetics, food, and pharmaceuticals. Such applications enhance the production chain of this species, contributing to the Amazonian bioeconomy development.

Keywords: Bioeconomy, bioactive, Oil, Extraction, Amazon.

INTRODUCTION

The Amazon is globally recognized for its rich biodiversity, encompassing a wide variety of plant species with bioeconomic potential. Among these species, tucumã (*Astrocaryum vulgare* Mart.) stands out, a native palm whose fruit pulp is rich in valuable bioactive compounds, such as β-carotene and oleic acid. The oil extracted from tucumã pulp is highly valued for its antioxidant and nutritional properties, which can be sustainably exploited in the food, cosmetics, and pharmaceutical industries, promoting the development of the Amazon bioeconomy [1,2].

To align the economic exploitation of natural resources with environmental conservation, green extraction methods, such as compressed propane extraction (CPE), have gained prominence. This technique allows for the efficient extraction of high-quality oils with a lower environmental impact compared to conventional methods, such as organic solvent extraction [3]. The propane is highly suitable for extracting nonpolar compounds, such as lipids, and can be efficiently recovered and recycled during the extraction process, minimizing waste and reducing overall solvent consumption. Additionally, equipment for compressed propane extraction generally operates at lower pressures than supercritical CO_2, potentially reducing capital and operational costs. These combined benefits highlight why CPE is becoming increasingly attractive as a greener and more sustainable solution for obtaining high-quality oils and bioactive compounds [4,5].

The application of CPE for obtaining tucumã oil rich in β-carotene emerges as a promising alternative since it is a liposoluble compound. β-Carotene (BC) and oleic acid (OA) are bioactive compounds with numerous advantages and wide industrial applications. β-Carotene, a potent antioxidant and precursor of vitamin A, plays a crucial role in maintaining eye health, strengthening the immune system, and preventing chronic diseases such as cancer and cardiovascular diseases [6,7]. Its ability to neutralize free radicals makes it a valuable ingredient in cosmetic products, promoting skin health and delaying the signs of aging [8]. Oleic acid, a monounsaturated fatty acid, is known for its cardiovascular benefits, including reducing LDL (bad) cholesterol and increasing HDL (good) cholesterol. It also possesses anti-inflammatory properties and improves insulin sensitivity [9]. The incorporation of oleic acid into food products not only improves the nutritional profile but also contributes to the stability and shelf life of foods due to its resistance to oxidation. These properties highlight the importance of β-carotene and oleic

acid as key components in innovative and functional formulations to promote health and well-being [10].

In this context, since there are no published works applying CPE to obtain tucumã pulp oil, the aim of this study is to investigate the effectiveness of compressed propane extraction in obtaining oils rich in oleic acid and β-carotene from tucumã pulp. The research will compare this technique with traditional extraction methods, assessing its efficiency and the characteristics of the extracted oils. The study seeks to provide a deeper understanding of the advantages of green extraction and the potential applications of tucumã pulp oil in the Amazonian bioeconomic scenario in order to enhance the species' production chain.

MATERIALS AND METHODS

TUCUMÃ FRUIT SAMPLES

Sample preparation

The tucumã fruits (*Astrocaryum vulgare* Mart.) applied in this study were collected in Cametá, Pará, Brazil (2° 15′ 15″ S, 49° 30′ 44″ W) (SisGen: AE72BF1). The fruits were sanitized with a sodium hypochlorite solution (200 ppm). The pulp was then separated from the seeds and freeze-dried (Liotop L101, Liobras, Brazil). The freeze-dried pulp was ground using a domestic food processor and classified with different Tyler series sieves (8, 12, 20, 24, 32 meshes) attached to a mechanical shaker (Produtest, São Paulo, Brazil). The average particle diameter was determined by the method described by Souza et al. [11]. Finally, all fractions were mixed, vacuum-sealed in low-density polyethylene bags, and stored in a freezer at -5 °C until analysis.

Proximate composition

The proximate composition of the freeze-dried tucumã pulp was determined by analyzing the moisture, lipid, protein, ash, and carbohydrate contents following the AOAC [12] methodologies. The moisture content was measured by drying the sample in

an oven at 105 °C until it reached a constant weight. The ash content was determined by incinerating the samples in a muffle furnace at 600 °C. Lipid content was measured using the Soxhlet method with hexane as solvent. Protein content was quantified using the Kjeldahl method with a nitrogen conversion factor of 6.25. The carbohydrate content was calculated by subtracting the sum of the moisture, ash, lipid, and protein contents from 100.

UMARI PULP OIL EXTRACTION

Soxhlet extraction

Soxhlet extraction yields were determined in triplicate using *n*-hexane (Neon, 99.95% purity) as the solvent. Approximately 5 g of ground tucumã pulp sample and 150 mL of solvent were refluxed to its boiling point in a Soxhlet extractor apparatus (UNIGLAS, Campinas, São Paulo, Brazil) for 6 h. After extraction, the solvent was removed from the oil using a rotary vacuum evaporator (Model RV 10 digital, IKA) and then placed in an oven at 60 °C to evaporate any remaining solvent residues. The oil was stored in an amber glass vessel and kept at -5 °C until further analysis. The percentage extraction yield was calculated in dry basis, according to Equation (1).

$$Y_{(\%db)} = \left(\frac{m_o}{m_s\left(1-\frac{U_s}{100}\right)} \right) 100 \quad (1)$$

Where: $Y_{(\%db)}$ is the yield in dry basis, m_o is the obtained extract mass, m_s is the sample mass used, and U_s is the percentage moisture of the freeze-dried tucumã pulp.

Compressed propane extraction (CPE)

Extracts were obtained using a laboratory-scale supercritical extraction system equipped with an extraction vessel measuring 22 cm in length, 1.9 cm in diameter, and with an internal volume of 62.4 mL, as detailed in earlier research by our group [13,14]. The extraction setup includes an extractor vessel connected to an ultra-thermostatic bath for temperature regulation, a syringe pump (ISCO, model 500D, Lincoln, NE, USA) for pressurizing the propane, a needle valve to control the solvent flow rate inside the

extractor, along with temperature and pressure sensors to monitor the process. The syringe pump was set to 10 °C for all extractions using an ultra-thermostatic bath. The extraction temperature was maintained and regulated by a separate bath attached to the extractor jacket. The oils were collected at atmospheric pressure and room temperature, with propane of 99.5% purity (White Martins S.A., Araucária, PR, Brazil) used in the process.

Approximately 22 g of freeze-dried tucumã pulp were utilized for each extraction. The static phase (confinement) was set for 30 min, based on previous studies by our research group. The dynamic phase lasted 70 min, with the extract mass being measured at 2.5-min intervals during the first 10 min, and subsequently at 5-min intervals. In this study, the pressure was maintained at 10 MPa, since the literature as well as the studies from our research group indicate that this variable has a minimal effect on the extraction yield of oil crops using compressed propane [15,16]. Thus, the solvent flow and temperature parameters were selected for variation. Although less frequently investigated in the literature, the flow rate can offer valuable insights regarding engineering and process costs, and it is less studied compared to other factors such as temperature, pressure, and particle size. To investigate the optimal combination of two independent factors affecting the extraction process, extractions were conducted using a two-level, two-factor experimental design. This design for compressed propane involved varying temperatures (40 - 80 °C) and solvent flow rates (1.0 - 3.0 mL min^{-1}), with triplicate measurements at the central point. Extraction temperatures were monitored from the extractor vessel connected to an ultra-thermostatic bath, while solvent flow rates were controlled via a needle valve, with the syringe pump operating at 10 °C and a pressure of 10 MPa for all extractions. The overall extraction yield of crude oil was calculated in dry basis using **Equation 1**, as described in Section 2.2.1.

OIL CHARACTERIZATION

Fatty acid composition

The fatty acid composition of umari extracts was determined by Gas chromatography with flame ionization detector (GC-FID). Samples were prepared according to the AOCS Official Method Ce 2–66 for converting oils into fatty acid methyl

esters (FAMEs) [17]. FAMEs were analyzed using a Shimadzu chromatograph (GC 2010 Plus), a capillary column (SH-Rtx-Wax, Shimadzu, 30 m x 0.32 mm, 0.25 μm), flame ionization detector (FID) and split injection (1:10). The injector and detector temperatures were 240 °C and 250 °C, respectively. The oven temperature was set to start at 100 °C and remain at this temperature for 5 min, followed by an increase to 240 °C at a rate of 4 °C min^{-1} and maintained at this temperature for 5 min. The carrier gas was helium at 32.5 cm^3 min^{-1}. FAMEs were identified by comparison with the retention times of a standard mixture of FAMEs (Supelco, MIX FAME 37, St. Louis, MO 63103, USA). Quantification of fatty acids was performed using the area normalization procedure. The results were expressed as a percentage of each fatty acid present in the sample.

β-Carotene content

The total β-carotene content was analyzed using the method developed by Young & Britton [18] and adapted by Cuco et al. [19]. In summary, a 10 mg sample of each oil was dissolved in 10 mL of n-hexane. The solution was then measured for absorbance at 450 nm using a UV-vis spectrophotometer (Spectro 3000W, Marte Científica, Brazil). The total β-carotene content was calculated using **Equation 2**. Results were obtained in triplicate and reported as milligrams of β-carotene per 100 grams of oil.

$$Total\ \beta\text{-}carotene = \frac{(Abs\ Vol\ 10^3)}{E_{1cm}^{1\%}\ W} \quad (2)$$

Where *Abs* is absorbance at 450 nm; *Vol* is the dilution volume (mL) and $E_{1cm}^{1\%}$ is the extinction coefficient (2592) of β-carotene in hexane proposed Ogawa et al. [20], and *W* is the mass of the sample (g).

Total pheolic content (TPC)

The total phenolic content (TPC) of umari oil extracted by different methods was determined using the Folin–Ciocalteu method [21]. Initially, 100 mg of oil was mixed with 1 mL of methanol (80:20, v/v), shaken, and then centrifuged for 10 min at 3000 rpm. To measure the TPC, 0.4 mL of the supernatant and 0.1 mL of methanol were combined with 2.5 mL of Folin-Ciocalteu reagent (diluted 1:10 in distilled water). This mixture was kept in the dark for 3 min, then 2 mL of a 7.5% (w/v) sodium carbonate solution was

added, and the mixture was incubated in the dark for 2 hours. Absorbance was then measured at 760 nm using a UV/Vis Spectrophotometer (UV-1800 Shimadzu). All assays were conducted in triplicate. The results were quantified using a gallic acid calibration curve and expressed as milligrams of gallic acid equivalent (GAE) per 100 grams of sample (mg GAE 100 g^{-1}).

Total flavonoids (TF)

Total flavonoids content of samples was determined based in the method proposed by Zhishen et al. [22], with some modifications. Aliquots (0.2 mL) of samples, prepared as previously described, and 1.8 mL of distilled water, and 0.12 mL of $NaNO_2$ (5%, w/v) were added to amber bottles and mixed. After 5 min, 0.12 mL of $AlCl_3$ (10%, w/v) was added; and after 6 min, 0.8 mL of NaOH (1 mol L^{-1}) and 0.96 mL of distilled water were added. Absorbance was measured at 510 nm in UV-Vis spectrophotometer (UV-1800 Shimadzu). The catechin was used as the standard for a calibration curve and the results were expressed as mg of catechin equivalent (CE) per 100 g of sample.

Antioxidant capacity

The $DPPH^{\bullet}$ assay was performed based at method described by Brand-Williams et al. [23]. A 3.9 mL aliquot of a 6×10^{-5} mol L^{-1} $DPPH^{\bullet}$ methanolic solution was mixed with 100 µL of samples methanol:H_2O solutions, previously described. The $DPPH^{\bullet}$ absorbance was monitored at 515 nm after 1 h. The quantification was performed using a Trolox analytical curve, and the results were expressed as µmol of Trolox equivalent antioxidant capacity (TEAC) per 100 g of sample (µmol TEAC.100 g^{-1}).

