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RAFAEL GABRIEL DA ROSA

SUSTAINABLE BIOPROCESS COMBINING SUBCRITICAL WATER PRETREATMENT FOLLOWED BY ANAEROBIC DIGESTION FOR THE VALORIZATION OF JABUTICABA (*Myrciaria cauliflora*) AGRO-INDUSTRIAL BY-PRODUCT IN BIOENERGY AND BIOFERTILIZER

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Orientadora: Profa. Dra. Tânia Forster Carneiro
Co-orientador: Prof. Dr. Mauro Donizeti Berni

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RESUMO

No processamento industrial da jabuticaba a polpa é responsável pela maior participação de mercado, enquanto as cascas e sementes são subprodutos ou matérias-primas para outros processos de valorização. O objetivo geral deste trabalho foi desenvolver um bioprocesso integrando as tecnologias de hidrólise com água subcrítica e digestão anaeróbia para valorização de resíduos do processamento de jabuticaba, identificando possíveis novas rotas industriais para a produção de bioenergia, biogás contendo alta concentração de metano. Uma análise bibliométrica sobre a jabuticaba (*Myrciaria cauliflora*) nos últimos 21 anos foi realizada para identificar os trabalhos existentes no banco de dados web of science© e os resultados mostram que foram publicados 255 artigos e 5 revisões relacionadas a pesquisas sobre jabuticaba. A continuação realizou-se um experimento contendo reatores com hidrólise em água subcrítica (180 °C, 15 MPa, vazão de água de 10 mL min^{-1} e tempo cinético de 45 min) seguido de digestão anaeróbica (DA) a partir de cascas de jabuticaba. Os resultados indicam que a hidrólise como pré-tratamento aumentou o rendimento do metano (239,04 L CH_4 kg^{-1} TVS) em relação ao reator controle (42,31 L CH_4 kg^{-1} TVS). O metano produzido no reator com pré-tratamento de hidrólise foi capaz de gerar 543 kWh t^{-1} de eletricidade e 2.243,17 MJ t^{-1} de calor, valores superiores ao reator controle e podendo evitar um total de 177,54 kg de CO_2-eq t^{-1}. O digestato gerado após reatores de digestão anaeróbia se aplicado como biofertilizante na concentração de 0,3 g L^{-1} não apresenta efeitos tóxicos na germinação de *Lactuca sativa*. Finalmente, o bioprocesso sustentável projetado poderia ser uma alternativa ao manejo do subproduto da jabuticaba dentro de uma estrutura de economia circular, produzindo bioenergia e fertilizantes agrícolas que possam reduzir as emissões de gases de efeito estufa e a poluição ambiental na indústria alimentícia.

Palavras-chave: *Biomassa; Digestão anaeróbica; Água subcrítica; Biogás; Metano.*

ABSTRACT

In the industrial processing of jabuticaba, the pulp is responsible for the largest market share, while the peels and seeds are by-products or raw materials for other valorization processes. The general objective of this work was to develop a bioprocess integrating hydrolysis technologies with subcritical water and anaerobic digestion to value residues from the processing of jabuticaba, identifying possible new industrial routes for the production of bioenergy, biogas containing high methane concentration. A bibliometric analysis of jabuticaba (*Myrciaria cauliflora*) in the last 21 years was carried out to identify existing works in the Web of Science© database. The results show that 255 articles and 5 reviews related to research on jabuticaba were published. Next, an experiment was carried out with reactors with hydrolysis in subcritical water (180 °C, 15 MPa, water flow of 10 mL $min^{-1,}$ and kinetic time of 45 min) followed by anaerobic digestion (AD) agro-industrial byproducts of jabuticaba. The results indicate that hydrolysis as a pretreatment increased the methane yield (239.04 L CH_4 kg^{-1} TVS) in relation to the control reactor (42.31 L CH_4 kg^{-1} TVS). The methane produced in the reactor with hydrolysis pretreatment was able to generate 543 kWh t^{-1} of electricity and 2,243.17 MJ t^{-1} of heat, values higher than the control reactor and being able to avoid a total of 177.54 kg of CO_2-eq t^{-1}. The digestate generated after anaerobic digestion reactors, if applied as a biofertilizer at a concentration of 0.3 g L^{-1} did not present toxic effects on the germination of Lactuca sativa. Finally, the designed sustainable bioprocess could be an alternative to the management of the jabuticaba by-product within a circular economy framework, producing bioenergy and agricultural fertilizers that can reduce greenhouse gas emissions and environmental pollution in the food industry.

Keywords: *Biomass; Anaerobic digestion; Subcritical water; Biogas; Methane.*

SUMÁRIO

CAPÍTULO 1- Introdução geral, objetivos e estrutura da dissertação

1.1. Introdução Geral

Pertencendo à família *Myrtaceae*, a jabuticaba possui uma vida de prateleira muito reduzida, isso ocorre devido à alta quantidade de açúcar e água que a fruta possui, que muitas vezes inviabiliza o seu consumo *in natura*. (MARTINS et al., 2021). Consequentemente, o processamento industrial desta fruta é muito utilizado usada para a produção de alimentos processados como por exemplo: sucos, geleias, sorvetes, chás, licores, etc. (TARONE et al., 2021). Anualmente, o Brasil produz cerca de 5.000 toneladas de jabuticaba, esse dado refere-se ao comércio formal, não contabilizando a quantidade referente ao consumo doméstico ou informal (MASSA et al., 2020). A jabuticaba é conhecida por possuir grandes quantidades de compostos bioativos, como polifenóis (antocianinas, flavonoides e taninos), que possuem principalmente propriedades antioxidantes (PLAZA et al., 2016), além de aminoácidos (triptofano e lisina), vitaminas (WU et al., 2013), ácido ascórbico (RUFINO et al., 2011), magnésio e cobre (ALEZANDRO et al., 2013), sendo grande objeto de estudos da extração desses compostos.

A polpa da jabuticaba representa a maior parte na indústria de processamento da jabuticaba, já as cascas e as sementes são descartados sem nenhuma utilização (MENDES et al., 2021; FIDELIS et al., 2021). No processamento industrial da jabuticaba, somente 50% do seu peso é aproveitado, o restante, que consiste em cascas e sementes, muitas vezes são descartadas sem nenhuma utilização e nenhum tratamento adequado (MORALES et al., 2016). O descarte inadequado, sem nenhum controle ou tratamento desses subprodutos orgânicos, faz com que eles liberem muitos macronutrientes, como fósforo e nitrogênio, que são associados à eutrofização da água, emissões de gases de efeito estufa (GEE), entre vários efeitos prejudiciais ao meio ambiente (PELTZER et al., 2008).

As principais causas do aquecimento global e das alterações climáticas são as emissões dos gases de efeito estufa (GEE), uma grande parte das emissões dos GEE são a combustão de combustíveis fósseis produzidos a partir do petróleo (NARAN; TOOR; KIM, 2016). Em 2021, 36,3 Gt de dióxido de carbono (CO_2) foram liberados na atmosfera devido ao uso de energia fóssil. Os enormes incentivos fiscais e monetários mundiais dos governos pós-pandemia elevaram os níveis anuais a um recorde histórico, com um aumento de 2,1 Gt em comparação com 2020 (IEA, 2022). A estratégia estimula o interesse por combustíveis renováveis que possam ser utilizados em substituição às fontes tradicionais de energia, como os biocombustíveis obtidos a partir da biomassa (SILLERO; SOLERA; PEREZ, 2022).

Na indústria de processamento de jabuticaba (*Myrciaria cauliflora*), gera-se uma grande quantidade de resíduo, esta biomassa possui um alto potencial para a geração de energia. Embora muitas vezes descartados sem nenhum aproveitamento, os subprodutros das agroindústrias podem ser vistos como uma matéria-prima promissora, uma vez que possui uma abundância e isso facilita sua aplicação tecnológica para a produção de energia limpa e produtos com alto valor agregado (AWASTHI et al., 2020). No entanto, para ter um desenvolvimento sustentável, que é baseado na bioeconomia é necessário observar as políticas sanitárias, sociais e ambientais para o seu descarte correto (GIROTTO; ALIBARDI; COSSU, 2015). Portanto, a busca de novas e mais limpas fontes de geração de energia pelas industrias e pelos governos atuais, ganharam força devido aos aumentos na procura global por bioenergia na medida que as pessoas passam a se preocuparem com as grandes alterações climáticas (WANG et al., 2020).

Para a geração de bioenergia atráves da biomassa, pode ser utilizada a tecnologia de digestão anaeróbica (DA). O processo de DA ocorre por meio de diversas transformações químicas que convertem a matéria orgânica em biogás através das fases de hidrólise, acidogênese, acetogênese e metanogênese, estas etapas são realizadas por vários microrganismos (AMPESE et al., 2022). Após o processo de DA, é gerado um digestato que possui uma grande concentração de componentes minerais e orgânicos que pode ser utilizado como biofertilizante do solo em substituição aos minerais fósseis (SILLERO; SOLERA; PEREZ, 2022; VENEGAS et al., 2021). O biogás rico em metano produzido pela DA pode ser utilizado em substituição ao combustível a base de petróleo, como em carros e na cozinha, podendo ser utilizado para a combustão para gerar eletricidade e calor (SCARLAT; DALLEMAND; FAHL, 2018).

A etapa de hidrólise em processos de digestão anaeróbia pode ser uma etapa limitante nos casos de biomassa lignocelulósica, uma vez que a estrutura polimérica do material dificulta a conversão em açúcares mais biodegradáveis (PINTO et al., 2018). Portanto, a biodegradação da biomassa lignocelulósica (por exemplo, subproduto de jabuticaba) em reatores anaeróbios necessita de um pré-tratamento prévio para facilitar a oxidação por parte das bactérias, fornecendo açúcares mais fermentáveis com cadeia mais curta (SUAREZ et al., 2022).

O pré-tratamento com a utilização de água subcrítica da biomassa, para a utilização subsequente na DA, tem ganhado notoriedade devido ao alto rendimento do biogás produzido a partir de resíduos lignocelulósicos, principalmente quando comparado aos procedimentos

convencionais sem nenhum pré-tratamento (CHEN et al., 2022). O pré-tratamento hidrotérmico permite menor geração de contaminantes sem insumos químicos e produtos e resíduos inibitórios reduzidos ou quase inexistentes (WELLS et al., 2020). Para a despolimerização de diferentes componentes da biomassa lignoceluolósica, à água subcrítica tem sido muito utilizada (COCERO et al., 2018). Para se obter a água no seu estado subcrítico é necessário que os parâmetros pressão e temperatura ultrapassem o ponto de ebulição da água (100 °C, 0.1 MPa) e se encontrem inferiores ao ponto crítico (374 °C, 22 MPa) (SGANZERLA et al., 2022).

A análise bibliométrica é uma ferramenta que permite analisar os trabalhos existentes na literatura. Esta análise utiliza modelos matemáticos combinado com métodos estatísticos para avaliar o conteúdo de áreas específicas da literatura, tais estudos permitem verificar os avanços do conhecimento em determinados campos e, consequentemente, identificar áreas de estudo pouco exploradas (JIANG et al., 2019). A análise bibliométrica é muito utilizada para ampliar as visões nos mais variados campos de pesquisa. A bibliometria ajuda a identificar as lacunas do conhecimento, auxiliando no planejamento de estudos futuros para melhor entender a área de pesquisa estudada. No entanto, não foi relatada na literatura uma análise bibliométrica para orientar futuras pesquisas sobre a jabuticaba, também não formam encontrados estudos que utilizam a tecnologia de água subcrítica como pré-tratamento de subproduto agroindustrial de jabuticaba com subsequente digestão anaeróbia.

A finalidade deste trabalho foi verificar se os resíduos do processamento de jabuticaba quando submetidos ao pré-tratamento em água subcrítica previamente a digestão anaeróbia, apresentam maior geração de biogás e metano quando comparados com reatores controles sem pré-tratamento. A integração das tecnologias supercrítica e de digestão anaeróbia pode contribuir para uma disposição final adequada do resíduo ao meio ambiente e pode contribuir para a transição para uma economia circular melhorando o desenvolvimento local e regional.

1.2. Objetivos

1.2.1. Objetivo Geral

O objetivo geral deste trabalho foi desenvolver um bioprocesso integrando as tecnologias de hidrólise com água subcrítica e digestão anaeróbia para valorização de resíduos do processamento de jabuticaba, identificando possíveis novas rotas industriais para a produção de bioenergia: biogás contendo alta concentração de metano.

1.2.2. Objetivos específicos

- ✓ Realizar análise bibliométrica dos estudos dos últimos 21 anos para verificar as áreas de estudos pouco exploradas utilizando a jabuticaba e analisar as possibilidades para a sua utilização.

- ✓ Realizar ensaios de hidrólise em água subcrítica, usando os parâmetros operacionais selecionados (temperatura de 180 ºC, vazão 10 mL min^{-1} e pressão de 15 MPa, tempo cinético de 45 min) para pré-tratamento do subproduto agroindustrial da jabuticaba;

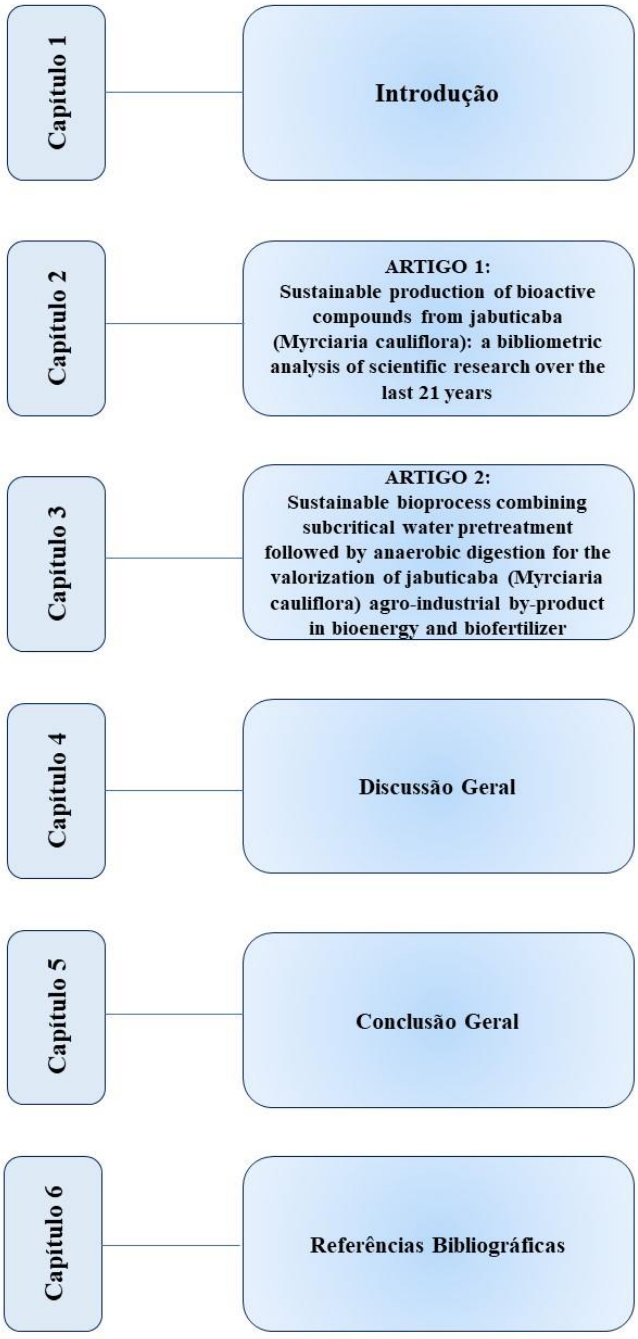
- ✓ Realizar ensaios com reatores anaeróbios em regime semi-contínuo, temperatura mesofílica (36,5ºC) e tipo seco (superior a 15% de sólidos), utilizando o hidrolisado como pré-tratamento e reatores controle a partir de cascas de jabuticaba sem pré-tratamento;

- ✓ Analisar o rendimento de metano com reatores anaeróbios, com pré-tratamento e sem, a partir de cascas de jabuticaba e analisar o potencial de energia elétrica e térmica e ambiental (emissões de gases de efeito estufa evitadas).

1.3. Estrutura da Dissertação

A presente dissertação encontra-se dividida em 6 capítulos. Os artigos apresentados, correspondem a artigos que foram publicados em revistas científicas internacionais. A estrutura da dissertação pode ser observada na **Figura 1.**

Figura 1. Fluxograma da estrutura da dissertação.



O **primeiro capítulo** contém a introdução, a qual consiste em apresentar as informações mais relevantes em relação ao tema proposto para a dissertação, realizando uma caracterização do problema, além de apresentar a finalidade, hipótese e os objetivos do trabalho.

O **segundo capítulo** apresenta o trabalho *"Sustainable production of bioactive compounds from jabuticaba (Myrciaria cauliflora): a bibliometric analysis of scientific research over the last 21 years"*. Este trabalho, contém uma análise bibliométrica, onde são apresentados os principais estudos utilizando a jabuticaba nos últimos 21 anos. A análise bibliométrica, visa elucidar as pesquisas envolvendo a jabuticaba, bem como os principais estudos realizados, para preencher as lacunas de conhecimento através de estudos futuros envolvendo a jabuticaba.

O **terceiro capítulo** traz o artigo *"Sustainable bioprocess combining subcritical water pretreatment followed by semi-continuous anaerobic digestion for the valorization of jabuticaba (Myrciaria cauliflora) agro-industrial by-product"*. Este trabalho, teve como o objetivo demonstrar o efeito de uma combinação do pré-tratamento dos subprodutos agroindustriais da jabuticaba com água subcrítica, seguido de digestão anaeróbica para a recuperação de bioenergia e fertilizantes agrícolas.

O **quarto capítulo** apresenta as discussões gerais da dissertação.

O **quinto capítulo** apresenta as principais conclusões da dissertação bem como as sugestões para trabalhos futuros.

O **sexto capitulo** contém as referências bibliográficas utilizadas para a realização desta dissertação.

CAPÍTULO 2 - Sustainable production of bioactive compounds from jabuticaba (*Myrciaria cauliflora*): a bibliometric analysis of scientific research over the last 21 years

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Sustainable production of bioactive compounds from jabuticaba (*Myrciaria cauliflora*): a bibliometric analysis of scientific research over the last 21 years

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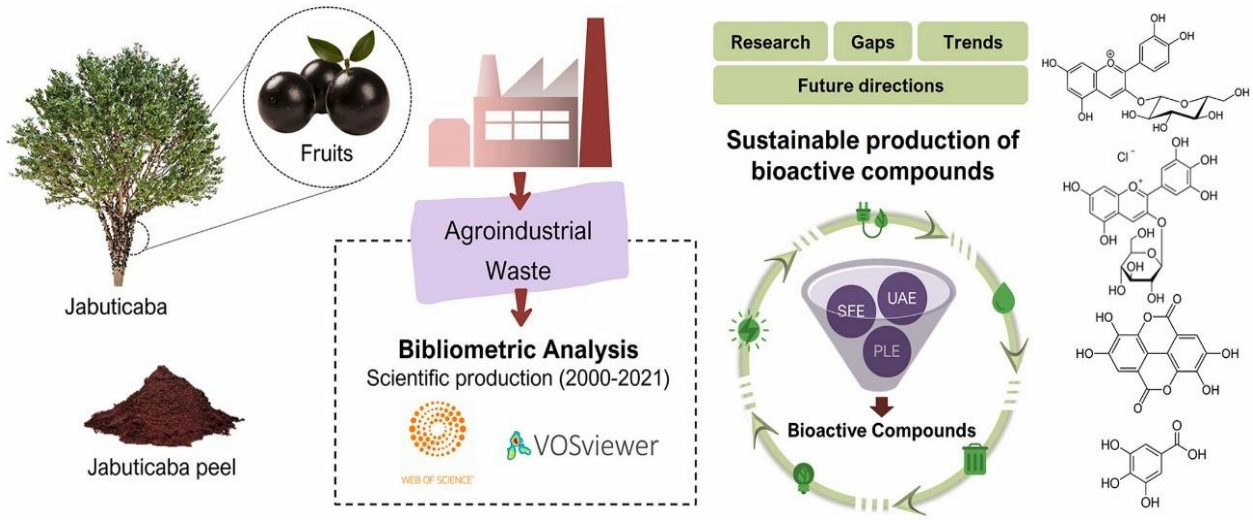
Graphical abstract



Abstract

The demand for pharmaceutical products based on bioactive compounds obtained from by-products and manufactured by sustainable technological routes is a worldwide demand to the circular economy transition. In this review, a bibliometric analysis on jabuticaba (*Myrciaria cauliflora*) research over the last 21 years was conducted to identify prospects for the sustainable production of bioactive compounds from its by-products. The Web of Science© database was used and the dataset was analyzed on VosViewer© bibliometric software's to perform maps based on the main keywords. In total, 255 articles and 5 reviews were published related to research on jabuticaba. The bibliometric analysis elucidated that *Food Science Technology* was the main research area, and the most frequent keywords on the field were associated with anthocyanins, demonstrating that there is a trend in determine the optimun extraction method of this bioactive compound from jabuticaba. Fourthmore, this review provides direction for the production of bioactive compounds (gallic acid, cyanidin, malvidin, peonidine, petunidine, pelargonidine, and many others) in different sustainable technological routes (ultrasound-assisted extraction, supercritical carbon dioxide extraction, and pressurized liquid extraction) using jabuticaba's by-products as feedstock.

Keywords*: Green chemistry; By-products valorization; Pharmaceutical products; value-added products; Circular economy; Biorefinery; Scientometrics.*

1. Introduction

Fruit consumption is encouraged and essential due to the richness of nutrients and bioactive compounds, which are vital in preventing chronic diseases and beneficial to human health (Schmidt et al., 2020). Brazil has in its territory the most significant flora diversity globally, with more than thirty thousand different species of angiosperms endorsed with flowers and fruits (Dutra et al., 2015). Among the diversity in Brazilian territory, the Myrtaceae family has ecological and economic prominence as the eighth largest family of flowering plants (Donado-Pestana et al., 2018; Schmidt et al., 2020). Its specimens are found in Australia, southeast Asia, and South America, with about 130 – 150 genera and more than 5800 species (Frauches et al., 2016; Schmidt et al., 2020). The *Myrtaceae* family includes guava (*Psidium* spp.), clove (*Syzygium aromaticum*), allspice (*Pimenta dioica*), cambuci (*Campomanesia phaea* Berg.), camu-camu (*Myrciaria dubia* McVaugh), jabuticaba (*Myrciaria cauliflora*), and many other fruits with high agro-industrial potential (Donado-Pestana et al., 2018).

Figure 1 shows the jabuticaba tree and the fruit in their development stages. Jabuticaba, belonging to the *Myrtaceae* family, is a very perishable fruit due to the high amount of sugar and water, with a low shelf life for *in natura* consumption (Martins et al., 2021). Therefore, this fruit is commonly used in derivatives processed products (e.g., juices, jellies, ice cream, tea, liqueurs, and others) to achieve a more integral use of the fruit and avoid waste (Tarone et al., 2021). Brazil has an annual production of 5000 tons of jabuticaba only within formal trade, not including the amount related to domestic or informal consumption (Massa et al., 2020).

Figure 1. Illustration of (a) jabuticaba tree, (b) fruits, and (c) different stages of fruit ripening of jabuticaba.

Jabuticaba presents a high amount of bioactive compounds, especially polyphenols (anthocyanins, flavonoids, and tannins), which have antioxidant activity (Plaza et al., 2016), as well as amino acids (tryptophan and lysine), vitamins (Wu et al., 2013), ascorbic acid (Rufino et al., 2011), magnesium, and copper (Alezandro et al., 2013). The consumption of jabuticaba can reduce serum triglycerides, improve insulin sensitivity, and anti-inflammatory action, promoting its consumption as beneficial to human health (Inada et al., 2020b; Lamas et al., 2018).

In the jabuticaba's industrial processing, the pulp is responsible for the highest market share, while peels and seeds are by-products (Mendes et al., 2021; Fidelis et al., 2021).

Approximately 50% of the weight of the jabuticaba fruit consists of peels and seeds, which are discarded without any recovery or utilization (Morales et al., 2016). When those organic by-products are dumped into the environment without treatment or disposal control, they release many macronutrients, such as phosphorus and nitrogen, which have been associated with water eutrophication, greenhouse gas (GHG) emissions, among other environmental side-effects (Peltzer et al., 2008).

Currently, landfilling is the leading destination of organic waste despite to risk of environmental pollution (Zhang et al., 2018). Other disposal routes to reduce this practice focusing on energy recovery should be employed (Chen et al., 2017), as well as green technologies to recover value-added compounds (Benvenutti et al., 2021; Oldfield et al., 2016; Peltzer et al., 2008). The compounds extracted from solid and food waste are proteins, fibers, vitamins, minerals, lipids, phenolic compounds, antioxidants, and phytomoniums (Coelho et al., 2001). The recovery, recycling, and reuse of organic waste are widely highlighted in the literature, especially to produce compounds of immediate biological interest, such as anthocyanins, which are abundant in jabuticaba peels (Ferreira et al., 2020).

Several technological routes for generating new value-added products from solid waste are possible (Ortiz et al., 2020). From identifying the industrial most economically attractive opportunities (Dantas et al., 2013), process and cost optimization can be designed (Hannan et al., 2020). It is necessary to evaluate the waste characterization to study the possibilities of applying the various technologies and knowledge of their industrial and commercial flows to manage them, preferably punctually and locally (Pimentel et al., 2020). Biotechnological advances, driven by environmental and economic benefits, provide notoriety to lignocellulosic wastes (Bilal et al., 2017). The possible technologies of valuing waste from the food industry are essential to support action plans and decision-making to avoid the loss of their biological

potential and produce bio-based products, a clean and desirable strategy to improve industry sustainability.