The ABTS method was performed based at procedure described by Re et al. [24]. ABTS was dissolved in water to a 7 mmol L^{-1} final concentration. This solution (5 mL) was mixed with 88 µL potassium persulfate solution (140 mmol L^{-1}) and then incubated in the dark for 16 h at room temperature to produce a stock solution of the radical cation ($ABTS^{\bullet+}$). The $ABTS^{\bullet+}$ working solution was prepared by diluting the stock solution with absolute ethanol until reaching an absorbance of 0.700 ± 0.020 at 734 nm. For the samples analyses, aliquots of methanol:H_2O solutions prepared as previously described, and methanol up to 30 µL was added to amber bottles and mixed with 3 mL of the $ABTS^{\bullet+}$

radical cation working solution ($A_{734\ nm}$ = 0.700 ± 0.020). Absorbance readings were taken at 734 nm after 6 min. Quantification was conducted using a Trolox analytical curve, and the results were expressed as μmol of Trolox equivalent antioxidant capacity (TEAC) per 100 g of sample (μmol TEAC.100 g^{-1}).

Thermal behavior of tucumã pulp oil

Thermal analyses of tucumã pulp oils extracted using compressed propane and Soxhlet methods were conducted to evaluate how different extraction techniques affect the oil's thermal properties. For both thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC), the operational condition with the highest yield was chosen for the compressed propane extraction, based on the industrial preference for processes with greater yields.

Thermogravimetric analysis (TGA)

The thermal stability of tucumã pulp oils extracted using different methods was assessed through thermogravimetric analysis with the TGA 4000 equipment (Perkin Elmer Inc., Waltham, MA, USA). For the compressed propane extraction (CPE), the condition yielding the highest amount of oil was selected. Approximately 10 mg of each tucumã oil sample was placed in a platinum pan and heated from 30 °C to 750 °C (at a rate of 10 °C per minute) in an oven under a synthetic air atmosphere with a flow rate of 50 mL per minute. The data were analyzed using Pyris™, and both thermogravimetric (TG) and derivative thermogravimetric (DTG) curves were processed with Origin 8.6 software (OriginLab, Massachusetts, USA).

Differential scanning calorimeter (DSC)

The differential scanning calorimeter were performed using a DSC (850 Perkin Elmer Inc. Waltham, USA) equipped with nitrogen (N_2, 99.999% purity) as purge gas. Tucumã pulp oil samples obtained from different extraction methods were analyzed. For CPE, the condition with the highest oil yield was chosen. Approximately 4 mg of oil were weighed and sealed in an airtight aluminum pan, then a cooling and heating tests were

carried out. The following time-temperature setting was used: equilibration at 20 °C for 5 min to ensure complete sample temperature homogenization; heating to 50 °C at 5 °C min^{-1} and holding for 5 min, followed by cooling to -80 °C at -5 °C min^{-1} to induce crystallization, keeping this temperature for 5 min. Finally, heating was carried out from -80 to 50 °C at 5°C min^{-1} to obtain the melting profile [25].

STATISTICAL DATA ANALYSIS

The experimental design was conducted using Statistica 7.0 software (Statsoft Inc., Tulsa, OK, USA), where data were analyzed for variance (ANOVA) and significant differences were assessed using Tukey's test at a 5% significance level. The chemical characterization results of the extracts were reported as mean ± standard deviation (SD). Graphs were generated with Origin 8.6 software (OriginLab, Northampton, MA, USA).

RESULTS AND DISCUSSION

RAW MATERIAL CHARACTERISTICS

The average particle diameter was 1.59 mm. The proximate composition of the freeze-dried tucumã pulp showed the following results: 5.10 ± 0.30 wt% moisture; 2.57 ± 0.08 wt% ash; 4,78 ± 0.11 wt% proteins; 35.21 ± 0.10 wt% lipids and 52.34 wt% total carbohydrates. The tucumã pulp presented a high lipid content, surpassing that of umari pulp (25.25 wt%) [3], uxi (25.61 wt%) [26], and inajá (28.77 wt%) [15], which are important Amazonian oil crops.

YIELDS AND EXTRACTION KINETICS

The oil yield obtained from tucumã pulp using Soxhlet extraction was 37.0±0.1wt% (dry basis) over 6 h with 11 solvent cycles. **Table 1** presents the oil yields obtained with the compressed propane, showing the highest yield (33.9 wt%) at 80 °C and 3 mL/min, while the lowest yield (31.5 wt%) was obtained at 40 °C and 1 mL/min.

The Soxhlet extraction yield was used as a reference for calculating the propane extraction efficiency. Although the highest extraction yield was achieved with hexane, the yields obtained using CPE were comparable, with extraction efficiency exceeding 90% under optimal conditions. Moreover, the extraction time with CPE is significantly shorter (70 min), and there is no need for subsequent steps to remove residual solvent from the final sample. This demonstrates that CPE can be an excellent green option for extracting oil from tucumã pulp in terms of yield and efficiency.

Table 1 - Experimental conditions and results of tucumã pulp extracts obtained with compressed propane.[@]

Run	Pressure (MPa)	T (°C)	Flow rate (mL min^{-1})	Density[#] (kg/m^3)	Extraction yield**(wt%)	Extraction efficiency[##] (%)
1	10	40	1	495.50	31.5	85.2
2	10	40	3	495.50	33.5	90.4
3*	10	60	2	467.20	33.3 ± 0.2	90.0
4	10	80	1	435.25	31.6	85.2
5	10	80	3	435.25	33.9	91.4

[@]Experimental design (2^2).

*Center points with mean and standard deviation values from three extractions.

[#]propane density in the extraction condition, obtained from the NIST database.

**Yields calculated in dry basis.

[##] Mass of extract obtained with propane by mass of extract obtained with hexane in Soxhlet.

Within the evaluated working range, the variation in flow rate significantly impacted ($p < 0.05$) the extraction yield of tucumã pulp, as demonstrated by the Pareto chart (**Figure 1**). This diagram highlights the influence of the independent variables (flow rate and temperature) on the overall extraction yield. Conversely, temperature did not significantly affect the overall extraction yield when using compressed propane. Freitas et al. [3] reported similar results using compressed propane to extract oil from umari pulp fruit.

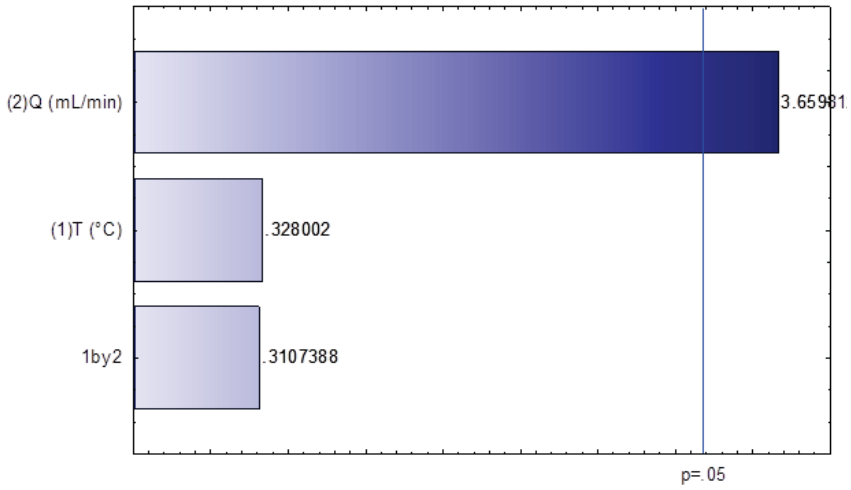

Figure 1 - Pareto chart showing the effects of process parameters on the extraction yield of tucumã pulp oil.

Figure 2 shows the overall extraction curves obtained using compressed propane as the solvent. The conditions of 80 °C with 3 mL/min, 60 °C with 2 mL/min, and 40 °C with 3 mL/min produced overall extraction curves with similar profiles. During the initial stage, from the start of dynamic extraction up to about 20 min, a constant extraction rate (CER) was observed. This was due to the high availability of oil on the surface of the particles, allowing for the efficient extraction of this readily accessible solute. At this phase, mass transfer primarily occurs through convection [27]. The falling extraction rate (FER) was observed during the intermediate period of 20 to 35 min. During this phase, the solute layer surrounding the particles begins to develop flaws, initiating the diffusion of solute into the interior of the particles. At this stage, both convection and diffusion contribute to the mass transfer process. Subsequently, the extraction rate decreases as the readily available oil is depleted, leading to the third stage known as the diffusional constant rate (DCR). In this final stage, solubility is constrained by internal diffusion mechanisms, with extraction primarily occurring through the diffusion of the solvent into the particle and the diffusion of the extracted solute to the particle's surface. Similar behavior was observed in a recent study on the extraction of umari pulp oil using compressed propane [3]. This pattern has also been observed in other studies [15,16,28,29].

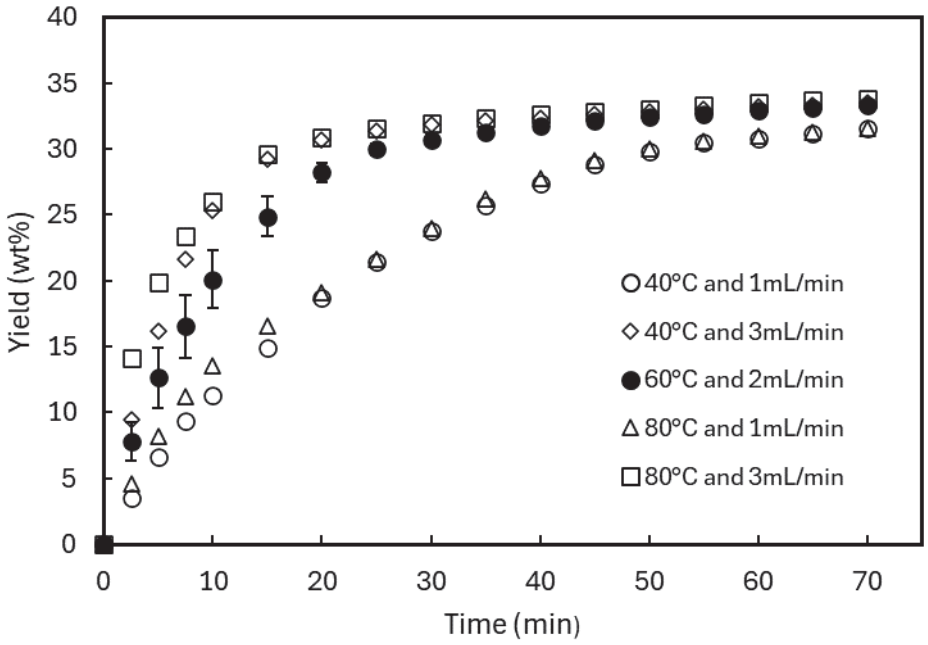

Figure 2 - Experimental overall extraction curves for tucumã pulp extraction using compressed propane as solvent.

The curves obtained at 40 °C with 1 mL/min and 80 °C with 1 mL/min exhibited very similar behavior, differing slightly from the other curves. This can be attributed to the low solvent flow rate in the extraction bed, which reduced the intensity of propane's contact with the particles. The curves remained within the constant extraction rate (CER) phase until about 55 min, when they entered the falling extraction rate (FER) phase, but did not reach a stability plateau. This indicates that additional time and solvent would be necessary to achieve higher extraction yields and fully exhaust the oil that compressed propane can extract.

Considering extraction yield as the primary parameter of interest, the condition of 80 °C and 3 mL/min can be deemed the best option, as it produced the highest extraction yield. However, the condition of 40 °C and 3 mL/min also represents a strong choice, as it yields similar results while operating at a milder temperature. This lower temperature may help preserve thermolabile compounds in the extract and reduce operational costs, particularly in terms of energy consumption.