Notwithstanding, the use of bibliometric analysis has been a tool widely used to disseminate the prospects in diverse research fields. For instance, in the areas of bio-waste (Obileke et al., 2020), energy recovery from macaúba husk (Ampese et al., 2021), valorization of orange industrial waste (Jiménez-Castro et al., 2020), biorefinery for the circular economy transition of the beer industry (Sganzerla et al., 2021), anaerobic digestion (Ampese et al., 2022b), biodiesel production (Rajeswari et al., 2021), and enzyme immobilization (Almeida et al., 2021), bibliometrics identified the knowledge gaps and elucidated the establishment of future studies to better understand the state of the art in the research field. However, a bibliometric analysis to guide future directions on the research of jabuticaba was not reported in the literature.

Based on the mentioned above, this study accomplished a bibliometric review on jabuticaba (*Myrciaria cauliflora*) research over the last 21 years, aiming to identify prospects for the sustainable production of bioactive compounds. It also intends to identify green technological routes to generate new bio-products from jabuticaba's by-products. This study will identify the most attractive opportunities for the industrial production of bioactive compounds from jabuticaba by-products, identifying the possible knowledge gaps and research opportunities for the jabuticaba production chain.

2. Methodology for literature search and bibliometric analysis

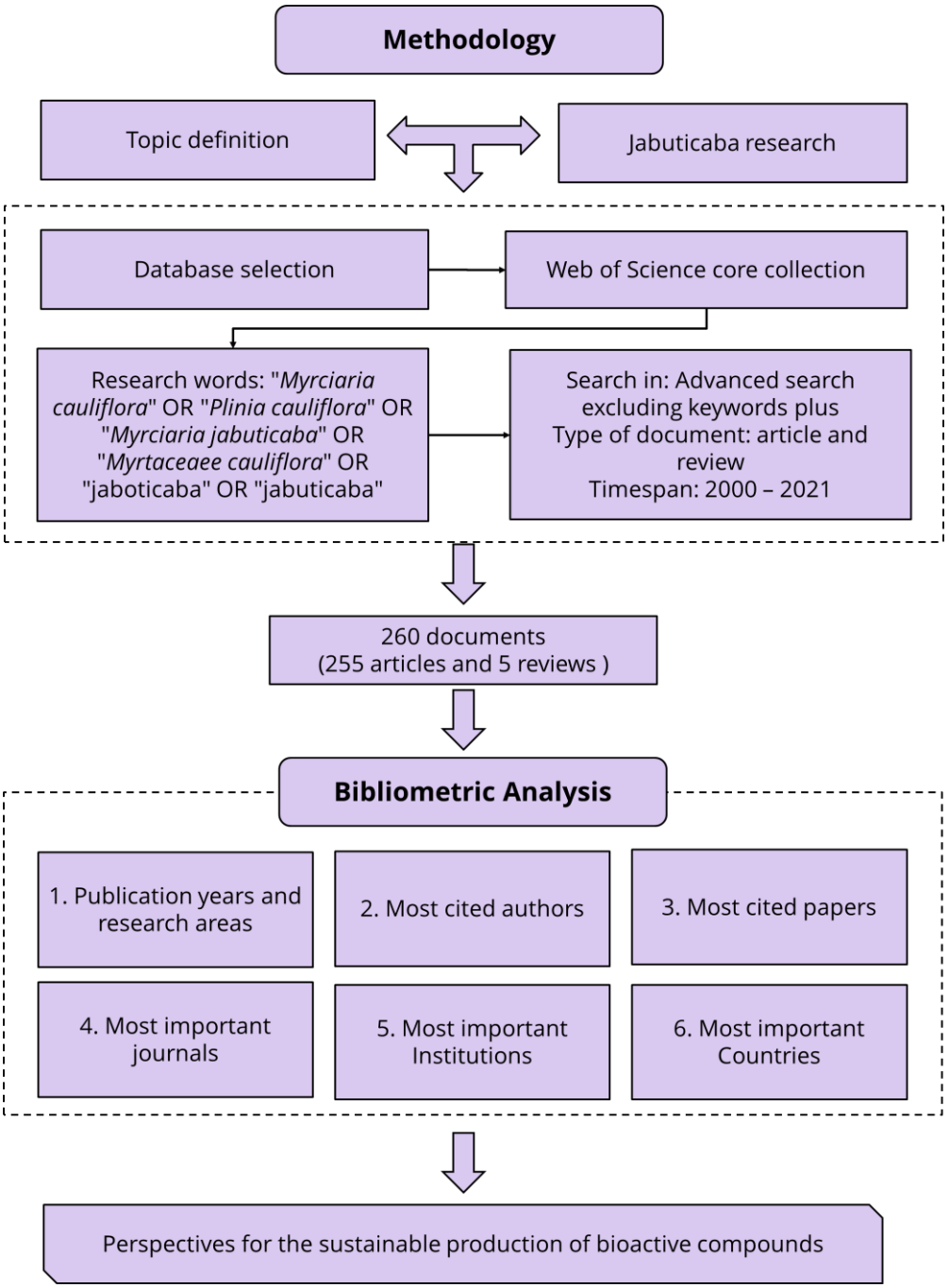
The systematic literature search was carried out by selecting documents to demonstrate how the research has been developed and changed over the years. Based on the number of publications, it is possible to hypothesize the trends and technical decisions on the field. This study retrieve the publications related to practical applications of jabuticaba and its by-products. Hence, based on the documents selected in the literature search, new technological routes can be proposed for the sustainable extraction of bioactive compounds.

For the literature search and bibliometric study, the data collection was based on scientific publications indexed in the Science Citation Index Expanded (SCI-E) – Clarivate Analytics' ISI – Web of Science© (WoS) database. The research in WoS core collection was conducted in the section “advanced search”, to obtain reliable and accurate data in the field. The WoS search was performed applying the following logic operation: TS=("*Myrciaria cauliflora*" OR "*Plinia cauliflora*" OR "*Myrciaria jabuticaba*" OR "*Myrtaceaee cauliflora*" OR "jaboticaba" OR "jabuticaba") NOT KP=("*Myrciaria cauliflora*" OR "*Plinia cauliflora*" OR "*Myrciaria jabuticaba*" OR "*Myrtaceae cauliflora*" OR "jaboticaba" OR "jabuticaba"). Through this search query, it was possible to filter the publications based on selected words included in the title, abstract, author’s keywords, and not in the keywords-plus of each document, avoiding mismatches with words that only appeared on references and are not the subject of the studies. The research was done for the period from 2000 to 2021, to cover the last 21 years of publication. **Figure 2** presents the methodological steps for the literature search and bibliometric study.

Furthermore, a filter based on the document type was used, where “article” and “review” were selected. The dataset obtained was exported and analyzed on VOsViewer bibliometric software’s. Maps based on the main keywords and connections between them were performed in the VOsViewer software (van Eck and Waltman, 2010). Lastly, the overlay visualization was

chosen to identify the documents that have received more attention from the scientific community and visualize the most important articles published.

Figure 2. Methodological steps for the bibliometric analysis.



3. Research trends on jabuticaba

3.1 Publication evolution and research areas

The evolution of the research related to jabuticaba over the last 20 years can be observed in **Figure 3**. From the literature search, two hundred sixty (260) documents (255 articles and 5 reviews) were recorded in the WoS database. According to the overall development trend, the relevant research progress can be divided into three stages. From 2000 until 2010, the number of publications was lower than 32. Afterward, from 2010 to 2017, an average of 14.5 documents was published per year. Finally, an exponential trend can be observed from 2018, reaching 38 documents published in 2020. In 2021, the number of publications recorded in WoS database decreased to 29. Articles involving jabuticaba research were ranked in the 10 top publication areas, affiliations, countries, and journals (**Table 1**). Food Science Technology (43.01%), Chemistry (22.30%), and Agriculture (21.15%) were the most predominant research field. Therefore, the documents published during the last several years demonstrate the speed and progress of research in Food Science Technology, reflecting the relevant studies fields.

Figure 3. Evolution of the publications and research fields on the use of jabuticaba.

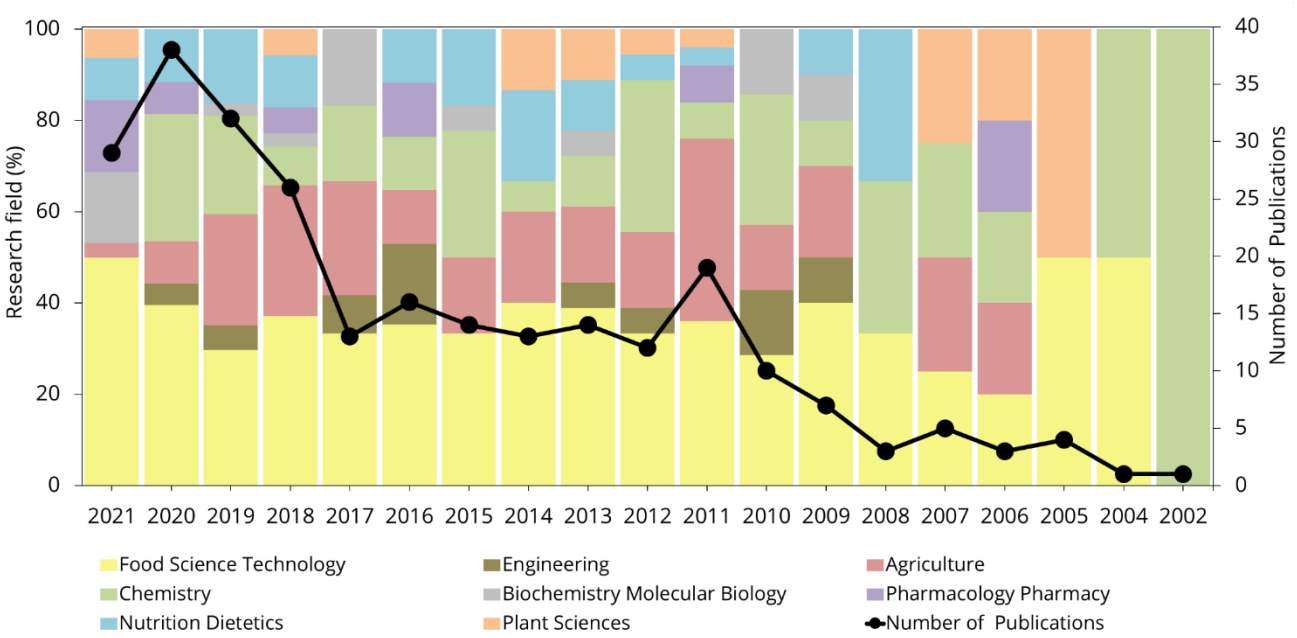


Table 1. Ranking of the 10 top publication areas, affiliations, countries, and journals.

Ranking	Research areas	Number	%[1]
1st	Food Science Technology	112	43.07
2nd	Chemistry	58	22.30
3rd	Agriculture	55	21.15
4th	Nutrition Dietetics	34	13.07
5th	Pharmacology Pharmacy	19	7.30
6th	Plant Sciences	18	6.92
7th	Biochemistry Molecular Biology	16	6.15
8th	Engineering	12	4.61
9th	Science Technology (other topics)	8	3.07
10th	Materials Science	7	2.69
Ranking	Affiliations (Country)	Number	%[1]
1st	University of São Paulo, USP (Brazil)	42	16.15
2nd	University of Campinas, UNICAMP (Brazil)	40	15.38
3rd	Federal University of Goias, UFG (Brazil)	22	8.42
4th	Federal Technological University of Parana, UTFPR (Brazil)	18	6.92
5th	Federal University of Lavras, UFLA (Brazil)	17	6.53
6th	Brazilian Agricultural Research Corporation, EMBRAPA (Brazil)	16	6.15
7th	São Paulo State University, UNESP (Brazil)	14	5.38
8th	Federal University of Viçosa, UFV (Brazil)	13	5.01
9th	Federal University of Santa Catarina, UFSC (Brazil)	11	4.23
10th	Federal University of Santa Maria, UFSM (Brazil)	11	4.23
Ranking	Countries	Number	%[1]
1st	Brazil	236	90.76
2nd	United Stated of America	15	5.76
3rd	Portugal	7	2.69
4th	Spain	7	2.69
5th	China	6	2.30
6th	Canada	5	1.92
7th	Taiwan	4	1.53
8th	Colombia	3	1.15
9th	Finland	3	1.15
10th	France	3	1.15
Ranking	Journals	Number	%[1]
1st	Food Research International	16	6.15
2nd	*Revista Brasileira de Fruticultura**	16	6.15
3rd	Food Chemistry	12	4.61
4th	Journal of Functional Foods	7	2.69
5th	Molecules	7	2.69
6th	Food Science and Technology	6	2.30
7th	Journal of Agricultural and Food Chemistry	6	2.30
8th	LWT – Food Science and Technology	6	2.30
9th	Acta Scientiarum Agronomy	5	1.92
10th	Journal of Food Engineering	5	1.92

[1] Percentage of 260 documents (automatically calculated in WoS). Data reatrived on WoS at February 02nd 2022. *In Portuguese.

3.2 Study of main keywords in the field

The literature suggests that the study of keywords is one of the most important aspects of a bibliometric study (Ampese et al., 2022b; Sganzerla et al., 2021). This analysis plays a crucial and facilitating role in information and co-word examination, acting as a filter in file searches (Melo et al., 2021; Rodríguez-Rojas et al., 2019). Therefore, **Figure 4** presents the 44 most frequent authors' keywords in the field, associated with a minimum number of occurrences of 3 keywords.

The map-based on different clusters indicates that the size of the circles is directly proportional to the keyword total link strength. In contrast, the distance between two of the terms suggests if they are closely related to each other or not. From the bibliometric coupling, 711 keywords were obtained from the search. **Table 2** shows the top 20 keywords on the field. The most frequent keywords were "jaboticaba" (33 ocurrences), "anthocyanins" (30 occurrences), "jabuticaba" (29 occurrences), and "phenolic compounds" (26 occurrences), indicating that these words are central and co-occurs with numerous others, as noted in the number of links (lines) in **Figure 4a**. Moreover, there are some variations in the therminology "jabuticaba" and "jaboticaba" to express the name of the fruit, and this fact also occurs in the scientific name of the species (*Myrciaria cauliflora*, *Myrciaria jabuticaba, Plinia cauliflora,* and *Plinia trunciflora*).

Table 2. The top 20 keywords on the field (rank based on total link strength).

Ranking	Keyword	Occurrences	Total link strength
1	Jaboticaba	33	49
2	Anthocyanins	30	46
3	Jabuticaba	26	39
4	Phenolic compounds	23	37
5	*Myrciaria cauliflora*	20	23
6	Obesity	12	23
7	Bioactive compounds	11	18
8	Polyphenols	10	18
9	Antioxidant activity	10	17
10	Ellagic acid	7	15
11	*Myrciaria jabuticaba*	11	13
12	Myrtaceae	20	13
13	*Plinia cauliflora*	10	13
14	Antioxidant	8	12
15	Flavonoids	6	12
16	Oxidative stress	5	12
17	Antioxidant capacity	7	11
18	*Plinia trunciflora*	4	11
19	Anthocyanin	6	8
20	Antimicrobial	3	8

Table 3 shows the clusters based on the keyword's analysis. It is possible to identify three clusters coupled with the most important keywords associated with the biological properties of bioactive compounds from jabuticaba. Also, the most critical keywords revealed that *in vivo* studies dealt with the effectiveness of jabuticaba for human health. Therefore, the keywords "antioxidants", "ellagic acid", "ellagitannins", "flavonoids", "antimicrobial", "insulin resistance", "lipid profile", "obesity", and "oxidative stress" demonstrate that jabuticaba can be a promising feedstock to extract bioactive compounds for technological applications (Inada et al., 2020b; Quatrin et al., 2020). The keywords analysis revealed that the phytotherapeutic properties of jabuticaba are relevant for its potential use in the food industry since most keywords are associated with antioxidant, antimicrobial, and anti-inflammatory activity (Inada et al., 2020b, 2020a). These properties are represented by chemical groups such

as phenols, flavonoids, and others, which generally benefit human health (do Nascimento et al., 2020; Fidelis et al., 2021; Massa et al., 2020).

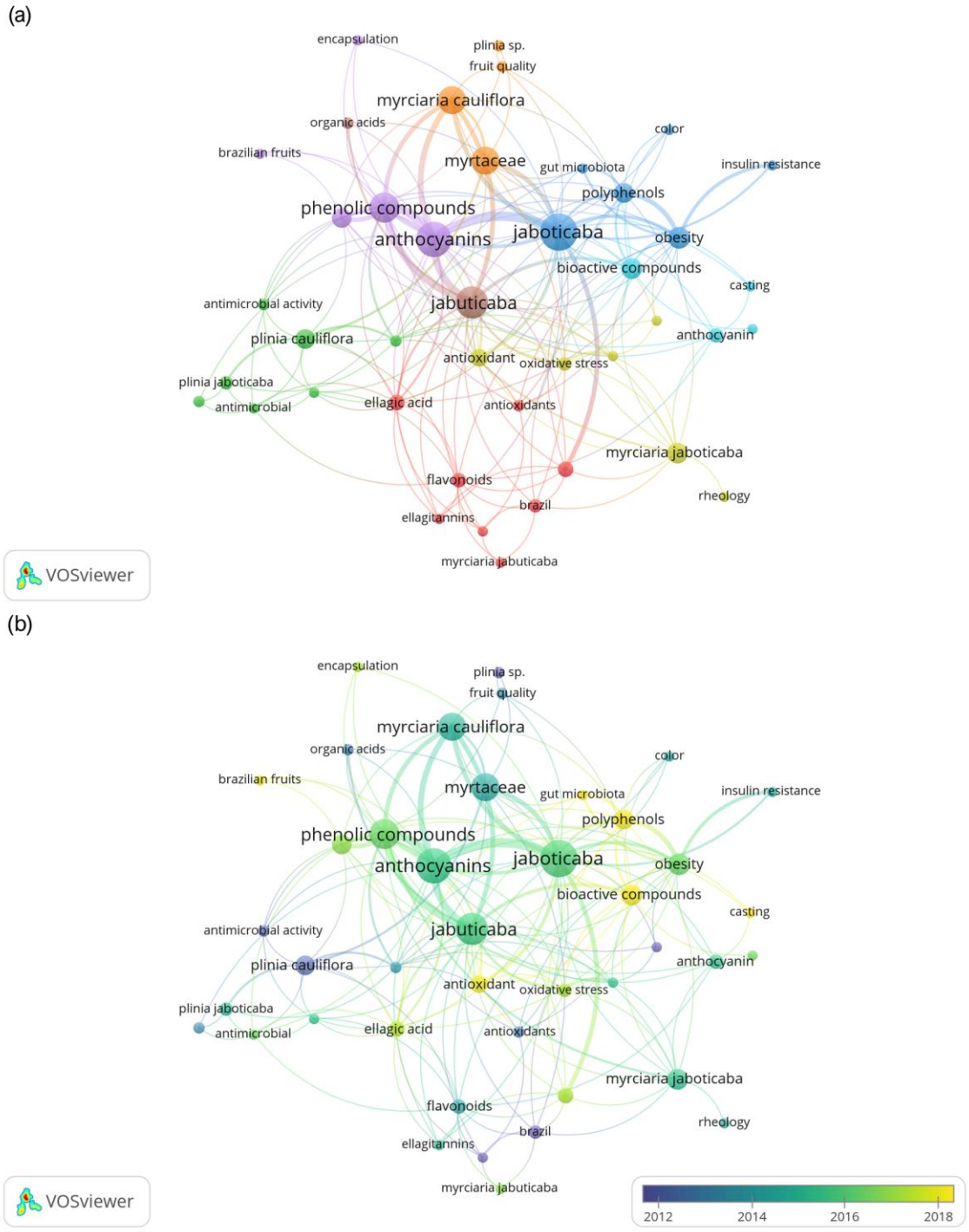
Table 3. Identification of the clusters based on the keywords analysis.

Clusters[1]	Number of items	Keywords
1	8	Antioxidant capacity, antioxidants, Brazil, ellagic acid, ellagitannins, flavonoids, fruits, and *Myrciaria jabuticaba*
2	7	Antimicrobial, antimicrobial activity, cytotoxicity, HPLC, *Plinia cauliflora*, *Plinia jaboticaba*, and *Plinia trunciflora*
3	6	Color, gut microbiota, insulin resistance, jaboticaba, obesity, and polyphenols
4	6	Antioxidant, lipid profile, *Myrciaria jaboticaba*, *Myrciaria jaboticaba* (vell.) berg, oxidative stress, and rheology.
5	5	Anthocyanins, antioxidante acitivity, brazilian fruits, encapsulation, and phenolic compounds
6	4	Fruit quality, bioactive compounds, casting, and postharvest
7	2	Jabuticaba and organic acids

[1] Cluster represented in **Figure 4a.**

Notwithstanding, **Figure 4b** demonstrates the term average year map, a tool to observe the publication evolution based on the keyword analysis. From **Figure 4b,** the hottest topics in the field are associated with the isolation of bioactive molecules (i.e., anthocyanins and ellagic acid) to determine antioxidant and antimicrobial activity. The jabuticaba properties have been extensively studied since 2010 and have been quoted mainly since 2015. The results showed that it was possible to identify the highest priority topic for the study of jabuticaba, which is the extraction of bioactive compounds with eco-friendly technologies for human health purposes.

Figure 4. The most employed authors' keywords. (a) Term map based on different clusters; (b) Term average year map.



3.3 The most cited publications

Table 4 presents the most cited articles related to research using jabuticaba. The article "*Blue sensitizers for solar cells: Natural dyes from Calafate and Jabuticaba*" was the most cited (146 citations), published at *Solar Energy Materials and Solar Cells*. In this study, the authors used anthocyanin extracts obtained from jabuticaba. The results indicated a high conversion of visible light into electricity or a broadband semiconductor in solar cells sensitized with anthocyanins (Polo and Iha, 2006).

The 2nd most cited article studied the extraction of anthocyanins using methanol as a solvent of twelve (12) different fruits, including jabuticaba, finding a radical DPPH scavenging assay value of 6.2 ± 0.7 μg mL^{-1} (IC_{50}) and identified the presence of cyanidine-3-glucode in the semi-purified fractions. This study is entitled "*Anthocyanin antioxidants from edible fruits*", presents 133 citations and was published at *Food Chemistry* (Einbond et al., 2004). In this same research area, the article "*Optimization and economic evaluation of pressurized liquid extraction of phenolic compounds from jabuticaba skins*", published at *Journal of Food Engineering*, presented 103 citations, the 6th most cited paper in the ranking. In this study, the authors concluded that the pressurized liquid extraction (PLE) allowed the obtention of anthocyanins (13.0 ± 0.9 mg cyanidin-3-glucose g^{-1} dry material) and phenolic compounds (7.8 ± 0.4 mg of GAE g^{-1} dry material) from the jabuticaba peels (Santos et al., 2012). Following, the 3rd most cited article was "*Parameter optimization for spray-drying microencapsulation of jabuticaba (Myrciaria jabuticaba) peel extracts using simultaneous analysis of responses*". The authors concluded that the use of maltodextrin and Arabic gum allowed the formation of more homogeneous particles, being cited 125 times and published at *Journal of Food Engineering* (Silva et al., 2013).

Table 4. Top 10 most cited documents in the field of jabuticaba research.

Ranking	Title	Journal	Publication Year	Total Citations*	Average Citation per Year	Reference
1st	Blue sensitizers for solar cells: Natural dyes from Calafate and Jabuticaba	Solar Energy Materials and Solar Cells	2006	146	10.82	Polo and Iha (2006)
2nd	Anthocyanin antioxidants from edible fruits	Food Chemistry	2004	133	9.05	Einbond et at. (2004)
3rd	Parameter optimization for spray-drying microencapsulation of jabuticaba (*Myrciaria jabuticaba*) peel extracts using simultaneous analysis of responses	Journal of Food Engineering	2013	125	14.8	Silva et al. (2013)
4th	Jabuticaba peel: Antioxidant compounds, antiproliferative and antimutagenic activities	Food Research International	2012	109	12.36	Leite-Legatti et al. (2012)
5th	Dietary anthocyanins against obesity and inflammation	Nutrients	2006	106	21.67	Lee et al. (2017)
6th	Optimization and economic evaluation of pressurized liquid extraction of phenolic compounds from jabuticaba skins	Journal of Food Engineering	2017	103	9.64	Santos et al. (2012)
7th	Screening of the chemical composition and occurring antioxidants in jabuticaba (*Myrciaria jabuticaba*) and jussara (*Euterpe edulis*) fruits and their fractions	Journal of Functional Foods	2012	102	13	Inada et al. (2015)
8th	Bioactive depsides and anthocyanins from jabuticaba (*Myrciaria cauliflora*)	Journal of Natural Products	2012	101	6.41	Reynertson et al. (2006)
9th	Metabolite profiling of jabuticaba (*Myrciaria cauliflora*) and other dark-colored fruit juices	Journal of Agricultural and Food Chemistry	2010	89	9.45	Wu et al. (2012)
10th	Characterization of different fruit wines made from cacao, cupuassu, gabiroba, jabuticaba and umbu	LWT – Food Science and Technology	2015	86	7.85	Duarte et al. (2010)

* Total citations recorded until 2021.

Following, a study quantified the anthocyanins content in jabuticaba peels and verified its *in vitro* antiproliferative activity (Leite-Legatti et al., 2012). This work was cited 109 times, occupying the 4th position in the ranking. The study concluded that lyophilized jabuticaba peels are rich in fiber and anthocyanins (delfinidine-3-glycoside and cyanid-3-glycoside), presenting high antioxidant activity. In the 5th position (106 citations), the article "*Dietary against obesity and inflammation*", published in *Nutrients*, described the beneficial effects of dietary anthocyanins on metabolic disorders caused by obesity and inflammation, citing various fruits that have a variety of anthocyanins, in addition to discussing the interaction between inflammation and obesity, and their subsequent regulation through the use of dietary anthocyanins (Lee et al., 2017).