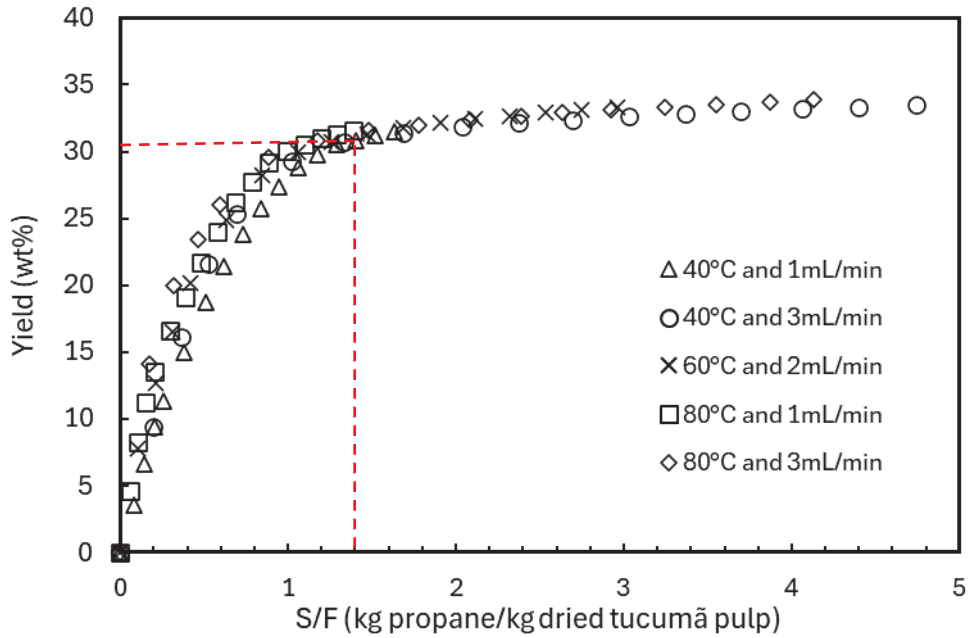

Figure 3 - Extraction curves using compressed propane at various temperatures and flow rates.

Figure 3 illustrates the extraction yields in relation to the mass ratio between solvent and feed (S/F). The results indicate that under all analyzed process conditions, an S/F ratio of 1.4 kg of propane per kg of dried tucumã pulp achieved a yield exceeding 30.0 wt% oil. This yield corresponds to more than 80% of the total oil content in tucumã pulp, based on the Soxhlet extraction yield (37.0 wt%) and assuming it fully recovers the lipid fraction from the tucumã pulp. Beyond this point (S/F = 1.4 kg), continuing the extraction process becomes less advantageous. The curves on the graph have nearly reached their plateau, indicating that further oil recovery would be minimal and not cost-effective from an engineering and process standpoint. According to Goto et al. [30], when extraction curves follow a single trend line, it suggests that the process is primarily governed by the solubility of the easily accessible solute, with minimal impact from internal diffusion mechanisms. Similar graphic profiles were observed by Han et al. [31] and Jesus et al. [32], aligning with the findings of this study.

OIL CHARACTERIZATION

Fatty acid composition of tucumã pulp oil

The fatty acid composition obtained by the two extraction methods (compressed propane and Soxhlet) are expressed in **Table 2**. The results showed similar values regardless of the extraction method used and the different extraction conditions used for compressed propane. Five fatty acids were identified (palmitic, stearic, oleic, linoleic, linolenic), of which oleic acid (59.76-60.72%) and palmitic acid (29.74-30.87%) were the predominant ones in the tucumã pulp oil, a profile quite similar to the oils from the pulp of umari fruit [3] and inajá [15] obtained with compressed propane. Menezes et al. [33] also found similar values for tucumã pulp (65.4 and 26.6% for oleic and palmitic acids, respectively), using supercritical CO_2 at 40 °C and 400 bar. The oil from tucumã pulp has a predominance of unsaturated fatty acids (65.72-67.24%), being higher than the concentration of saturated fatty acids (32.76-34.28%).

Tucumã pulp oil is rich in oleic acid (OA), which has several benefits and industrial applications due to its chemical and biological properties. OA is widely used in cosmetic and pharmaceutical products, where it acts as an emollient and vehicle for active ingredients, providing hydration and better absorption of components by the skin [34]. In addition, OA is a monounsaturated fatty acid with antioxidant, anti-inflammatory, cardioprotective and metabolic properties and is applicable in preclinical and clinical trials [35]. In fact, it has been shown that the incorporation of OA into topically applied nanostructured lipid carriers alleviated the severity of skin inflammation in a murine model of cutaneous inflammation, demonstrating its applicability for the pharmaceutical industry in transdermal treatments and for nanotechnology-based therapies [36]. The infusion of OA in an animal model indicated that this fatty acid signals metabolic and neuronal events within the central nervous system (CNS) designed to control food intake by its action on the hypothalamic axes and by mimicking the action of insulin and leptin, as an approach for the treatment of obesity and type 2 diabetes [37].

Studies indicate that replacing saturated fats with monounsaturated fatty acids, such as oleic acid, in the diet can lead to a significant decrease in total and LDL cholesterol levels, thus reducing the risk of cardiovascular disease [38]. It was demonstrated that oral administration of OA over two consecutive days led to a decrease in cardiac hypertrophy indices and biomarkers of cardiac injury (LDH, CK-MB, cardiac troponin-I, MMP-9), as well as a reduction in heart rate in an experimental model of myocardial injury (MI). Additionally, there was a notable reduction in lipid peroxidation levels and an enhancement of the antioxidant response [39]. Furthermore, dietary

incorporation of OA reduced inflammatory markers in the retina, modulating the inflammatory response and reestablishing ocular microvascular integrity in an animal model [40].

Moreover, OA is employed in the production of biolubricants, which are sustainable alternatives to petroleum-derived lubricants, offering lower toxicity and greater biodegradability [41]. In the food industry, it is valued for its ability to improve the oxidative stability of oils and margarines, contributing to a longer shelf life of products. Its antioxidant and anti-inflammatory properties are also explored in dietary supplements, promoting cardiovascular health benefits and supporting the immune system. In sum, the versatility of oleic acid makes it an essential component in various industries, driving innovation and sustainability [39,42].

Table 2 - Fatty acid composition (wt%) of tucumã pulp oil extracted by compressed propane and Soxhlet.

Fatty acids	**propane (40 °C/10 MPa/ 1 mL min^{-1})**	**propane (40 °C/10 MPa/ 3 mL min^{-1})**	**propane[a] (60 °C/10 MPa/ 2 mL min^{-1})**	**propane (80 °C/10 MPa/ 1 mL min^{-1})**	**propane (80 °C/10 MPa/ 3 mL min^{-1})**	**Soxhlet (Hexane)**
Palmitic (C16:0)	30.37	29.74	30.55	30.24	30.29	30.87
Stearic (C18:0)	2.42	3.03	3.73	2.64	3.64	3.31
Oleic (C18:1)	60.64	60.07	59.76	60.04	60.58	60.72
Linoleic (C18:2)	1.99	2.56	2.94	2.87	2.66	3.08
Linolenic (C18:3)	4.58	4.60	3.02	4.21	2.84	2.02
∑ SFA[1]	32.79	32.76	34.28	32.88	33.92	34.18
∑ MUFA[2]	60.64	60.07	59.76	60.04	60.58	60.72
∑ PUFA[3]	6.57	7.16	5.96	7.08	5.50	5.10
∑ PUFA + MUFA	67.21	67.24	65.72	67.12	66.08	65.82

The standard deviation for all fatty acids was lower than 0.8%.

[a] Center point for compressed propane extraction.

[1] Saturated fatty acids.

[2] Monounsaturated fatty acids.

[3] Polyunsaturated fatty acids.

β-Carotene content

The results expressed in **Table 3** show high β-carotene (BC) contents in all extraction conditions with compressed propane, ranging from 200.28±3.65 to 208.87±2.39 mg $100g^{-1}$, with the highest values being obtained in lower temperatures. On the other hand, the oil obtained by Soxhlet showed a BC content (9.56±1.74 mg $100g^{-1}$) much lower than that obtained with CPE, which may have occurred due to the long extraction period (6 h) in the Soxhlet method and the need for a second solvent separation step, which was carried out in an oven at 60 °C for several hours. Since β-carotene is a thermosensitive compound, exposing the extract to relatively high temperatures for extended periods may have caused its degradation. This degradation is evidenced by the reduction in orange pigmentation in the extract after all process steps, as shown in **Figure 4**.

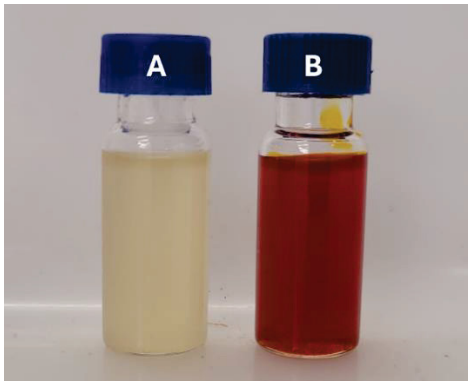

Figure 4 - Tucumã oils obtained with Soxhlet (A) and compressed propane (B).

The compressed propane provided tucumã pulp oil with 21 times more β-carotene compared to the oil obtained using the Soxhlet method. Additionally, the β-carotene was not degraded in the subsequent steps of organic solvent separation, as this step is not required. In addition, CPE is a green extraction method with reduced extraction time when compared to the Soxhlet method. As carotenoids are lipophilic compounds of low polarity, propane becomes an excellent solvent for obtaining BC. Furthermore, the efficiency of propane for BC extraction can be justified due to the high penetrating ability of the pressurized fluid, solubilizing carotenoids by convective and diffusive mechanisms. Menezes et al. [33] found BC content of 135.17 mg $100g^{-1}$ in tucumã oil obtained with supercritical CO_2 at 40 °C and 400 bar, values lower than those found in the present work, which corroborates the efficiency and selectivity of propane for BC

extraction. Ferreira et al. [43] and Santos et al. [44] reported BC values of 77.82 and 51.12 mg 100 g^{-1}, respectively, for tucumã oils obtained by conventional extraction techniques.

β-carotene is a carotenoid found in abundance in tucumã oil obtained with compressed propane, presenting numerous benefits for human health. This compound is a precursor of vitamin A, essential for vision, cell growth and the immune system [45]. Studies indicate that adequate intake of β-carotene can reduce the risk of chronic diseases, such as cardiovascular disease and certain types of cancer, due to its antioxidant properties that fight free radicals in the body [46]. In the cosmetic industry, β-carotene is widely used in skin care products, as it helps to improve the appearance of the skin, protecting it against damage caused by UV radiation and delaying signs of aging [47,48]. In the food industry, it is often added as a natural coloring and nutritional supplement, enhancing the value of products due to its health-beneficial properties [49].

The Amazon offers a promising source of β-carotene, especially through fruits and vegetables native to the region, such as buriti, peach palm and tucumã. The sustainable exploitation of these resources can promote the bioeconomy, encouraging practices that combine economic development and environmental conservation. The extraction and commercialization of β-carotene from Amazonian sources using green methods can generate employment and income for local communities, in addition to adding value to regional products.

Table 3. β-Carotene, total phenolic contents, total flavonoids and antioxidant capacity for tucumã pulp oil obtained from compressed propane and Soxhlet extraction.

Run	Extraction conditions	Solvent	β-Carotene (mg 100 g^{-1})	TPC (mg GAE 100g^{-1})	TF (mg CE 100 g^{-1})	DPPH (µmol TEAC 100 g^{-1})	ABTS (µmol TEAC 100 g^{-1})
1	40 °C; 1 mL min^{-1}	Propane	208.87±2.39	n.d.	606.17±7.47	62.94±0.63	165.66±7.58
2	40 °C; 3 mL min^{-1}	Propane	207.18±2.60	n.d.	510.18±4.16	70.24±0.63	236.06±13.85
3*	60 °C; 2 mL min^{-1}	Propane	204.22±3.32	0.71±0.06	442.89±4.45	52.43±0.50	171.88±6.89
4	80 °C; 1 mL min^{-1}	Propane	200.28±3.65	n.d.	510.84±4.24	61.94±0.31	161.38±3.80
5	80 °C; 3 mL min^{-1}	Propane	205.73±2.61	0.24±0.02	499.25±4.38	61.99±0.85	152.82±6.78
6	Soxhlet; 68 °C	Hexane	9.56±1.74	1.11±0.02	752.59±22.14	71.81±0.53	216.57±4.25

* Triplicate at central point for compressed propane extraction.

n.d.: not detected

Total phenolic content (TPC) and total flavonoids (TF)

The TPC and TF results for tucumã pulp oils obtained with compressed propane and Soxhlet are expressed in **Table 3**. The oils showed low TPC values (0.24±0.02 to 1.11±0.02 mg GAE $100g^{-1}$) in all extractions (CPE and Soxhlet), even not being detected in some conditions analyzed. According to Boni et al. [50] tucumã oil does not contain significant values of phenolic compounds, but has a very high carotenoid content, corroborating the results obtained in the present study. On the other hand, the oil obtained with CPE presented relevant TF results, ranging from 442.89±4.45 to 606.17±7.47 mg CE 100 g^{-1}, with the highest value found in the condition of lower temperature and solvent flow rate (40 °C and 1 mL min^{-1}), suggesting a possible influence of temperature for the maintenance of flavonoids in the oil sample. The tucumã pulp oil obtained by Soxhlet presented a higher TF value (752.59±22.14 mg CE 100 g^{-1}) when compared to the oils obtained by CPE. This difference can be attributed to the selectivity of the methods studied and to the differences in polarity of the solvents used in the different extraction techniques. Comparing with data obtained from the extraction of macaúba pulp oil using compressed propane by Trentini et al. [5] the results obtained in this present work were higher than the reported range (11.03 ± 0.07 to 13.12 ± 0.10 mg CE 100 g^{-1}).

Antioxidant capacity

Table 3 shows the results obtained for two antioxidant assays (DPPH and ABTS) for oils extracted by compressed propane and Soxhlet. The DPPH radical scavenging capacity of the samples extracted using compressed propane exhibited a range from 52.43±0.50 to 70.24±0.63 μmol TEAC 100 g^{-1}, with the highest value obtained in the condition of 40 °C/3 mL min^{-1} and 40 °C/1 mL min^{-1}, suggesting that the use of lower temperatures is an important factor in preserving thermosensitive bioactive compounds, such as β-carotene. The result obtained for the oil extracted by Soxhlet was superior (71.81±0.53 μmol TEAC 100 g^{-1}), which may be related to solvent selectivity and affinity for certain bioactive compounds.

Concerning the results obtained for ABTS, there was a variation from 152.82±6.78 to 236.06±13.85 μmol TEAC 100 g^{-1}, with the highest value observed at 40 °C and 3 mL min^{-1}. The condition of 40 °C and 3 mL min^{-1} showed the highest values of

antioxidant capacity, even surpassing those obtained by Soxhlet (216.57±4.25 μmol TEAC 100 g^{-1}), suggesting that the use of lower temperatures may have contributed to the maintenance of some thermosensitive bioactive compounds. This supports the higher values obtained for β-carotene under lower temperature conditions, which are frequently cited in the literature as being responsible for substantial antioxidant activity [3,15,19].

Thermal behavior of tucumã pulp oil

Thermogravimetric analysis (TGA)

The thermogravimetric analysis yielded curves representing mass reduction (TGA) and the rate of mass reduction (DTG) for tucumã pulp oil extracted using hexane and compressed propane, respectively (**Figures 5A and 5B**). Because of the nearly identical fatty acid composition, both solvents displayed comparable thermal characteristics, as outlined in **Table 4**, regarding the phases of mass decomposition.

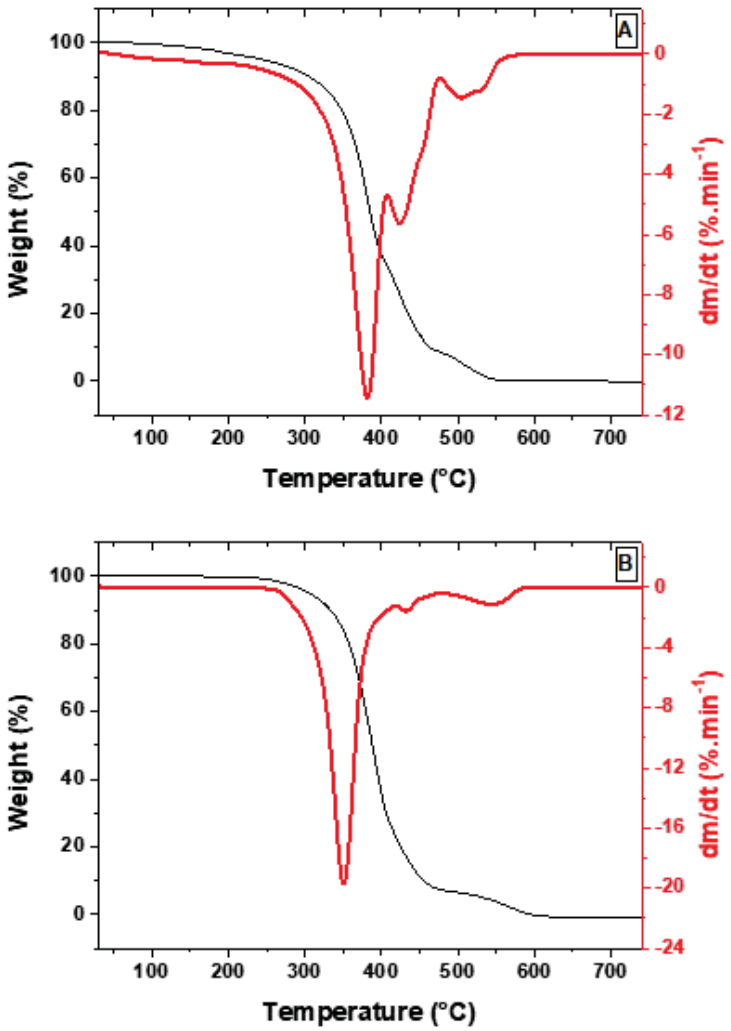

Figure 5 - TGA and DTG curves represented by the red and black lines, respectively, for tucumã pulp oil extracted using (A) hexane (Soxhlet) and (B) compressed propane (80 °C/10 MPa/3 mL min^{-1}).

As shown in **Figures 5A** and **5B**, mass degradation occurs in three main stages (**Table 4**), evidenced by three peaks. The first stage (I) could be associated with thermal decomposition and the onset of polyunsaturated fatty acid triglycerides degradation. The following steps (II and III) correspond to the decomposition of monounsaturated and saturated fatty acids, in addition to peroxides and hydroperoxides formed by the polymerization of fatty acids degradation products from the initial phase [15,51,52].

Table 4 - TGA and DTG curve data for the extraction of tucumã pulp oil using hexane and compressed propane.

Solvent	Phase of thermal degradation	T_i (°C)	T_m (°C)	T_f (°C)	Δm (%)
Hexane	Stability	30.17	-	256.21	-
	I	256.21	380.90	407.62	65.80
	II	407.62	429.55	472.85	19.41
	III	472.85	507.12	564.51	11.98
Propane 80 °C/3 mL min^{-1}	Stability	30.17	-	263.67	-
	I	263.67	350.47	417.10	92.22
	II	417.10	432.70	463.62	1.17
	III	482.08	546.88	579.79	5.79

T_i, initial temperature; T_m, maximum mass loss temperature; T_f, final temperature; Δm, mass loss.

In this study, extraction with compressed propane provided slightly superior thermal stability of the oil, as mass degradation initiates at 263.67 °C, while oil extracted with hexane begins to degrade at 256.2 °C. Very similar degradation temperature values were obtained by Freitas et al. [3] for umari pulp oil, where the same solvents were evaluated under identical conditions. Barbi et al. [15] also reported a similar thermal behavior profile in the TGA results for inajá pulp oil. During the inajá oil extraction using compressed propane (at 10 MPa, 20 °C, and a flow rate of 2 mL min^{-1}), mass degradation began at 269 °C and presented three peaks (at 368 °C, 424 °C, and 562 °C). This indicates a similarity in the fatty acid composition between the oils from these matrices (inajá, tucumã, and umari).

Differential scanning calorimeter (DSC)

Differential Scanning Calorimetry (DSC) was used to generate the cooling curves (**Figure 6A**) and heating curves (**Figure 6B**) of tucumã pulp oil, extracted using hexane and compressed propane as solvents. In general, the thermal profiles exhibited striking resemblance between the solvents, not only in the temperatures at which events occurred but also in their intensity. This similarity was anticipated due to the oils having nearly identical fatty acid compositions.

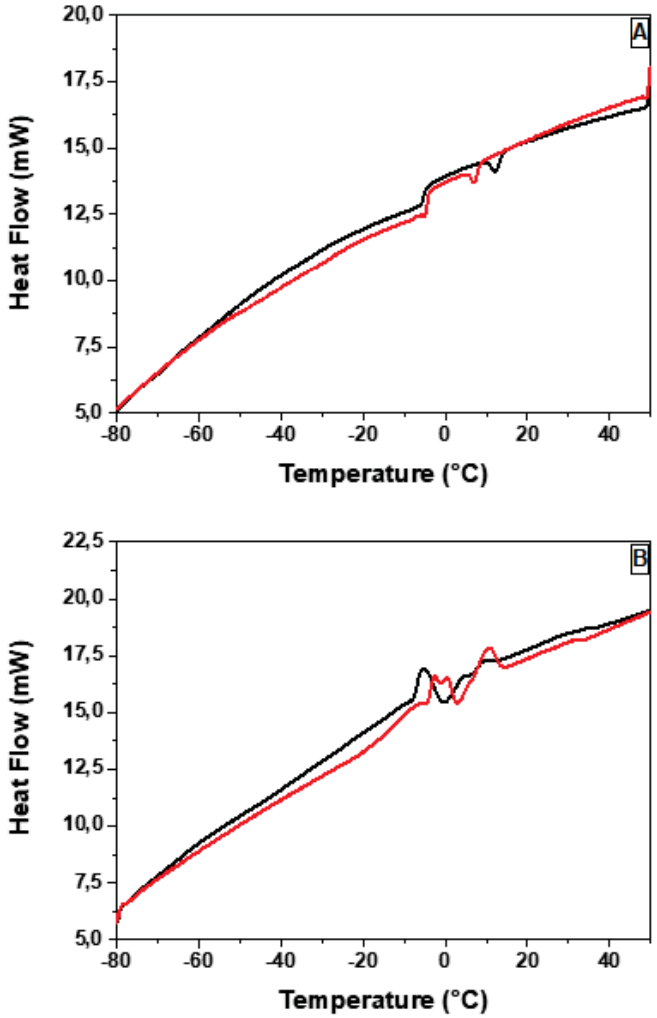

Figure 6 - DSC curves of tucumã pulp oil extracted using hexane (Soxhlet), represented by black lines, and compressed propane (80 °C/10 MPa/3 mL min^{-1}), represented by red lines. (A) crystallization (50 to -80 °C) and (B) melting (-80 to 50 °C) behavior.

Figure 6A indicates two main exothermic peaks. The first peak occurs at 13 °C, relating to hexane extraction, and at 7 °C relating to compressed propane extraction. At approximately -5°C there is the second crystallization peak, for both solvents, when samples are completely crystallized. The different exothermic crystallization phases are likely due to the presence of different fractions of a specific type of triacylglycerol [53,54]. The initial phase of crystal formation is associated with the rearrangement of molecules caused by the presence of highly saturated triacylglycerols. The final phase of crystallization results from the aggregation and compaction of these molecules [55,56].

Figure 6B present endothermic peaks related to sample melting. The oil obtained from extraction with hexane begins to melt at -8°C, while that obtained from extraction with compressed propane begins at -4°C. Both samples are completely liquid at 14°C.

Freitas et al. [3] and Fetzer et al. [16], applied DSC thermal analysis to umari pulp and cumaru seed oils, respectively (also extracted with hexane and compressed propane), and obtained thermal behavior similar to that of the present study, with complete melting of the samples at approximately 19°C. Generally, the higher the degree of unsaturation in fatty acids, the lower their melting point [57].

CONCLUSION

The application of compressed propane for extracting tucumã pulp oil was successfully carried out, yielding high extraction rates and efficiency, making it a green alternative to conventional methods. Tucumã pulp oils obtained with CPE exhibited superior characteristics for β-carotene (a vitamin A precursor), with values 21 times higher than those obtained by Soxhlet, which degraded the bioactive compound. Additionally, CPE produced oil rich in oleic acid (omega-9), maintaining values similar to those obtained with Soxhlet. The preservation of these bioactives in tucumã pulp oil adds value to the production chain of this species, as they can be applied in various industrial segments, generating income for local producers and boosting the Amazonian bioeconomy.