Also, concerning bioactive compounds, the 7th work, published at *Journal of Functional Foods*, was cited 102 times, with the title "*Screening of the chemical composition and occurring antioxidants in jabuticaba (Myrciaria jabuticaba) and jussara (Euterpe edulis) fruits and their fractions*". The authors found eleven phenolic compounds, the most abundant anthocyanins, demonstrating a high commercial potential due to nutritional and functional properties (Inada et al., 2015). Beyond, the polar and non-polar extracts showed antiproliferative effects against leukemia and prostate cancer, respectively. Another study with a similar purpose was published at *Journal of Natural Products*, entitled "*Bioactive depsides and anthocyanins from jabuticaba (Myrciaria cauliflora)*", accounting for 101 citations, is the 8th most cited article. The authors showed that anthocyanins and jabuticaba depsides have good antiradical and cytotoxicity activity, inhibiting the production of IL-8 in epithelial cells of the small airways, treated and not treated with cigarette smoke extract (Reynertson et al., 2006). The 9th most cited article reported that jabuticaba is a bright fruit with good antioxidant capacity. However, during processing, the levels of some of the anthocyanins and other polyphenols significantly decrease,

reducing the antioxidation capacity of these products concerning fresh fruit, thus diminishing the benefits to human health (Wu et al., 2012).

In the 10th position, the study "*Characterization of different fruit wines made from cacao, cupuassu, gabiroba, jabuticaba and umbu*" published at *LWT – Food Science and Technology* presents 86 citations. This study evidenced that the use of tropical fruits for wine production is a viable alternative that allows harvest surplus and a nobler destination for underused fruits, resulting in the introduction of new products into the market (Duarte et al., 2010).

Finally, it is observed that all of the ten (10) most cited studies using jabuticaba focused on the bioactive compounds of the fruit in the areas of food, nutrition, and health. From the studies carried out, it is possible to indicate research gaps in the scientific literature, to define new alternatives for the valorization of jabuticaba, and to identify other possible technological routes or applications.

3.4 Bibliometric study of authors, journals, institutions, and countries

The bibliometric study of jabuticaba research covered the analysis of cited authors, journals, institutions (affiliations), and countries. The most productive and cited authors are Marostica MR (18 publications), Wagner A (11 publications), Citadin I (11 publications), Batista AG (9 publications), and Correa AD (8 publications). The most critical journals in the field were *Food Research International* (6.15%), *Revista Brasileira de Fruticultura* (6.15%), *Food Chemistry* (4.61%), and *Journal of Functional Foods* (2.69%), accounting for 51 documents in total. Otherwise, from the 260 documents, around 20% were published in the four mentioned journals, indicating that studies on jabuticaba have been published in a range of journals, highlighting the studies on Food Science Technology. Moreover, the ten (10) most prestigious journals are related to the scope and research area of bioactive compounds,

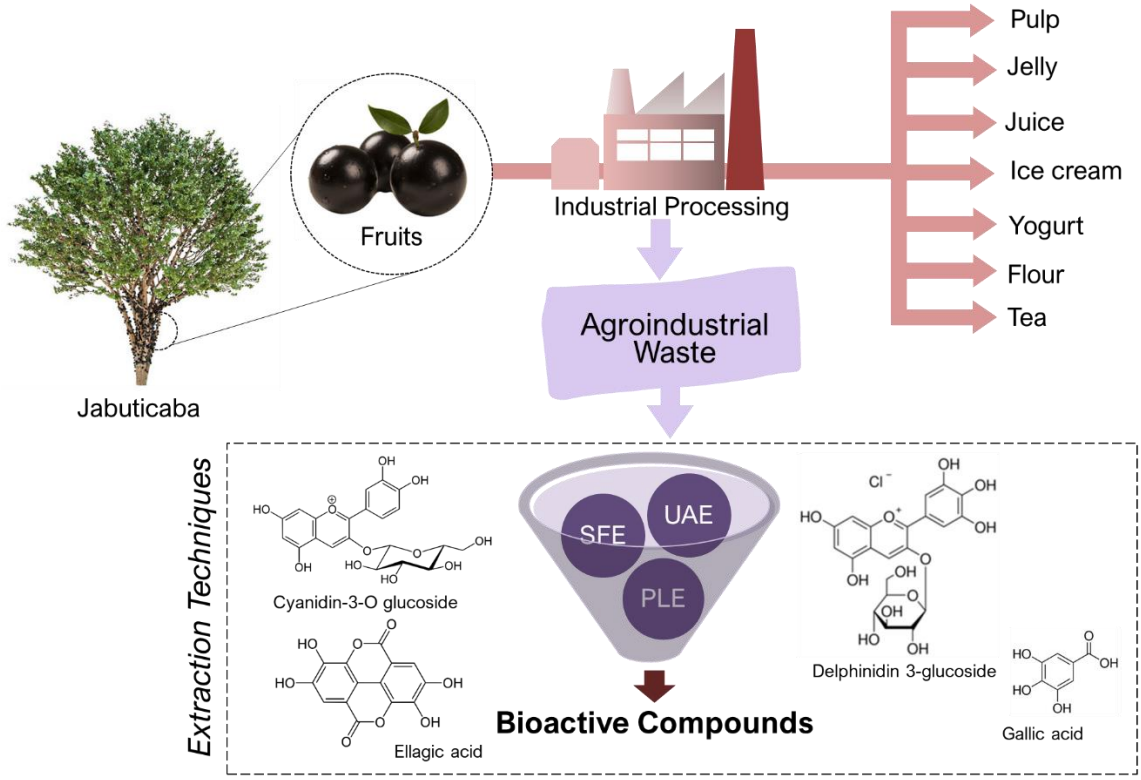
phytochemicals, biological activity, and food processing, indicating that most of the publications with jabuticaba are associated with these research field.

Several publications were used to evaluate the countries and institutions involved in the study of jabuticaba. The leading country is Brazil (236 publications), followed by the United States of America (15 publications), Portugal (7 publications), and Spain (7 publications). A network between the countries was observed, revealing that the scientific community is joining efforts to propose solutions to extract bioactive compounds from jabuticaba. The most productive institutions were universities from Brazil: the University of São Paulo (USP) (42 publications) and the University of Campinas (UNICAMP) (40 publications), affiliations with the highest number of publications.

4. Industrial processing of jabuticaba

After the harvest of jabuticaba, the fruit presents rapid senescence due to the high sugar and water content (de Sá et al., 2014). The industrial processing of jabuticaba is an excellent option to increase the shelf life. For this, processing goes through a few steps, starting with fruits selection, washing and sanitizing, followed by heating application to extract pigments from the skin, and finally pulping in machines that separate the pulp from skin and seeds (Benvenutti et al., 2021). From the industrial processing juices, ice creams, liqueurs, wines, jams, syrups, yogurts, and powdered products can be obtained (Tarone et al., 2021). **Figure 5** illustrates the industrial processing of jabuticaba and the marketable product generated.

Figure 5. Illustration of industrial processing of jabuticaba and value-added products generated.



The industrial processing of jabuticaba generates large amounts of by-products (peel, seeds, and residual pulp) with a considerable amount of bioactive compounds, fibers, proteins, and vitamins than whole fruit (Albuquerque et al., 2020; Benvenutti et al., 2021; Gurak et al., 2014). Thus, it is crucial to identify and evaluate the new industrial process to recover value-added compounds presented in the by-product that would be discarded without any use, reducing the environmental impacts simultaneously as it aggregates value to the by-products.

Jabuticaba peels do not yet have an established commercial profile, although verified their potential of phytochemical compounds in their composition, is traditionally used for tea. The high concentration of phenolic compounds opens the perspective to the user as a dye (Dallabona et al., 2020), incorporation in foods (Oliveira et al., 2018) to claim functionality (Morales et al., 2016), and the strict isolation of these compounds for the use of the

pharmacological and chemical sectors (Albuquerque et al., 2020). For example, cyanidin-3-O-glucoside and delphinidin-3-O-glucoside isolated with purity above 95% in Sigma-Aldrich® (10 mg packages) is sold by 162 to 613 USD.

Notwithstanding, studies on the practical applications of jabuticaba by-products are recent and involve technological innovation due to in-depth knowledge on its potential characteristics hitherto unknown (Soares et al., 2018). There are several patents involving the utilization of jabuticaba by-products. Most of them have been granted since innovative processes and technologies were developed. For instance, Gonçalves et al. (2016) reported the patent "*Jabuticaba shell has undergoing dehydration processes has antioxidants, phenolic compounds, carotenoids and tannins*", which consists in the fact that jabuticaba peel has bioactive compounds and allows the consumption of healthy food. The jabuticaba peel should be subjected to osmotic dehydration and oven drying. The osmotic solution was used at concentrations of 40-70 °Brix. The resulting product can replace different kinds of sugars such as sucrose, glucose, fructose, dextrose, maltodextrin, maltose, sweeteners such as aspartame, sucralose, stevia, and sorbitol as individuals or as mixtures. Moreover, a patent reported the composition of *Myrciaria cauliflora*'s extract and its use for obesity-related metabolic abnormalities control, especially the inhibition of diseases caused by high-fat diets, including obesity, fatty liver, and chronic inflammation (Huang and Wang, 2008).

Regarding the development of technological processes for food production, another patent focused in bioactive compounds extraction system using supercritical carbon dioxide as solvent and water, ethanol, or isopropanol as co-solvents (Meireles et al., 2018). The final product comprised a probiotic microorganism containing a supercritical extract of jabuticaba peels with antioxidant effect. The preferred operational conditions reported are solvent and co-solvent ratio of 80:20 (w/w), at 50 °C under 10 to 30 MPa. This extract can be used for yogurt or cheese (petit suisse) formulation. Beyond, Oliveira and Steel (2019) produced cereal in co-

rotating double screw extruder from jabuticaba peel flour. The extruded and expanded cereal contained 80% of wheat flour, 10% of jabuticaba peel flour, and 10% of corn flour. For this, the extrusion conditions were fixed as follows: feed moisture content of 16%, feed rate of 13 kg h^{-1}, roscade speed of 325 RPM, 75 °C in the first zone, and 100 °C in the second, third, and fourth zone. Therefore, jabuticaba by-products can be applied in different ways, however, the most appropriate technologies should be used to increase the profitability and to decrease environmental impacts. In the next section, innovative processes for the valorization of jabuticaba by-products was described to elucidade the production of bioactive compounds.

5. Technological routes to the valorization of jabuticaba by-products

The production chain of jabuticaba does not take advantage of the peels and seeds, so these parts of the fruit are seen as waste, totaling 50% (w/w) of peels and seeds (Morales et al., 2016). Nevertheless, the scientific literature works to the great biological potential of these by-products. For instance, the peel of jabuticaba has high antioxidant activity (Albuquerque et al., 2020), as well as anti-inflammatory, antimutagenic, antimicrobial, antiproliferative action (Tarone et al., 2021). Other studies reported the potential applications as increased insulin resistance (Lenquiste et al., 2012), the oxidizing effect of blood plasma (Leite et al., 2011) without hepatotoxicity, and the absence of toxicity (Albuquerque et al., 2020).

The compounds found in jabuticaba are mostly phenolic, produced as secondary metabolites, and part of the defense mechanisms of plants against ultraviolet rays, attacks of insects or animals, and pathogens (Alara et al., 2021). Phenolic compounds have applications in different areas, such as food, aesthetics, and pharmaceutical products (Mikołajczak et al., 2021). In the food industry, its application has increased in products stability and nutritional and sensory properties (Heck et al., 2020; Martins et al., 2021). Due to its antioxidant action, it

also has application in the human diet as a preventive action against diseases and deceleration of reactions related to oxidative stress (Alara et al., 2018).

Table 5 presents the compounds, quantity, and method of extraction of phytochemical compounds of jabuticaba (pulp, seed, and peels). **Figure 6** presents the chemical structure of bioactive compounds obtained from jabuticaba. From the peels, cyanidin-3-O-glucoside and delphinidin-3-O-glucoside have been widely extracted. Malvidin, peonidine, petunidine, and pelargonidine, complete the group of anthocyanin compounds relevant to the food industry (Santos-Buelga et al., 2019), either for their bioactive properties or as a dye (Pires et al., 2021). The greater extraction of phenolic compounds from a plant material depends on the sample type and phenolics content. The quantification and highest yield of phenolic compounds depend on the extraction technique (Alara et al., 2021). Still, crude extracts are traditionally obtained by solvent extraction methods, maceration, infusion, percolation, and decoction (Alara et al., 2018). Modern ultrasound-assisted extraction techniques, pressurized wate, supercritical fluids, solid-phase dispersion, and microwaves have shown high extraction efficiency (Fernandes et al., 2020; Selvamuthukumaran and Shi, 2017; Senes et al., 2021).