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CHAPTER IV

EXTRACTION OF TUCUMÃ-DO-AMAZONAS (*Astrocaryum aculeatum*) ALMOND OIL USING COMPRESSED PROPANE AS SOLVENT

This chapter is part of the article entitled “Extraction of tucumã-do-Amazonas (*Astrocaryum aculeatum*) almond oil using compressed propane as solvent”**.**

EXTRACTION OF TUCUMÃ-DO-AMAZONAS (*Astrocaryum aculeatum*) ALMOND OIL USING COMPRESSED PROPANE AS SOLVENT

ABSTRACT

Tucumã-do-Amazonas almonds (TAA) are considered an agro-industrial byproduct, being usually discarded. Therefore, this research aimed to study TAA by applying the green extraction method, compressed propane extraction (CPE), to obtain high-quality oils, in addition to compare it with the conventional Soxhlet extraction method. For CPE, temperature and solvent flow parameters were varied. The obtained oils were characterized for global yields, fatty acid composition, β-carotene content, antioxidant activity, total phenolics content (TPC), total flavonoids (TF), and thermal behavior. The results showed the highest extraction yield (35.9 wt%) at 80 °C and 1 mL/min, with an extraction efficiency of 96.5%. The variation in temperature showed a significant effect ($p < 0.05$) on the oil extraction yield. The obtained oil is rich in lauric and myristic acids, presenting good thermal stability, and has potential for industrial applications as it is similar to coconut oil.

Keywords: Bioeconomy, Fatty acids, Oil, Extraction, Amazon.

INTRODUCTION

The development of the Amazonian bioeconomy highlights the increasing necessity to use native species from this region in a sustainable way. In this context, tucumã-do-Amazonas (*Astrocaryum aculeatum*) emerges as an important raw material with technological potential, as it contains bioactive compounds that can be applied in various industrial sectors [1,2]. The tucumã-do-Amazonas pulp is widely consumed, especially in the city of Manaus, Amazonas, Brazil, where it is a key ingredient in a traditional local dish known as “X-caboquinho”. This sandwich, which features tucumã pulp as its main ingredient, is one of the most popular local delicacies in the region. However, once the pulp is extracted, the tucumã almond remains and is classified as an agro-industrial byproduct.

The tucumã-do-Amazonas almond (TAA) is often discarded as agro-industrial waste and remains underutilized for industrial purposes. This fruit fraction is rich in lipids and has a fatty acid profile similar to that of coconut oil, predominantly containing lauric and myristic acids. This composition suggests significant potential for applications in the cosmetic, food, and pharmaceutical industries, offering an opportunity to add value to this byproduct and reduce its environmental impact [3,4].

Among the applications of TAA oil, its use in cosmetic formulations is particularly noteworthy. It can be incorporated into creams, lotions, shampoos, and conditioners due to its moisturizing, emollient, and antioxidant properties, which help to keep the skin and hair hydrated and protected against free radical damage [5,6]. In the food industry, TAA oil can be incorporated into products such as chocolates and margarine, enhancing their texture and stability while also functioning as a natural antimicrobial agent [5,7]. The predominant fatty acids in TAA oil, lauric and myristic acids, exhibit neuroprotective, anticancer, anti-inflammatory, and antibacterial activities, making them suitable for application in the pharmaceutical industry [2,3,5].

Given the numerous advantages and versatility of TAA oil, it is essential to employ an environmentally friendly extraction method, such as compressed propane extraction (CPE). This method is highly effective for extracting nonpolar compounds, such as lipids. Compressed propane has been considerably used for oil extraction from oilseeds, as it yields products that are completely free of organic solvents, eliminating the need for additional purification steps. Furthermore, propane enables relatively rapid

extraction and can be efficiently recovered and recycled in the extraction process, thereby reducing solvent consumption costs. [8–10]

As there are no published studies involving the extraction of TAA oil using compressed propane, the objective of this work is to obtain TAA oil using CPE and compare it with the conventional Soxhlet extraction method, highlighting the advantages of the new approach. The research also aims to characterize the obtained oils and suggest potential applications for it within the Amazonian bioeconomic scenario, seeking to increase the value of the species and promote the economic development of the region.

MATERIALS AND METHODS

TUCUMÃ-DO-AMAZONAS FRUIT SAMPLES

Sample preparation

The tucumã-do-Amazonas (*Astrocaryum aculeatum*) used in this study were collected in Cametá, Pará, Brazil (2° 15′ 15″ S, 49° 30′ 44″ W) (SisGen: AE72BF1). The fruits were sanitized using a sodium hypochlorite solution at 200 ppm. The seeds were then separated from the pulp and dried in a convection oven at 50 °C for 18 h (Fabbe, São Paulo, Brazil). The dried seeds were mechanically broken with a bench mechanical press, thus obtaining the tucumã almond. The almonds were cut into smaller pieces and ground in a knife mill. The ground material was classified with different Tyler series sieves (8, 12, 20, 24, 32 meshes) attached to a mechanical shaker (Produtest, São Paulo, Brazil). The average particle diameter was determined by the method described by Souza et al. [11]. Finally, all fractions were mixed, vacuum-sealed in low-density polyethylene bags, and stored in a freezer at -5 °C until analysis.

Proximate composition

The proximate composition of the dried TAA was determined by analyzing the moisture, lipid, protein, ash, and carbohydrate contents following the AOAC [12] methodologies. The moisture content was measured by drying the sample in an oven at

105 °C until it reached a constant weight. The ash content was determined by incinerating the samples in a muffle furnace at 600 °C. Lipid content was measured using the Soxhlet method with hexane as solvent. Protein content was quantified using the Kjeldahl method with a nitrogen conversion factor of 6.25. The carbohydrate content was calculated by subtracting the sum of the moisture, ash, lipid, and protein contents from 100.

TAA OIL EXTRACTION

Soxhlet extraction

Soxhlet extraction yields were determined in triplicate using *n*-hexane (Neon, 99.95% purity) as the solvent. Approximately 5 g of ground TAA sample and 150 mL of solvent were refluxed to its boiling point in a Soxhlet extractor apparatus (UNIGLAS, Campinas, São Paulo, Brazil) for 6 h. After extraction, the solvent was removed from the oil using a rotary vacuum evaporator (Model RV 10 digital, IKA) and then placed in an oven at 60 °C to evaporate any remaining solvent residues. The oil was stored in an amber glass vessel and kept at -5 °C until further analysis. The percentage extraction yield was calculated in dry basis, according to **Equation (1)**.

$$Y_{(\%db)} = \left(\frac{m_o}{m_s\left(1-\frac{U_s}{100}\right)}\right)100 \quad (1)$$

Where: $Y_{(\%db)}$ is the yield in dry basis, m_o is the obtained extract mass, m_s is the sample mass used, and U_s is the percentage moisture of the freeze-dried tucumã pulp.

Compressed propane extraction (CPE)

Extracts were obtained using a laboratory-scale supercritical extraction system equipped with an extraction vessel measuring 22 cm in length, 1.9 cm in diameter, and with an internal volume of 62.4 mL, as detailed in earlier research by our group [9,13]. The extraction setup includes an extractor vessel connected to an ultra-thermostatic bath for temperature regulation, a syringe pump (ISCO, model 500D, Lincoln, NE, USA) for pressurizing the propane, a needle valve to control the solvent flow rate inside the extractor, along with temperature and pressure sensors to monitor the process. The

syringe pump was set to 10 °C for all extractions using an ultra-thermostatic bath. The extraction temperature was maintained and regulated by a separate bath attached to the extractor jacket. The oils were collected at atmospheric pressure and room temperature, with propane of 99.5% purity (White Martins S.A., Araucária, PR, Brazil) used in the process.

Approximately 32.5 g of dried TAA were utilized for each extraction. The static phase (confinement) was set for 30 min, based on previous studies by our research group. The dynamic phase lasted 70 min, with the extract mass being measured at 2.5-min intervals during the first 10 min, and subsequently at 5-min intervals. In this study, the pressure was maintained at 10 MPa, since the literature as well as the studies from our research group indicate that this variable has a minimal effect on the extraction yield of oil crops using compressed propane [14,15]. Thus, the solvent flow and temperature parameters were selected for variation. Although less frequently investigated in literature, the flow rate can offer valuable insights regarding engineering and process costs, and it is less studied compared to other variables such as temperature, pressure, and particle size. To investigate the optimal combination of two independent factors affecting the extraction process, extractions were conducted using a two-level, two-factor experimental design. This design for compressed propane involved varying temperatures (40 - 80 °C) and solvent flow rates (1.0 - 3.0 mL min^{-1}), with triplicate measurements at the central point. Extraction temperatures were monitored from the extractor vessel connected to an ultra-thermostatic bath, while solvent flow rates were controlled via a needle valve, with the syringe pump operating at 10 °C and a pressure of 10 MPa for all extractions. The overall extraction yield of crude oil was calculated in dry basis using **Equation 1**, as described in Section 2.2.1.

OIL CHARACTERIZATION

Fatty acid composition

The fatty acid composition of umari extracts was determined by Gas chromatography with flame ionization detector (GC-FID). Samples were prepared according to the AOCS Official Method Ce 2–66 for converting oils into fatty acid methyl esters (FAMEs) [16]. FAMEs were analyzed using a Shimadzu chromatograph (GC 2010

Plus), a capillary column (SH-Rtx-Wax, Shimadzu, 30 m x 0.32 mm, 0.25 μm), flame ionization detector (FID) and split injection (1:10). The injector and detector temperatures were 240 °C and 250 °C, respectively. The oven temperature was set to start at 100 °C and remain at this temperature for 5 min, followed by an increase to 240 °C at a rate of 4 °C min^{-1} and maintained at this temperature for 5 min. The carrier gas was helium at 32.5 cm^3 min^{-1}. FAMEs were identified by comparison with the retention times of a standard mixture of FAMEs (Supelco, MIX FAME 37, St. Louis, MO 63103, USA). Quantification of fatty acids was performed using the area normalization procedure. The results were expressed as a percentage of each fatty acid present in the sample.

β-Carotene content

The total β-carotene content was analyzed using the method developed by Young & Britton [17] and adapted by Cuco et al. [18]. In summary, a 10 mg sample of each oil was dissolved in 10 mL of *n*-hexane. The solution was then measured for absorbance at 450 nm using a UV-vis spectrophotometer (Spectro 3000W, Marte Científica, Brazil). The total β-carotene content was calculated using **Equation 2**. Results were obtained in triplicate and reported as milligrams of β-carotene per 100 grams of oil.

$$Total\ \beta\text{-}carotene = \frac{(Abs\ Vol\ 10^3)}{E_{1cm}^{1\%}\ W} \quad (2)$$

Where *Abs* is absorbance at 450 nm; *Vol* is the dilution volume (mL) and $E_{1cm}^{1\%}$ is the extinction coefficient (2592) of β-carotene in hexane proposed Ogawa et al. [19], and *W* is the mass of the sample (g).

Total phenolic content (TPC) and Total flavonoids (TF)

The total phenolic content (TPC) of umari oil extracted by different methods was determined using the Folin–Ciocalteu method [20]. Initially, 100 mg of oil was mixed with 1 mL of methanol (80:20, v/v), shaken, and then centrifuged for 10 min at 3000 rpm. To measure the TPC, 0.4 mL of the supernatant and 0.1 mL of methanol were combined with 2.5 mL of Folin-Ciocalteu reagent (diluted 1:10 in distilled water). This mixture was kept in the dark for 3 min, then 2 mL of a 7.5% (w/v) sodium carbonate solution was added, and the mixture was incubated in the dark for 2 h. Absorbance was then measured

at 760 nm using a UV/Vis Spectrophotometer (UV-1800 Shimadzu). All assays were conducted in triplicate. The results were quantified using a gallic acid calibration curve and expressed as milligrams of gallic acid equivalent (GAE) per 100 grams of sample (mg GAE 100 g^{-1}).

Total flavonoids content of samples was determined based in the method proposed by Zhishen et al. [21], with some modifications. Aliquots (0.2 mL) of samples, prepared as previously described, and 1.8 mL of distilled water, and 0.12 mL of $NaNO_2$ (5%, w/v) were added to amber bottles and mixed. After 5 min, 0.12 mL of $AlCl_3$ (10%, w/v) was added; and after 6 min, 0.8 mL of NaOH (1 mol L^{-1}) and 0.96 mL of distilled water were added. Absorbance was measured at 510 nm in UV-vis spectrophotometer (UV-1800 Shimadzu). The catechin was used as the standard for a calibration curve and the results were expressed as mg of catechin equivalent (CE) per 100 g of sample.

Antioxidant capacity

The $DPPH^{\bullet}$ assay was performed based at method described by Brand-Williams et al. [22]. A 3.9 mL aliquot of a 6×10^{-5} mol L^{-1} $DPPH^{\bullet}$ methanolic solution was mixed with 100 µL of samples methanol:H_2O solutions, previously described. The $DPPH^{\bullet}$ absorbance was monitored at 515 nm after 1 h. The quantification was performed using a Trolox analytical curve, and the results were expressed as µmol of Trolox equivalent antioxidant capacity (TEAC) per 100 g of sample (µmol TEAC 100 g^{-1}).

The ABTS method was performed based at procedure described by Re et al. [23]. ABTS was dissolved in water to a 7 mmol L^{-1} final concentration. This solution (5 mL) was mixed with 88 µL potassium persulfate solution (140 mmol L^{-1}) and then incubated in the dark for 16 h at room temperature to produce a stock solution of the radical cation ($ABTS^{\bullet+}$). The $ABTS^{\bullet+}$ working solution was prepared by diluting the stock solution with absolute ethanol until reaching an absorbance of 0.700 ± 0.020 at 734 nm. For the samples analyses, aliquots of methanol:H_2O solutions prepared as previously described, and methanol up to 30 µL was added to amber bottles and mixed with 3 mL of the $ABTS^{\bullet+}$ radical cation working solution ($A_{734\ nm}$ = 0.700 ± 0.020). Absorbance readings were taken at 734 nm after 6 min. Quantification was conducted using a Trolox analytical curve, and the results were expressed as µmol of Trolox equivalent antioxidant capacity (TEAC) per 100 g of sample (µmol TEAC 100 g^{-1}).

Thermal behavior of TAA oil

Thermal analyses of TAA oils extracted using compressed propane and Soxhlet methods were conducted to evaluate how different extraction techniques affect the oil's thermal properties. For both thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC), the operational condition with the highest yield was chosen for the compressed propane extraction, based on the industrial preference for processes with higher yields.

Thermogravimetric analysis (TGA)

The thermal stability of TAA oils extracted using different methods was assessed through thermogravimetric analysis with the TGA 4000 equipment (Perkin Elmer Inc., Waltham, MA, USA). For the compressed propane extraction (CPE), the condition yielding the highest amount of oil was selected. Approximately 10 mg of each TAA oil sample was placed in a platinum pan and heated from 30 °C to 750 °C (at a rate of 10 °C per minute) in an oven under a synthetic air atmosphere with a flow rate of 50 mL per minute. The data were analyzed using Pyris^{TM}, and both thermogravimetric (TG) and derivative thermogravimetric (DTG) curves were processed with Origin 8.6 software (OriginLab, Massachusetts, USA).

Differential scanning calorimeter (DSC)

The differential scanning calorimeter were performed using a DSC (850 Perkin Elmer Inc. Waltham, USA) equipped with nitrogen (N_2, 99.999% purity) as purge gas. TAA oil samples obtained from different extraction methods were analyzed. For CPE, the condition with the highest oil yield was chosen. Approximately 4 mg of oil were weighed and sealed in an airtight aluminum pan, then a cooling and heating tests were carried out. The following time-temperature setting was used: equilibration at 20 °C for 5 min to ensure complete sample temperature homogenization; heating to 50 °C at 5 °C min^{-1} and holding for 5 min, followed by cooling to -80 °C at -5 °C min^{-1} to induce crystallization, keeping this temperature for 5 min. Finally, heating was carried out from -80 to 50 °C at 5°C min^{-1} to obtain the melting profile [24].

STATISTICAL DATA ANALYSIS

The experimental design was conducted using Statistica 7.0 software (Statsoft Inc., Tulsa, OK, USA), where data were analyzed for variance (ANOVA) and significant differences were assessed using Tukey's test at a 5% significance level. The chemical characterization results of the extracts were reported as mean ± standard deviation (SD). Graphs were generated with Origin 8.6 software (OriginLab, Northampton, MA, USA).

RESULTS AND DISCUSSION

RAW MATERIAL CHARACTERISTICS

The average particle diameter was 2.02 mm. The proximate composition of the dried TAA showed the following results: 3.10 ± 0.21 wt% moisture; 1.92 ± 0.09 wt% ash; 5.67 ± 0.17 wt% proteins; 36.0± 0.20 wt% lipids and 53.21 wt% total carbohydrates.

YIELDS AND EXTRACTION KINETICS

The oil yield from TAA using Soxhlet extraction was 37.2±0.2wt% (dry basis) over 6 h with 12 solvent cycles. **Table 1** presents the oil yields obtained with compressed propane, with the highest yield (35.9 wt%) at 80 °C and 1 mL/min, and the lowest yield (34.2 wt%) at 40 °C and 3 mL/min. Carvalho et al. [3] found a maximum yield of 34.41 wt% using supercritical CO_2 at 250 bar and 50 °C, a lower yield than that found in this work, which demonstrates the efficiency of compressed propane for the extraction of oilseeds. The Soxhlet extraction yield was used as a reference for calculating the efficiency of propane extraction. Although the highest extraction yield was achieved with hexane, the yields obtained using CPE were comparable, with extraction efficiency exceeding 96% under optimal conditions. Moreover, the extraction time with CPE is significantly shorter (70 min), and there is no need for subsequent steps to remove residual

solvent from the final sample. This demonstrates that CPE can be an excellent green option for extracting oil from TAA in terms of yield and efficiency.

Table 1 - Experimental conditions and results of TAA extracts obtained with compressed propane.[@]

Run	Pressure (MPa)	T (°C)	Flow rate ($mL.min^{-1}$)	Density[#] (kg/m^3)	Extraction yield**(wt%)	Extraction efficiency[##] (%)
1	10	40	1	495.50	35.5	95.6
2	10	40	3	495.50	34.2	92.1
3*	10	60	2	467.20	35.4±0.3	95.3
4	10	80	1	435.25	35.9	96.5
5	10	80	3	435.25	35.6	95.9

[@]Experimental design (2^2).

*Center points with mean and standard deviation values from three extractions.

[#]propane density in the extraction condition, obtained from the NIST database.

**Yields calculated in dry basis (db).

[##] Mass of extract obtained with propane by mass of extract obtained with hexane in Soxhlet.

In the evaluated working range, temperature variation had a significant difference on the extraction yield of TAA ($p < 0.05$), as demonstrated by the Pareto chart (**Figure 1**). This diagram highlights the influence of the independent variables (flow rate and temperature) on the overall extraction yield. Conversely, the solvent flow rate did not significantly affect the overall extraction yield when using compressed propane. Differently, Freitas et al. [8] reported a significant difference in solvent flow rate when extracting oil from umari pulp with propane under the same conditions. This may be related to the different compositions of the oils, particularly in terms of fatty acid profile.

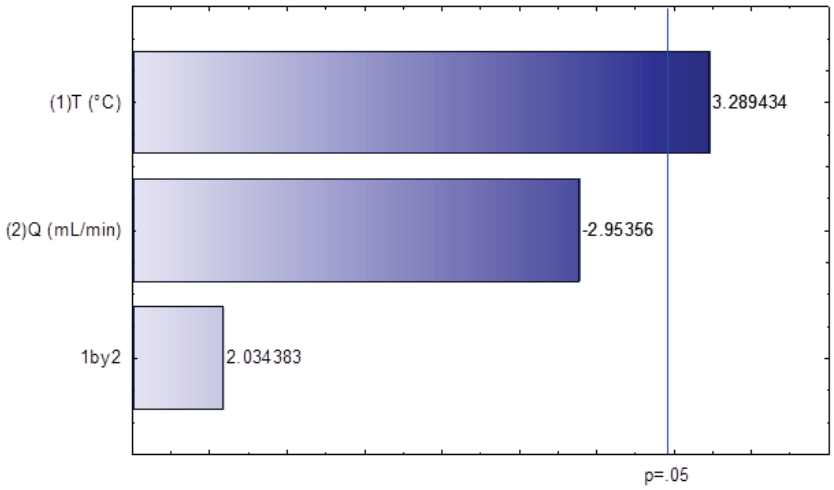

Figure 1 - Pareto chart showing the effects of process parameters on the extraction yield of TAA oil.

Figure 2 shows the overall extraction curves obtained using compressed propane as the solvent. Under the conditions of 80 °C with 3 mL/min and 40 °C with 3 mL/min, a constant extraction rate (CER) was observed up to approximately 10 min of extraction. This was due to the high availability of oil on the surface of the particles, allowing for the rapid and efficient extraction of this readily accessible solute. At this phase, mass transfer primarily occurs through convection [25]. Subsequently, between 10 and 20 min, a falling extraction rate (FER) was observed, where the solute layer surrounding the particles begins to develop flaws, initiating the diffusion of solute into the interior of the particles. At this stage, both convection and diffusion contribute to the mass transfer process. Finally, after 20 min of extraction, the curves reached their plateaus, showing an almost constant trend with minimal changes in yield, characterizing the diffusional constant rate (DCR). At this stage, solubility is controlled by internal diffusion mechanisms, with extraction primarily occurring through the diffusion of the solvent into the particle and the diffusion of the extracted solute to the particle's surface, as the readily available surface oil is depleted.

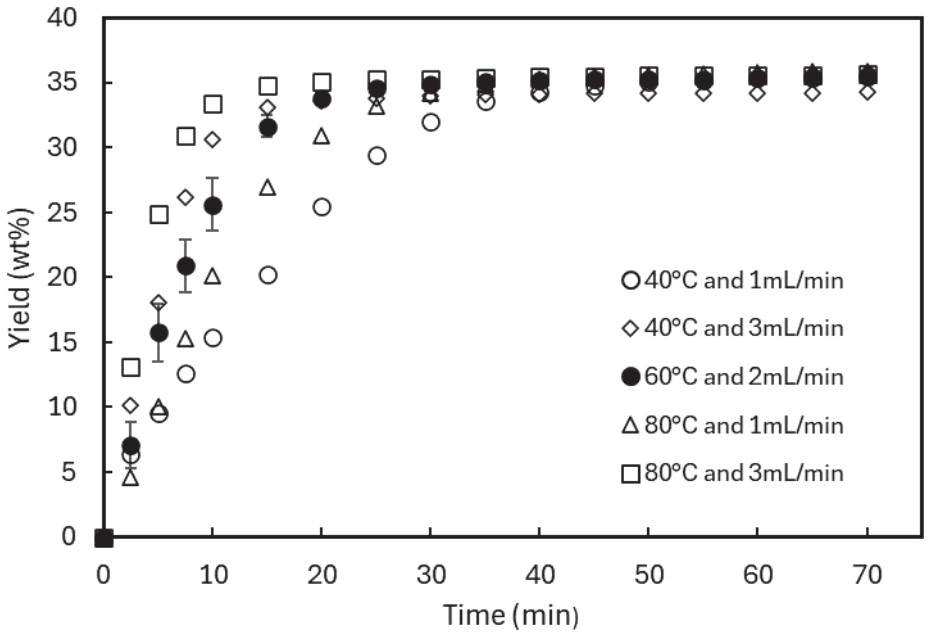

Figure 2 - Experimental overall extraction curves for TAA extraction using compressed propane as solvent.

In the curve for the intermediate condition (60 °C and 2 mL/min), the CER phase was observed up to 15 min of extraction, followed by the FER phase from 15 to 25 min, and finally, the DCR phase after 25 minutes of extraction. Regarding the curves for the conditions of 40 °C with 1 mL/min and 80 °C with 1 mL/min, the CER phase is observed from 0 to 30 min, followed by the FER phase until approximately 40 min, when the graphical plateaus are reached, characterizing the DCR phase. Therefore, given that extraction yield is an important parameter of interest, the condition of 80 °C and 1 mL/min is considered the optimal choice, as it achieved the highest extraction yield during the 70 min extraction time. However, extraction at 80 °C and 3 mL/min is also a good option, as it achieves similar results with only 15 min of extraction.