Figure 6. Chemical structure of bioactive compounds obtained from jabuticaba. (a) 3,4-dihydroxybenzoic acid; (b) 4-hydroxybenzoic acid; (c) catechin; (d) caffeic acid; (e) chlorogenic acid; (f) cyanidin-3-O-glucoside; (g) delphinidin-3-O-glucoside; (h) ellagic acid; (i) ferulic acid; (j) gallic acid; (k) isoquercitrin; (l) isorhamnetin; (m) kaempferol; (n) luteolin; (o) malvidin; (p) myricetin; (q) naringenin; (r) p-coumaric acid; (s) pelargonidine; (t) peonidine; (u) petunidine; (v) pinobanksin; (w) quercetin; and (x) syringic acid

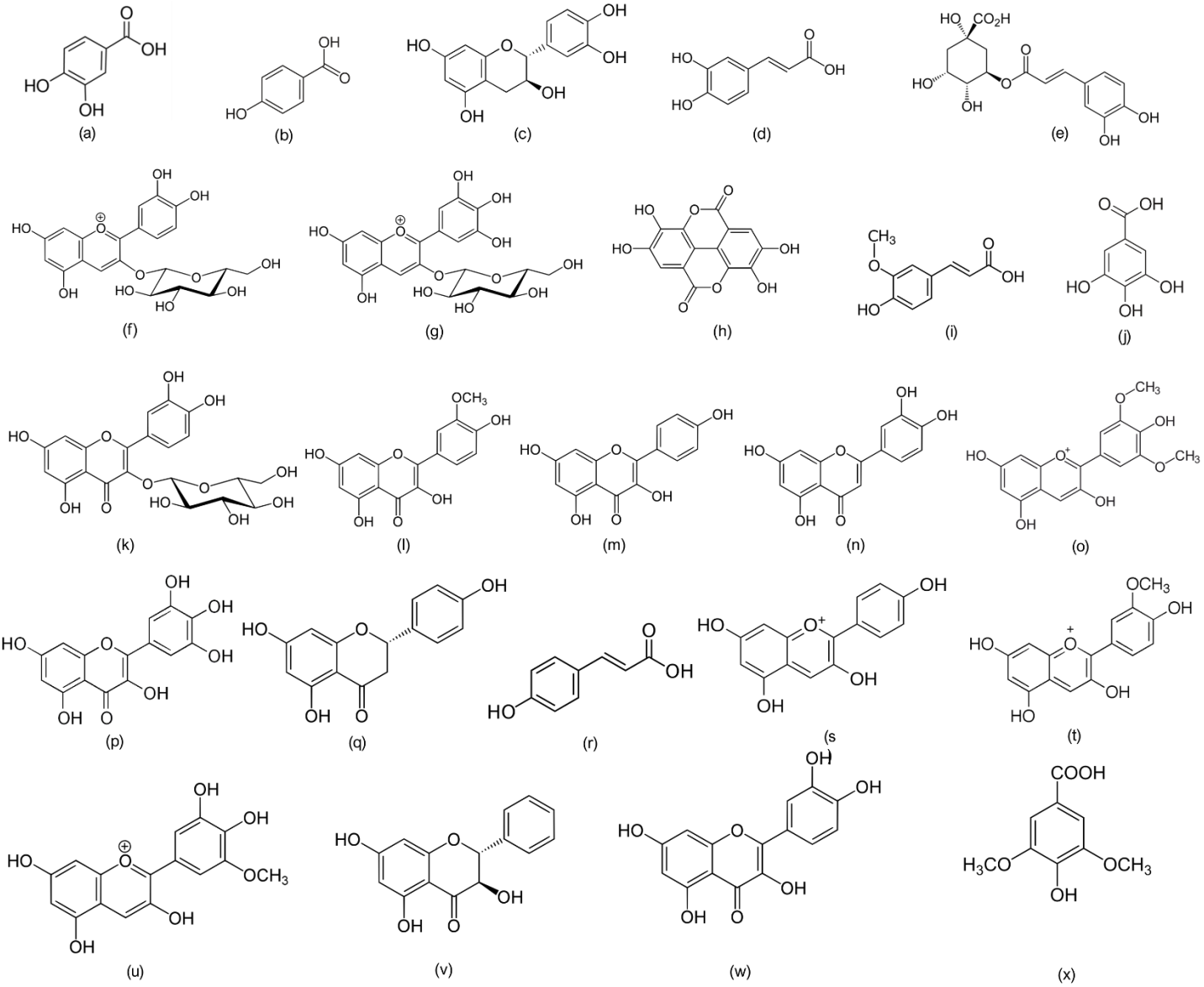


Figure 7 illustrates the most usual extraction techniques of bioactive compounds from jabuticaba. Briefly, new extraction procedures based on pressurized liquid extraction were reported in the literature. This process uses organic solvents at high pressure and a temperature above their boiling point to extract the analytes from the sample matrices. Higher pressure increases the contact between the extracting fluid and sample, while higher temperature is used to break the analyte-matrix bonds (Dias et al., 2021). Beyond, supercritical fluid extraction has been used with gas as solvent in its critical point (Singh et al., 2021). Carbon dioxide (CO_2) is usually used as solvent in this method because it can extract lipid-soluble compounds and it enables a high-level recovery.

Moreover, there are several benefits for the use of CO_2, such as its low cost, non-hazardous and non-flammable aspects, and safety (Lefebvre et al., 2021). In the case of ultrasound-assisted extraction, mass transfer increase is observed, and the diffusion of the solvent into the matrix is enhanced due to pores creation in the membranes, which allows higher access to the bioactive compounds extraction. The ultrasound technology has been demonstrated as a rapid and highly effective one for mass transfer due to cavitation phenomena, being more and more applied in food processing and natural products extraction (Khadhraoui et al., 2021).

Studies characterized jabuticaba concerning its phenolic compounds, antioxidant capacity, total monomeric anthocyanin, sugars, and minerals during ripening (Seraglio et al., 2018). The authors concluded that jabuticaba could be considered a source of natural bioactive and nutritional compounds is a promising raw material for the food industry. The jabuticaba has high antioxidant power, presenting excellent protection against lipid oxidation, potential to be used as an additive in the food industry, with possible benefits to consumer health (Lima et al., 2011)

Figure 7. Scheme of the most usual extraction techniques of bioactive compounds from jabuticaba. (a) ultrasound-assisted extraction; (b) solid-liquid extraction; (c) supercritical carbon dioxide extraction; and (d) pressurized liquid extraction.

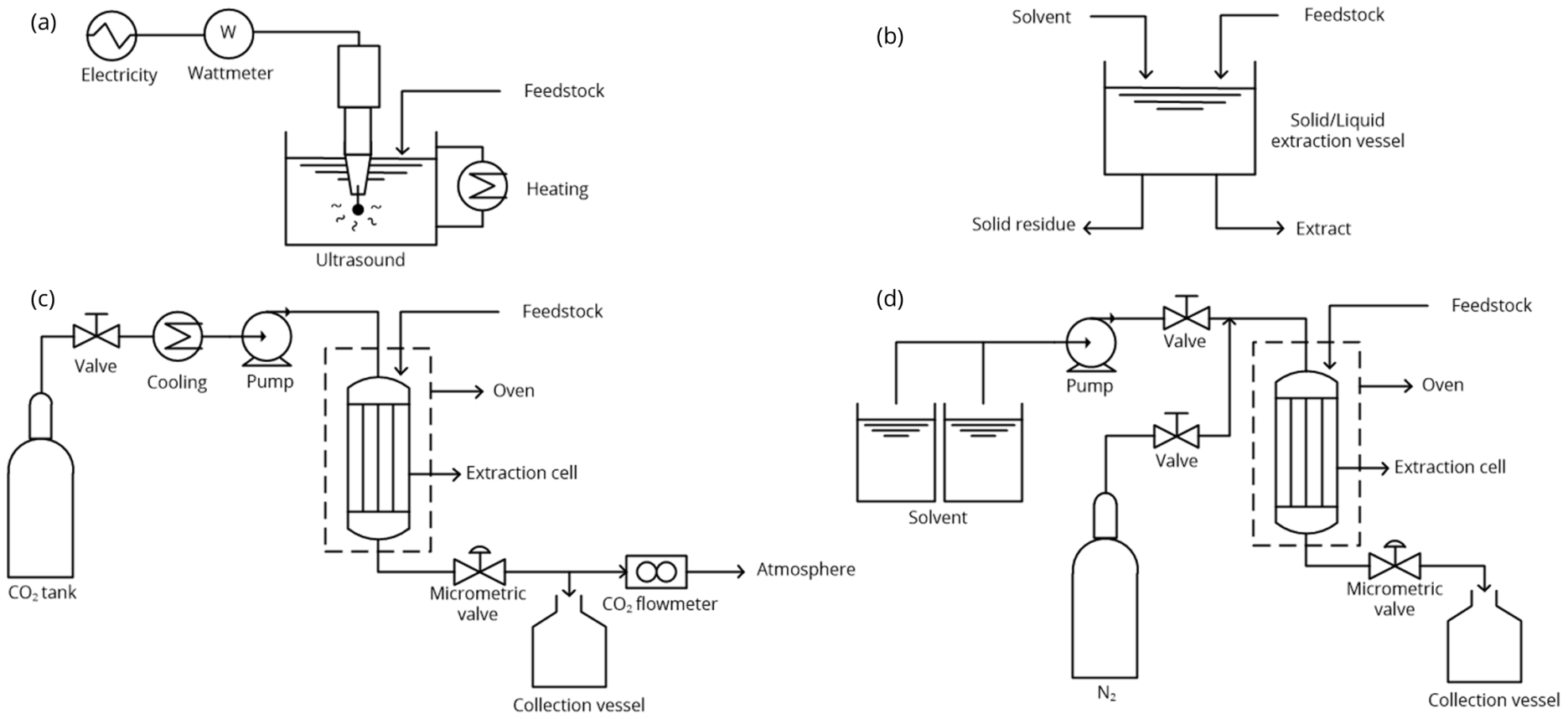


Figure 8 illustrates the several technological routes for the valorization of jabuticabas by-products to obtain bioactive compounds. The main technologies reported are extraction by maceration, solid-phase extraction, ultrasound-assisted, high-intensity ultrasound, pressurized hot water, and high-pressure carbon dioxide. All of them obtained bioactive compounds from jabuticaba peels varying yield, concentration, or isolation depending on the selected technology, as can be observed in **Table 6.**

Figure 8. Technological routes for the valorization of jabuticaba by-products to obtain value-added products.

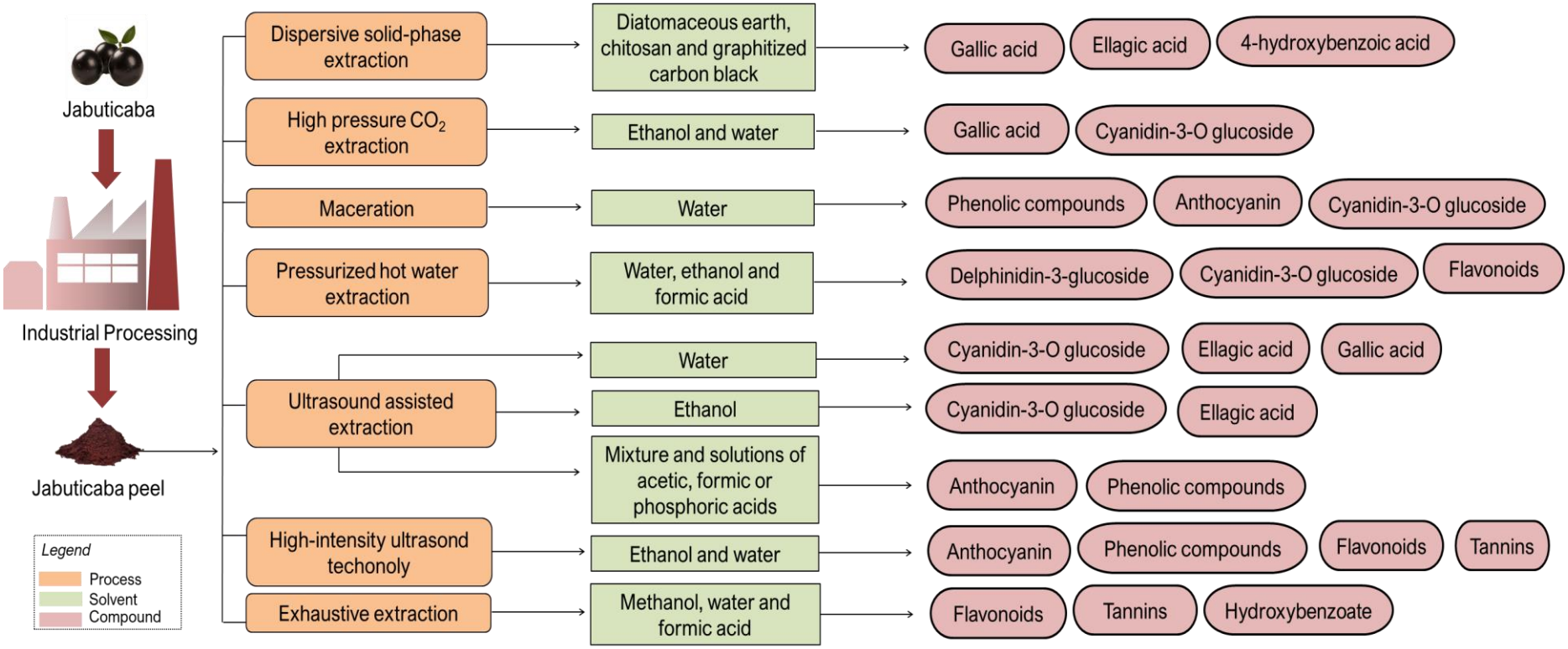


Table 5. Compounds, quantity, and method of extraction of phytochemical compounds of Jabuticaba (*Myrciaria cauliflora*).