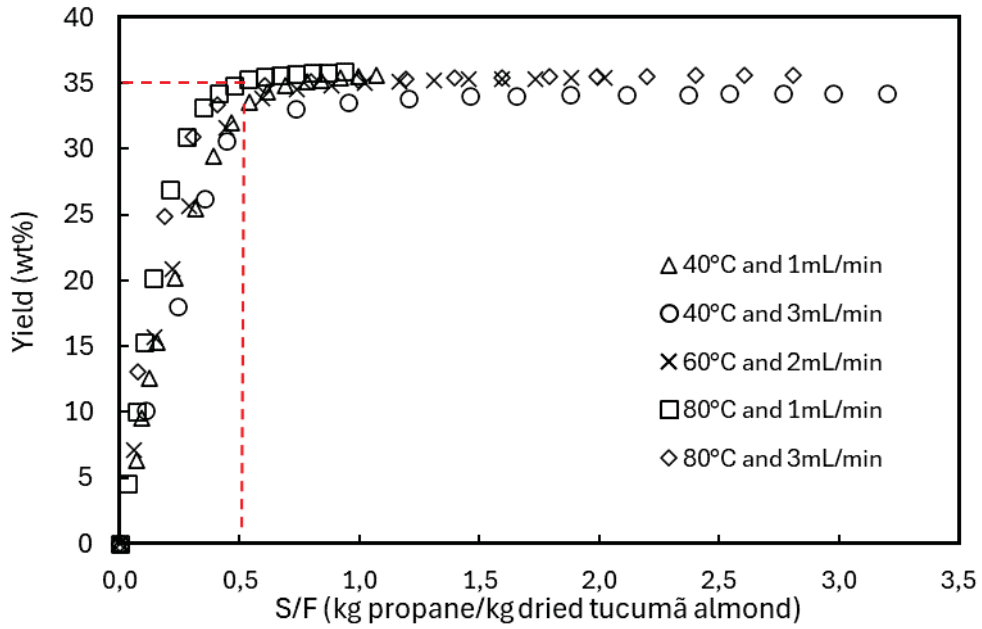

Figure 3 - TAA extraction curves using compressed propane at various temperatures and flow rates.

Figure 3 depicts the extraction yields as a function of the mass ratio between the solvent and feed (S/F). The results indicate that the S/F ratio of 0.5 kg propane per kg dried TAA (S/F=0.5) achieved nearly the maximum extraction yield, exceeding 35.0 wt% oil at higher temperatures conditions (80 °C with 1 mL/min and 80 °C with 3 mL/min). This supports the finding that temperature positively impacts extraction yield. This yield corresponds to more than 94% of the total oil content in TAA, based on the Soxhlet extraction yield (37.2 wt%) and assuming it fully recovers the lipid fraction from TAA. After an S/F ratio of 0.75 kg, there is an almost constant trend, with all extraction curves reaching their respective plateaus. Therefore, continuing the extraction process becomes less advantageous, as further oil recovery would be minimal and not cost-effective from both engineering and process standpoints. According to Goto et al. [26], when extraction curves follow a single trend line, it suggests that the process is predominantly dominated by the solubility of the easily accessible solute, with minimal impact from internal diffusion mechanisms. Similar graphic profiles were observed by Freitas et al. [8], Han et al. [27] and Jesus et al. [28], in agreement with the findings of this research.

OIL CHARACTERIZATION

Fatty acid composition of TAA oil

Table 2 shows the fatty acid composition results of TAA oil obtained by different extraction methods (Soxhlet and compressed propane). Eight fatty acids were identified (caprylic, capric, lauric, myristic, palmitic, stearic, oleic and linoleic), with lauric acid (49.54-52.77%) being the predominant, followed by myristic acid (24.57-26.91%). Carvalho et al. [3] found similar values (52.94 and 25.61% of lauric and myristic acids, respectively) for TAA oil using supercritical CO_2 extraction at 35 MPa and 40 °C. Similarly, Freitas et al. [29] found values of 53.14% for lauric acid and 22.59% for myristic acid when studying the production of biodiesel from TAA oil. The results show that TAA oil is composed of approximately 90% saturated fatty acids (SFA) and only 10% unsaturated fatty acids, of which oleic acid stands out with values ranging from 6.52 to 8.93%. It is worth noting that the fatty acid composition of TAA oil is very similar to that of coconut oil, which already has numerous industrial applications [30].

The lauric (LA) and myristic (MA) fatty acids found in the TAA oils are bioactive compounds present in many Amazonian natural products. LA and MA have several applications in the food, cosmetics and pharmaceutical sectors due to their unique properties. These fatty acids exhibit neuroprotective, anticancer, anti-inflammatory, and antibacterial pharmacological activities and are already used in the cosmetic industry [2]. LA has been shown to promote neuroprotection by alleviating infarct volume and cerebral edema in a murine model of stroke, reducing oxidative stress and increasing the antioxidant response, in addition to improved behavioral and functional outcomes in the animals [31]. Notably, LA treatment in human cancer cell lines suppressed the expression of cancer signaling pathways while increasing the expression of tumor suppressor miRNAs [32]. Furthermore, LA inhibited the formation of *Escherichia coli* bacterial persister cells, demonstrating that medium-chain saturated fatty acids can serve as antipersistent and antibiofilm agents that can be applied to treat bacterial infections [33]. The MA demonstrated anxiolytic effects similar to Diazepam (2mg/kg) in an animal model [34]. Furthermore, it was demonstrated that MA treatment induced significant antinociceptive and anti-inflammatory activity in acute and chronic inflammation in mice subjected to experimental tests [35].

In cosmetics, LA is valued for its emollient and antimicrobial properties and is commonly used in products such as soaps, creams, and shampoos. Its ability to form micellar structures enhances the cleansing and hydration of the skin and hair, while its antimicrobial properties help protect against infections and irritations [5]. Besides, LA and MA therapies have been routinely used in the treatment of acne vulgaris as dermocosmetics, inhibiting the growth of bacteria involved in this condition such as *Cutibacterium acnes, Staphylococcus aureus and Staphylococcus epidermidis*, in addition to preventing antibiotic resistance and side effects of other treatments [36,37]. In the food sector, LA is widely used in products such as chocolates and margarine, where it contributes to the texture and stability of foods, in addition to acting as a natural antimicrobial agent [5,7]. The versatility of LA and MA makes it an essential ingredient in formulations that aim for both functionality and product safety, providing a wide range of applications for high quality oils obtained with compressed propane.

Table 2 - Fatty acid composition (wt%) of TAA oil extracted by compressed propane and Soxhlet.

Fatty acids	propane (40 °C/10 MPa/ 1 mL min^{-1})	propane (40 °C/10 MPa/ 3 mL min^{-1})	propane[a] (60 °C/10 MPa/ 2 mL min^{-1})	propane (80 °C/10 MPa/ 1 mL min^{-1})	propane (80 °C/10 MPa/ 3 mL min^{-1})	Soxhlet (Hexane)
Caprylic (C8:0)	1.85	1.92	1.72	2.85	1.76	1.93
Capric (C10:0)	1.37	1.68	1.41	2.09	1.38	1.66
Lauric (C12:0)	51.98	51.38	52.77	49.54	51.30	51.30
Myristic (C14:0)	26.78	26.77	24.58	24.57	26.91	24.79
Palmitic (C16:0)	5.28	5.13	5.99	5.44	6.01	7.77
Stearic (C18:0)	3.12	2.35	2.84	4.31	2.95	2.77
Oleic (C18:1)	6.52	8.46	8.41	8.93	7.02	7.68
Linoleic (C18:2)	3.12	2.31	2.29	2.27	2.68	2.25
∑ SFA[1]	90.36	89.22	89.30	88.80	90.30	90.23
∑ MUFA[2]	6.52	8.46	8.41	8.93	7.02	7.68
∑ PUFA[3]	3.12	2.31	2.29	2.27	2.68	2.25
∑ PUFA + MUFA	9.64	10.78	10.70	11.20	9.70	9.93

The standard deviation for all fatty acids was lower than 0.8%.

[a] Center point for compressed propane extraction.

[1] Saturated fatty acids.

[2] Monounsaturated fatty acids.

[3] Polyunsaturated fatty acids.

β-Carotene content

Table 3 show β-carotene (BC) contents in oils obtained with CPE ranging from 6.21±0.41 to 8.52±0.82 mg $100g^{-1}$, with the highest values being obtained at 40 °C and 3 mL min^{-1}. On the other hand, the oil obtained by Soxhlet showed BC content of 0.32±0.1 mg $100g^{-1}$, a lower value than that obtained with CPE. This may have occurred due to the extended extraction time (6 h) used in the Soxhlet method and the additional solvent separation step, which was carried out in an oven at 60 °C for several hours. Since β-carotene is heat-sensitive, prolonged exposure of the extract to relatively high temperatures may have led to its degradation. This degradation is reflected in the difference in pigmentation of the final extracts, as illustrated in **Figure 4**. Mambrin & Barrera Arellano [38] also reported low BC values (0.3 mg $100g^{-1}$) for tucumã-do-Pará (*Astrocaryum vulgare*) almonds oil using conventional hexane extraction method. Silva et al. [39] found BC content of 193.4 mg $100g^{-1}$ in the oil obtained from tucumã-do-Amazonas pulp, indicating that TAA oil has a much lower BC content compared to the pulp, which is more suitable for BC extraction.

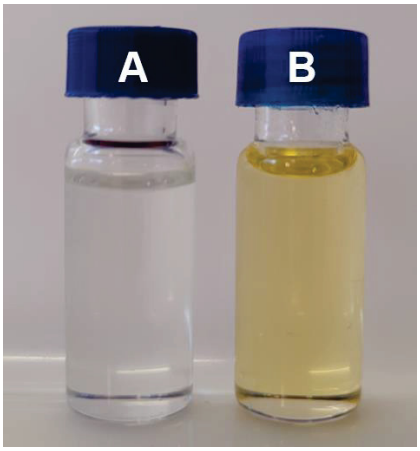

Figure 4 - TAA oils obtained with Soxhlet (A) and compressed propane (B).

Table 3 - β-Carotene, total phenolic contents, total flavonoids and antioxidant capacity for TAA oil obtained from compressed propane and Soxhlet extraction.

Run	Extraction conditions	Solvent	β-Carotene (mg/100 g oil)	TPC (mg GAE 100 g^{-1})	TF (mg CE 100 g^{-1})	DPPH (µmol TEAC 100 g^{-1})	ABTS (µmol TEAC 100g^{-1})
1	40 °C; 1 mL min^{-1}	Propane	8.01±0.22	n.d.	113.22±11.14	72.59±0.62	250.63±4.93
2	40 °C; 3 mL min^{-1}	Propane	8.52±0.82	n.d.	145.77±4.31	70.41±0.73	225.42±5.83
3*	60 °C; 2 mL min^{-1}	Propane	7.60±0.33	n.d.	130.10±8.57	74.20±0.31	257.63±6.64
4	80 °C; 1 mL min^{-1}	Propane	6.62±0.70	n.d.	142.14±7.81	73.47±0.33	234.16±2.64
5	80 °C; 3 mL min^{-1}	Propane	6.21±0.41	n.d.	130.34±11.36	72.03±0.48	239.66±10.47
6	Soxhlet; 68 °C	Hexane	0.32±0.01	1.07±0.06	260.66±4.49	74.64±0.19	245.28±1.52

* Triplicate at central point for compressed propane extraction.

n.d.: not detected

Total phenolic content (TPC) and total flavonoids (TF)

Table 3 presents the TPC and TF results for tucumã pulp oils extracted using compressed propane and the Soxhlet method. The TAA oils obtained by CPE did not show detectable TPC values with the method used. On the other hand, the oil obtained by the Soxhlet method showed a detectable TPC value, with a very low value of 1.07±0.06 mg GAE.100 g^{-1}. Regarding the TF results, the TAA oil exhibited values ranging from 113.22±11.14 to 145.77±4.31 mg CE.100 g^{-1}, with the highest concentration observed at 40 °C and 3 mL min^{-1}. The TAA oil obtained by Soxhlet presented a higher TF value (260.66±4.49 mg CE 100 g^{-1}) when compared to the oils obtained by CPE. This variation can be attributed to the selectivity of the extraction methods and the differences in the polarity of the solvents employed in the different techniques.

Antioxidant capacity

The results obtained for the two antioxidant assays (DPPH and ABTS) for oils extracted using compressed propane and Soxhlet methods are presented in **Table 3**. The DPPH radical scavenging capacity of the samples extracted with compressed propane ranged from 70.41±0.73 to 74.20±0.31 µmol TEAC 100 g^{-1}, with the highest value recorded at 60 °C and 2 mL min^{-1}. The oil extracted by the Soxhlet method showed a superior result (74.64±0.19 µmol TEAC 100 g^{-1}), which may be attributed to the solvent's selectivity and affinity for specific bioactive compounds. Likewise**,** for ABTS assays there was a variation from 225.42±5.83 to 257.63±6.64 µmol TEAC 100 g^{-1}, with the highest value observed at 60 °C and 2 mL min^{-1}, surpassing the result obtained by Soxhlet (245.28±1.52 µmol TEAC 100 g^{-1}).

Thermal behavior of TAA oil

Thermogravimetric analysis (TGA)

The thermogravimetric analysis produced curves showing mass loss (TGA) and the rate of mass loss (DTG) for TAA oil extracted with hexane and compressed propane. These curves are presented in **Figures 5A** and **5B**, respectively. Due to the nearly

identical fatty acid profiles, both solvents exhibited similar thermal behaviors, as detailed in **Table 4**, concerning the stages of mass decomposition.

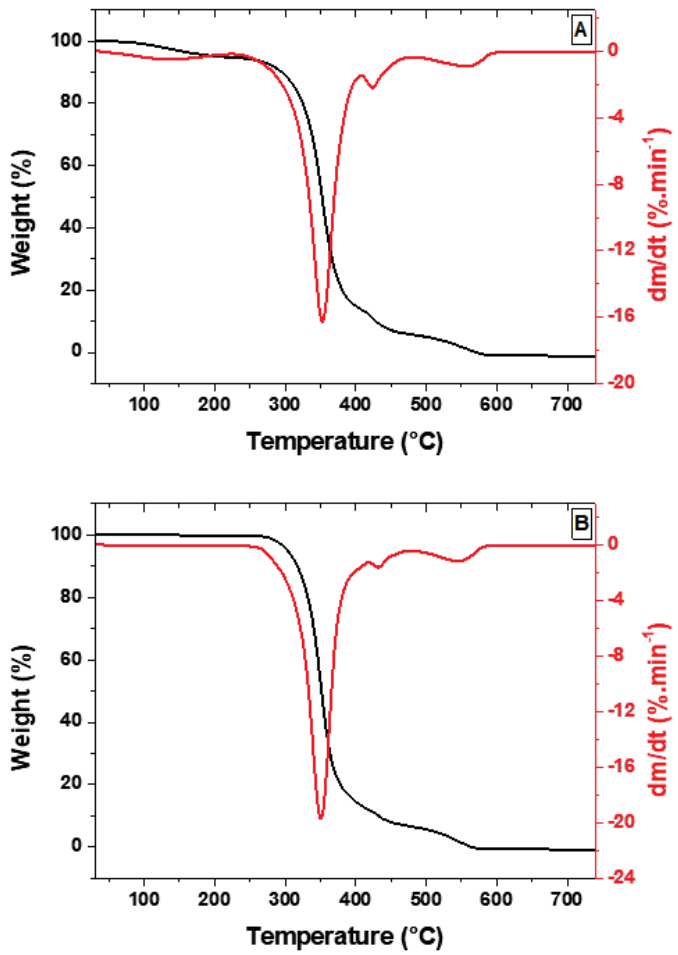

Figure 5 - TGA and DTG curves represented by the red and black lines, respectively, for TAA oil extracted using (A) hexane (Soxhlet) and (B) compressed propane (80 °C/10 MPa/3 mL min^{-1}).

Figures 5A and **5B** illustrate that mass degradation occurs in three distinct phases, as outlined in **Table 4**, each marked by a peak. The literature suggests that the first phase (I) is primarily associated with the thermal decomposition and initial breakdown of polyunsaturated fatty acid triglycerides. The following phases (II and III) involve the decomposition of monounsaturated and saturated fatty acids, and the polymerization of the by-products formed along the initial phase, generating peroxides and hydroperoxides [14,40,41].

Table 4 - TGA and DTG curve data for the extraction of TAA oil using hexane and compressed propane.

Solvent	Phase of thermal degradation	T_i (°C)	T_m (°C)	T_f (°C)	Δm (%)
Hexane	Stability	30.17	-	253.33	-
	I	253.33	352.67	407.31	89.24
	II	407.31	424.50	459.24	3.14
	III	475.67	557.00	594.30	6.31
Propane 80 °C/1 mL min^{-1}	Stability	30.17	-	258.83	-
	I	258.83	350.50	417.79	92.37
	II	417.79	432.84	452.81	1.20
	III	481.81	546.52	583.85	5.56

T_i, initial temperature; T_m, maximum mass loss temperature; T_f, final temperature; Δm, mass loss.

In this study, both samples begin the degradation phase at very similar temperatures (253.33 and 258.83°C), therefore it is not possible to state that one solvent provides greater thermal stability than another. Freitas et al. [8] reported similar degradation temperatures for umari pulp oil, using the same solvents (hexane and pressurized propane) under conditions of 10 MPa, 80 °C, and a flow rate of 3 mL min^{-1}. Barbi et al. [14] also presented the same thermal behavior profile in the TGA results for inajá pulp oil. In the inajá oil extraction using pressurized propane (at 10 MPa, 20 °C, and a flow rate of 2 mL min^{-1}) the mass degradation began at 269 °C, presenting three peaks (at 368 °C, 424 °C, and 562 °C).

Differential scanning calorimeter (DSC)

Differential Scanning Calorimetry (DSC) analysis generated the cooling curves (**Figure 6A**) and heating curves (**Figure 6B**) for TAA oil, extracted with hexane and compresses propane. Overall, the thermal profiles showed a notable similarity between the solvents, not only in the temperatures at which events occurred but also in their intensity. This resemblance was expected given the oils' nearly identical fatty acid compositions.

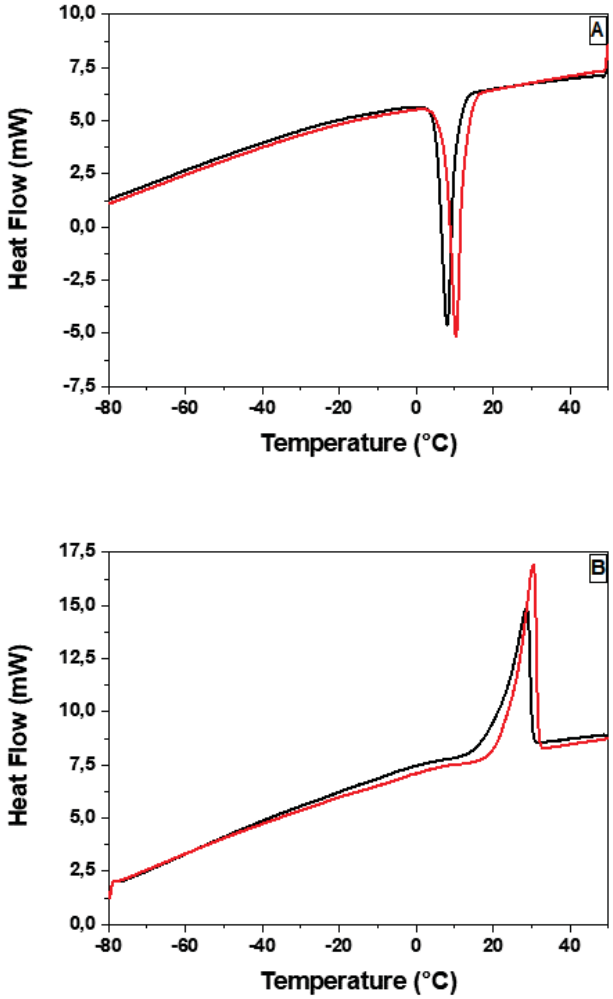

Figure 6. DSC curves of TAA oil extracted using hexane (Soxhlet), represented by black lines, and compressed propane (80 °C/10 MPa/1 mL min^{-1}), represented by red lines. (A) crystallization (50 to -80 °C) and (B) melting (-80 to 50 °C) behavior.

In both crystallization and melting events, a single well-defined peak was evident, with very minor differences between the two solvents used in the extractions. In **Figure 6A**, the exothermic crystallization peak occurs at approximately 10 °C for the extraction with compressed propane and at 8 °C for the extraction with hexane. The crystal formation is related to molecule rearrangement due to the presence of highly saturated triacylglycerols, then occurring molecule aggregation and compaction [42,43].

The endothermic peak related to the samples melting occurs at 28 °C and 30 °C when hexane and compressed propane are used as solvents in the extractions, respectively (**Figure 6B**). Both samples are completely liquid at 32 °C. In the study conducted by Carvalho et al. [3], which applied supercritical CO_2 for the extraction of TAA oil, a very similar thermal behavior was obtained, with crystallization occurring at approximately

11°C and melting at 32°C. Freitas et al. [8] and Fetzer et al. [15] applied DSC thermal analysis to umari pulp and cumaru seed oils, respectively (also extracted with hexane and compressed propane), and the samples in these studies completed their melting in approximately 19°C. In general, the higher the degree of unsaturation of fatty acids, the lower the melting point [44].

CONCLUSION

Compressed propane extraction has proven to be an efficient green alternative for obtaining TAA oils, providing excellent yields of high-quality oil. The extracted oils are rich in lauric and myristic fatty acids, with a profile very similar to coconut oil, and can therefore be applied in several industrial segments, such as the cosmetic, food and pharmaceutical industries. In addition, TAA oil showed good thermal stability and considerable values for antioxidant capacity and flavonoids. Thus, TAA, which is normally discarded as an agro-industrial byproduct, can be inserted into the Amazonian bioeconomic scenario, generating development and income for local communities.

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GENERAL CONCLUSIONS

This study brought to light relevant information about the current situation of the Amazon bioeconomy, highlighting its limitations and growth prospects. To develop the Amazon bioeconomy, it is necessary to combine science with traditional knowledge, involving the local population in the production chain that leads to the production of sustainable products with less environmental impact. Government investments and incentives are necessary to enhance the Amazon bioeconomy, as the region still faces several limitations that hinder the establishment of companies focused on the bioeconomy. Replacing conventional extraction processes with green, environmentally friendly processes seems to be the healthiest way to keep the forest standing and preserve the Amazon and its biodiversity. In this regard, the application of compressed propane extraction has proven to be a good alternative for obtaining oils rich in bioactive compounds, providing high efficiency and quality of the oils extracted from umari pulp, tucumã-do-Pará pulp, and tucumã-do-Amazonas almonds. Additionally, there is potential for applying this technique to several other oilseed species in the Amazon, adding value and positively impacting their respective production chains. Thus, the application of green technologies is a path to the Amazon bioeconomy development.

RECOMMENDATIONS FOR FUTURE STUDIES

Although the present study provides significant insights into the application of green technologies, especially compressed propane extraction (CPE), to obtain high-quality Amazonian oils, some questions remain open and deserve future investigation, as highlighted below:

- Expand the scope to include a wider range of Amazonian plant species, exploring their potential applications in the bioeconomy;
- More in-depth ethnobotanical studies could help identify new bioactive compounds, further enhancing Amazonian biodiversity. Collaboration with indigenous and traditional communities could reveal sustainable practices and knowledge that are not widely documented in the scientific literature;
- Apply supercritical CO_2 extraction to obtain umari oil, in order to compare it with extraction using compressed propane, providing a more comprehensive view of the efficiency and sustainability of these technologies;
- Apply the PLE (pressurized liquid extraction) method for the sequential extraction of the resulting defatted sample, in order to extract compounds with more polar profiles that were not extracted with propane;
- Evaluate the application of the shell surrounding the tucumã almond for the production of biochar;
- Application of oils rich in β-carotene and omega-9 for the production of oleogel, aiming at a healthier replacement for trans, saturated and interesterified fats in foods;
- Study the possible application of umari oil for the production of dermocosmetics;
- Study the scale-up of the processes reported in this study, evaluating their technical-economic aspects;
- Investigate the species studied in this research in terms of biorefinery;
- Conduct research focusing on the analysis of existing policies and the proposal of new governance models that promote environmental sustainability and regional economic development.

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