Compunds	Part of the plant	Quantity (mg kg^{-1})	Method	Reference
3,4-dihydroxybenzoic acid	Pulp and peel	12.99	Acid hydrolysis with HCl and detection by LC-ESI-MS/MS	Seraglio et al. (2018)
4-hydroxybenzoic acid	Pulp and seed	0.6 – 1.8	Extraction with VA-MSPD and detection by UHPLC-MS / MS	Senes et al. (2021)
Caffeic acid	Pulp and peel	0.26	Acid hydrolysis with HCl and detection by LC-ESI-MS/MS	Seraglio et al. (2018)
Catechin	Pulp and seed	3.0 – 15.0	Extraction with VA-MSPD and detection by UHPLC-MS/MS	Senes et al. (2021)
Chlorogenic acid	Pulp and peel	1.93	Acid hydrolysis with HCl and detection by LC-ESI-MS/MS	Seraglio et al. (2018)
Ellagic acid	Pulp and seed	39.6 – 198	Extraction with VA-MSPD and detection by UHPLC-MS/MS	Senes et al. (2021)
	Pulp, peel and seed	5.05×10^{4}	Extraction with methanol, water, and acetic acid solution (70:30:0.5, v/v/v) and detection with HPLC-PDA	Alezandro et al. (2013)
Ferulic acid	Pulp and peel	1.99	Acid hydrolysis with HCl and detection by LC-ESI-MS/MS	Seraglio et al. (2018)
Gallic acid	Pulp and seed	4 – 12	Extraction with VA-MSPD and detection by UHPLC-MS/MS	Senes et al. (2021)
	Pulp and peel	41.64	Acid hydrolysis with HCl and detection by LC-ESI-MS/MS	Seraglio et al. (2018)
Syringic acid	Pulp and peel	0.97	Acid hydrolysis with HCl and detection by LC-ESI-MS/MS	Seraglio et al. (2018)
	Pulp and seed	3 – 15	Extraction with VA-MSPD and detection by UHPLC-MS/MS	Senes et al. (2021)
p-coumaric acid	Pulp and peel	3.58	Acid hydrolysis with HCl and detection by LC-ESI-MS/MS	Seraglio et al. (2018)
p-coumaric acid	Pulp and seed	0.18 – 0.54	Extraction with VA-MSPD and detection by UHPLC-MS/MS	Senes et al. (2021)
Cyanidin-3-O-glucoside	Peel	19.45×10^{3}	Extraction with water and ethanol (80:20) (v/v) and detection by HPLC-PDA-ESI/MS	Albuquerque et al. (2020)
	Pulp, peel and seed	1.23×10^{3}	Extraction with methanol, water, and acetic acid solution (70:30:0.5, v/v/v) and detection with HPLC-PDA	Alezandro et al. (2013)
	Peel	2.58×10^{4}	Maceration with ethanol acidified with 1.5 mol L^{-1} HCl (85:15, v/v) and quantification by HPLC-PDA	Lima et al. (2011)

	Pulp	1.8×10^{2}	Maceration with ethanol acidified with 1.5 mol L^{-1} HCl (85:15, v/v) and quantification by HPLC-PDA	Lima et al. (2011)
Delphinidin-3-O-glucoside	Peel	5.09×10^{3}	Extraction with water and ethanol (80:20) (v/v) and detection by HPLC-PDA-ESI/MS	Albuquerque et al. (2020)
	Pulp, peel and seed	2.35×10^{2}	Extraction with methanol, water, and acetic acid solution (70:30:0.5, v/v/v) and detection with HPLC-PDA	Alezandro et al. (2013)
	Peel	3.09×10^{3}	Maceration with ethanol acidified with 1.5 mol L^{-1} HCl (85:15, v/v) and quantification by HPLC-PDA	Lima et al. (2011)
Isoquercitrin	Pulp and peel	8.64	Acid hydrolysis with HCl and detection by LC-ESI-MS/MS	Seraglio et al. (2018)
Isorhamnetin	Pulp and peel	0.79	Acid hydrolysis with HCl and detection by LC-ESI-MS/MS	Seraglio et al. (2018)
Kaempferol	Pulp and seed	3 – 15	Extraction with VA-MSPD and detection by UHPLC-MS/MS	Senes et al. (2021)
	Pulp and peel	0.33	Acid hydrolysis with HCl and detection by LC-ESI-MS/MS	Seraglio et al. (2018)
Luteolin	Pulp and peel	0.05	Acid hydrolysis with HCl and detection by LC-ESI-MS/MS	Seraglio et al. (2018)
Myricetin	Pulp and seed	0.3 – 1.5	Extraction with VA-MSPD and detection by UHPLC-MS/MS	Senes et al. (2021)
Naringenin	Pulp and seed	0.12 – 0.36	Extraction with VA-MSPD and detection by UHPLC-MS/MS	Senes et al. (2021)
	Pulp and peel	0.37	Acid hydrolysis with HCl and detection by LC-ESI-MS/MS	Seraglio et al. (2018)
Pinobanksin	Pulp and peel	0.42	Acid hydrolysis with HCl and detection by LC-ESI-MS/MS	Seraglio et al. (2018)
Quercetin	Pulp and seed	0.3 – 1.5	Extraction with VA-MSPD and detection by UHPLC-MS/MS	Seraglio et al. (2018)
	Pulp and peel	52.11	Acid hydrolysis with HCl and detection by LC-ESI-MS/MS	Seraglio et al. (2018)

Label: VA-MSPD, vortex-assisted matrix solid-phase dispersion; UHPLC-MS/MS, ultra-high performance liquid chromatography-mass spectrometry; LC-ESI-MS/MS, liquid chromatography electrospray ionization tandem mass spectrometric; HPLC-PDA, high-performance liquid chromatography with photodiode array detection; HPLC-PDA-ESI/MS, high performance liquid chromatography with photodiode array detection and electrospray ionization tandem mass spectrometry.

Table 6. Synthesis of research on the extraction of bioactive compounds from jabuticaba peel.

Obtained compounds	Methodology	Yield	Additional information	References
Cyanidin-3-O-glucoside and ellagic acid.	Ultrasound assisted extraction using ethanol as solvent.	4.8 mg of anthocyanin g^{-1} dry peel; 92.8 mg of gallic acid g^{-1} dry peel; 4.9 mg of cyanidin-3-O-glucoside g^{-1} dry peel; 7.8 mg ellagic acid g^{-1} dry peel.	The results showed that with an adequate operating condition it was possible to reach good yields using a simple extraction process.	Rodrigues et al. (2015)
Anthocyanin and phenolic compounds.	Ultrasound assisted extraction using a hydroalcoholic mixture and solutions of acetic, formic or phosphoric acids to regulate pH.	3.4 mg anthocyanin g^{-1} raw material; 841 μmol Trolox g^{-1} raw material for antioxidant capacity.	The acid that presented the best recovery of anthocyanin and best antioxidant capacity was formic acid in pH 1.0.	Barros et al. (2019)
Anthocyanin, phenolic compounds and Cyanidin-3-O-glucoside	Maceration with distilled water and solutions of hydrochloric acid and sodium hydroxide to regulate pH.	9.7 mg anthocyanin g^{-1} raw material; 230.48 mg GAE g^{-1} raw material; 7.18 mg cyanidin-3-O-glucoside g^{-1} raw material.	The optimal conditions for extraction were 88 °C and pH 1.0.	Avila et al. (2020)
Cyanidin-3-O-glucoside, ellagic acid and gallic acid.	Ultrasound assisted extraction using water as solvent.	8.9 mg GAE g^{-1} dry peel; 0.9 mg ellagic acid g^{-1} dry peel; 7.9 mg cyanidin-3-O-glucoside g^{-1} dry peel.	The highest yield of bioactive compounds was attained at 25 kHz, 20 min of extraction and pH 1.5.	Fernandes et al. (2020)
Cyanidin-3-glucoside and gallic acid.	High pressure carbon dioxide assisted extraction.	2.2 mg cyanidin-3-glucoside g^{-1} dry peel; 13 mg GAE g^{-1} dry peel.	The best extraction conditions were achieved at 117 bar, 80 °C and 20% volume ratio of solid–liquid mixture/pressurized CO_2.	Santos and Meireles (2011)

Polyphenol, anthocyanin, flavonoids and tannins.	High-intensity ultrasound technology with water and ethanol as solvents.	3391 mg GAE L^{-1}; 287 mg anthocyanin L^{-1}; 667 mg flavonoids L^{-1}; 11,265 mg tannins L^{-1}.	This tecnique promoted the best recovery of bioactive compounds at an ultrasound intensity of 3.7 W cm^{-2} and 50 g water 100 g^{-1} ethanol.	Gadioli Tarone et al. (2021)
Ellagic acid, gallic acid and 4-hydroxybenzoic acid.	Extraction with methanol / H_2O solution (70:30 v/v) for 30 min at 30°C in an ultrasonic bath with a frequency of 37 kW and 320 W,	1643 mg ellagic acid kg^{-1}; 252 mg gallic acid kg^{-1}; 154mg 4-hydroxybenzoic acid kg^{-1}.	The use of diatomaceous earth with graphitized carbon black in a ratio of 100:10 (w/w) was the best clean-up step condition.	Senes et al. (2020)
Cyanidin 3-glucoside, delphinidin 3-glucoside, ellagitannins, gallotannins, ellagic acid and derivatives, and flavonols.	Pressurized hot water extraction using water, ethanol and formic acid as solvents.	2866.2 mg cyanidin 3-glucoside 100 g^{-1} dry peel, representing around 63% of the total phenolic compounds.	Even though cyanidin 3-glucoside is in the highest concentration, it represents only 25% of the total antioxidant capacity.	Plaza et al. (2016)
Anthocyanins, flavonols and ellagic acid derivates and hydroxybenzoate derivatives or tannins.	Exhaustive extraction with solutions of methanol, water and formic acid as solvents.	1813 mg total anthocyanins 100 g^{-1} dry peel; 356.5 mg ellagic acid derivates 100 g^{-1} dry peel; 3315.4 total hydroxybenzoate 100 g^{-1} dry peel.	There were analyzed two different species of jabuticaba: *Myrciaria jabuticaba* and *M. trunciflora*. The second one presented the highest levels of bioactive compounds in general.	Quatrin et al. (2019)

The ultrasound-assisted extraction technique using ethanol as a solvent demonstrates promising results for dry jabuticaba peel, of 4.8 mg of anthocyanin g^{-1}, 92.8 mg of gallic acid g^{-1}, 4.9 mg of cyanidin-3-O-glucoside g^{-1}, and 7.8 mg ellagic acid g^{-1} (Rodrigues et al., 2015). With the same technique with water as the solvent, 8.9 mg gallic acid equivalent g^{-1}, 0.9 mg ellagic acid g^{-1}, and 7.9 mg cyanidin-3-O-glucoside g^{-1} can be obtained, with the highest yields of bioactive compounds at 25 kHz, 20 minutes of extraction, and pH 1.5 (Fernandes et al., 2020). Also, the ultrasound-assisted extraction technique showed the effect of the addition of formic, acetic, and phosphoric acids to the extraction process (Barros et al., 2019). The authors concluded that formic acid with pH 1.0 had the best recovery of anthocyanins obtaining 3.4 mg anthocyanin g^{-1} material and an antioxidant capacity of 841 μmol Trolox g^{-1} material.

Using the high-intensity ultrasound technology technique, at the intensity of 3.7 W cm^{-2} and 50 g water 100 g^{-1} ethanol, the best results of recovery of bioactive compounds were obtained: 3391 mg GAE L^{-1}; 287 mg anthocyanin L^{-1}; 667 mg flavonoids L^{-1}; and 11625 mg tannins L^{-1} (Tarone et al., 2021). Thus, phenolic compounds can be recovered with fast, relatively inexpensive, and simple technology that reduces environmental costs and impacts compared to conventional extraction processes.

Using an exhaustive extraction of dried jabuticaba peel with water, ethanol, and formic acid solution as a solvent in the ratio 85:15:0.5 (v/v), a polyphenolic profile of two varieties of jabuticaba (*M. trunciflora*) was evaluated (Quatrin et al., 2019). In the study, the authors obtained high levels of bioactive compounds with a total of 1813 mg anthocyanins 100 g^{-1}, 356.5 mg ellagic acid 100 g^{-1}, and 3315.4 total hydroxybenzoate 100 g^{-1}. Beyond using maceration with distilled water, the optimum extraction condition was at 88 ºC with pH 1. The results obtained (9.7 mg anthocyanin g^{-1}, 230.48 mg GAE g^{-1}, and 7.18 mg cyanidin-3-glucoside g^{-1}) showed that the peels have a high content of total phenolics, antioxidant activity, and anthocyanins (Avila et al., 2020).

Another technological route is the extraction with methanol and water solution (70:30, v/v) for 30 min at 30 ºC in an ultrasonic bath with a frequency of 37 kHz and 320 W (Senes et al., 2020). After this, the extracts were filtered before the cleaning step with diatomaceous earth, chitosan, and graphite carbon black as adsorbents. Eight compounds were found, the majority being ellagic acid (1643 mg kg^{-1}), cutting acid (252 mg kg^{-1}), and 4-hydroxybenzoic acid (154 mg kg^{-1}).

With pressurized fluid technology, high-pressure carbon dioxide-assisted extraction was used to optimize the process variables for the maximum recovery of anthocyanins and phenolic compounds of dried jabuticaba peels (Santos and Meireles, 2011). The best conditions were 117 bar, 80 ºC, and 20% pressurized solid-liquid/CO_2 mixture, finding the total content of phenolic compounds of 2273 mg cyanidin-3-glucoside g^{-1} and 13 mg gallic acid equivalents g^{-1}. Beyond, using pressurized hot water extraction with water, ethanol, and formic acid (94:5:1, v/v) as a solvent, for the extraction of dried jabuticabas peels, the results show that the amount of cyanidin 3-glucoside (2866.2 ± 40.1 mg 100 g^{-1}) represents the total of 63% of the total phenolic compounds of the peels (Plaza et al., 2016).

Currently, jabuticaba peels are considered by-products and are generally discarded without any use. However, the previous research showed that it is possible to extract high-value bioactive compounds, allowing this by-product insertion into a production cycle. The bibliometric analysis presented shows that the scientific community addressed the recovery of bioactive compounds from jabuticaba peels. Moreover, the recycling of agro-industrial by-products is placed within the circular economy, a worldwide concept advocating the industrial process's circularity, closing raw material cycles to maximize resource use (Ghisellini et al., 2016). A new end-of-life concept to reduce, reuse, recycle, and recover resources supports sustainable development from energy, economic, social, and environmental perspectives (Kirchherr et al., 2017).

5. Conclusions and future perspectives

The demand for pharmaceutical products enriched with natural biocompounds to replace synthetic ones is worldwide. Active biocompounds, such as anthocyanins, contribute to human health. Jabuticaba and its by-products present high biological potential, like antioxidant, antimicrobial, anti-inflammatory, antidiabetic, among other functional properties. Despite this, this potential remains scarcely used. Currently, by-products materials constitute a niche market, especially because of environmental, economic, and social implications in the circular economy concept. The jabuticaba peels can be recycled to produce bioproducts with health benefits employing green technologies in a circular economy. Besides reducing industrial waste, the use of emerging green technologies to process jabuticaba by-products allows the recovery of high-quality bioactive compounds. Furthermore, using jabuticaba by-products can support the efficient utilization of a natural resource, this little-explored Brazilian fruit. However, further investigation is necessary to properly address the adoption of biotechnologies to achieve economically viable bioproducts using jabuticaba by-products, redesigning and extending the uses and applications of the potential present in its by-products.

In this study, a systematic review of jabuticaba industrial by-products to obtain valuable active compounds revealed new trends and technologies for the recovery of bioactive compounds. The bibliometric analysis of jabuticaba research indicated that 255 articles and 5 reviews were published over the last 21 years. The most predominant research fields were Food Science Technology, Chemistry, and Agriculture. In addition, from the keywords analysis, it was possible to identify that most of the research is associated with the biological properties of bioactive compounds extracted from jabuticaba. This feedstock and its by-products have been submitted to bioactive compounds extraction in different sustainable technological routes, such

as ultrasound-assisted extraction, supercritical carbon dioxide extraction, and pressurized liquid extraction.

CRediT authorship contribution statement

Rafael Gabriel da Rosa: Conceptualization, Methodology, Formal analysis, Writing - Original Draft; **William Gustavo Sganzerla:** Conceptualization, Methodology, Formal analysis, Writing - Original Draft, Writing - Review & Editing; Tiago L.C.T. Barroso: Writing - Original Draft; Luz S. Buller: Writing - Review & Editing; **Mauro D. Berni:** Writing - Review & Editing, Supervision, Project administration, Funding acquisition; **Tânia Forster-Carneiro**: Writing - Review & Editing, Supervision, Project administration, Funding acquisition.

Declaration of competing interest

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

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CAPÍTULO 3 - Sustainable bioprocess combining subcritical water pretreatment followed by anaerobic digestion for the valorization of jabuticaba *(Myrciaria cauliflora)* agro-industrial by-product in bioenergy and biofertilizer

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Sustainable bioprocess combining subcritical water pretreatment followed by anaerobic digestion for the valorization of jabuticaba (*Myrciaria cauliflora*) agro-industrial by-product in bioenergy and biofertilizer

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Abbreviations

AD, anaerobic digestion; sCOD, soluble chemical oxygen demand; tCOD, total chemical oxygen demand; FID, flame ionization detector; GC, gas chromatograph; GHG, greenhouse gas; HPLC, high-performance liquid chromatography; HRT, hydraulic retention time; OLR, same organic load rate; RID, refractive index detector; SWP, subcritical water pretreatment; SEM, scanning electron microscopy; TFS, total fixed solids; TS, total solids; TVS, total volatile solids; UASB, upflow anaerobic sludge blanket reactor; VFA, volatile fatty acids; VSR, volatile solids loading rate

Abstract

The management of agri-food by-products has received worldwide attention due to concerns about the environmental impacts caused by incorrect deposition. This study presented a sustainable bioprocess combining subcritical water pretreatment (SWP) followed by semi-continuous anaerobic digestion (AD) for the valorization of jabuticaba (*Myrciaria cauliflora*) agro-industrial by-product in bioenergy and agricultural fertilizer. The SWP was conducted at 180 °C, 15 MPa, water flow rate of 10 mL min^{-1}, solvent to feed of 22.5 g g^{-1}, and a kinetic time of 45 min. The AD process was operated in semi-continuous mode under mesophilic and methanogenic conditions. The results demonstrated that the hydrolysate presented glucose (5.78 g L^{-1}), fructose (3.63 g L^{-1}), arabinose (1.82 g L^{-1}), and cellobiose (1.28 g L^{-1}) as major compounds. The use of pretreated jabuticaca by-product increased the methane yield (239.04 L CH_4 kg^{-1} TVS) in the designed bioprocess combining SWP and AD when compared to the AD without pretreatment (42.31 L CH_4 kg^{-1} TVS). The methane produced in the bioprocess with SWP followed by AD can generate 543 kWh t^{-1} of electricity and 2,243.17 MJ t^{-1} of heat, avoiding a total of 177.54 kg $CO_{2\text{-eq}}$ t^{-1}. The digestate generated after AD can be used as a biofertilizer up to a concentration of 0.3 g L^{-1}, without toxic effects on the germination of *Lactuca sativa*. Finally, the sustainable bioprocess designed could be an alternative to the management of jabuticaba by-product within a circular economy framework, producing bioenergy and agricultural fertilizer that can reduce greenhouse gas emissions and environmental pollution in the food industry.

Keywords*: Biorefinery; Biogas; Biomethane; Biofuel; Fertilizer; Circular economy.*

1. Introduction

The combustion of petroleum-based fuels for energy purposes results in significant air pollution and greenhouse gas (GHG) emissions, which are the primary cause of global warming and climate change [1]. In 2021, 36.3 Gt CO_2 were released into the atmosphere because of fossil energy use. The massive worldwide tax and monetary incentives from postpandemic governments have pushed annual levels to an all-time high, with an increase of 2.1 Gt when compared to 2020 [2]. The strategy stimulates interest in renewable fuels from biomass that can be used to replace traditional energy [3].

The agri-food industry is responsible for the generation of high amounts of lignocellulosic by-products during processing. In underdeveloped countries, most of the residues generated are not disposed of in an environmentally friendly manner [4]. However, agri-food by-products can be considered promising raw materials for producing clean energy, and their worldwide abundance facilitates their application in technological routes [5]. For instance, it is possible to use lignocellulosic sugars from switchgrass to produce acetone-butanol-ethanol [6,7]. Hydrogen and biobutanol can be recovered from food waste fermentation with *Clostridium* [8,9]. Hence, the valorization of agri-food by-products with aerobically or anaerobically microorganisms can be an eco-friendly solution to produce biofuels, electrical energy, biosurfactants, bioplastics, biofertilizers [10]. Notwithstanding, there are social, political, and sanitary concerns about the proper disposal of agri-food waste and achieving sustainable development based on the bioeconomy [11]. Industries and governments began seeking novel and cleaner processes to generate energy, driven by the global demand for bioenergy increasing as people become more aware of climate change [12].

In the food industry, the jabuticaba (*Myrciaria cauliflora*) agro-industrial by-product is an example of lignocellulosic biomass that can be exploited for energy generation. During the industrial processing of jabuticaba, several marketable products are produced in the production

chain, including sweets, jellies, extracts, and liqueurs [13]. However, the peel and seeds correspond to approximately 50 % of the weight of the jabuticaba fruit [14]. A recent bibliometric analysis elucidated that the jabuticaba by-product could be used as feedstock to recover biobased products and bioenergy. However, for bioenergy recovery, a pretreatment step should be required to hydrolyze the lignocellulosic biomass and release monosaccharides [15].

Anaerobic digestion (AD) technology can be used to recover bioenergy from biomass. After AD, it is possible to produce a digestate rich in mineral and organic components that can be used as a soil biofertilizer to replace fossil minerals [3,16]. The AD process entails the conversion of organic matter into biogas via the phases of hydrolysis, acidogenesis, acetogenesis, and methanogenesis, which are carried out by various microorganisms [17]. The methane-rich biogas produced by AD can be used as a renewable fuel for automobiles and cooking and can be burned to generate power and heat [18].

However, hydrolysis can be a limiting stage in the AD of lignocellulosic biomass since the polymeric structure of the material may hinder or prevent the conversion to more biodegradable sugars [19]. Therefore, for the management of lignocellulosic biomass (e.g., jabuticaba by-product) with AD, a previous pretreatment may be necessary to facilitate the metabolism of bacteria, providing more fermentable sugars with a shorter chain [20]. Several pretreatment methods have been suggested in the literature, including alkalis, acids, and enzymes [21]. However, these methods present some environmental and economic drawbacks for industrial applications [22].

Hydrothermal pretreatment of biomass has gained attention due to the high yield of biogas produced from lignocellulosic waste, especially when compared to conventional processes without pretreatment [23]. Hydrothermal pretreatment allows for less generation of contaminants without chemical inputs and reduced inhibitory products and residues [24]. Subcritical water has been proposed as a promising hydrothermal pretreatment for

depolymerizing different components from lignocellulosic biomass [25]. To reach the subcritical state of water, the temperature and pressure conditions should be higher than the boiling point (100 °C, 0.1 MPa) and lower than the critical point (374 °C, 22 MPa) [26]. Therefore, subcritical water pretreatment (SWP) can be an alternative to the hydrolysis of the lignocellulosic structure of the jabuticaba by-product for a further AD process for bioenergy recovery.

Based on the above, the aim of this study was to evaluate the effect of a combined bioprocess based on SWP followed by semi-continuous AD of jabuticaba by-product to recover bioenergy and agricultural fertilizer. The performance of methanogenic reactors was evaluated by operational parameters, biogas production, methane composition, and volatile fatty acids. This study focused on the recovery of bioenergy from biogas, avoided GHG emissions, and the application of digestate after AD in the germination of lettuce. Hence, this study provides scientific information for the application of SWP and AD as alternatives for waste management in a circular economy framework, recovering bioenergy from biogas and biofertilizer from digestate.

2. Materials and methods

2.1. Raw materials and inoculum

The jabuticaba agro-industrial by-product (wet basis) was provided by the company *Maria Preta* (Campinas, SP, Brazil). The mesophilic inoculum was obtained from an upflow anaerobic sludge blanket (UASB) reactor of a poultry slaughterhouse (Dacar Company, Tietê, SP, Brazil). **Table 1** presents the characterization of the raw materials used for SWP and AD.

Table 1. Initial characterization of the raw materials.

Parameters	Jabuticaba by-product	Hydrolysate	Inoculum	Feed	Unit
pH	3.25 ± 0.01	4.17 ± 0.03	7.03 ± 0.13	3.45 ± 0.05	–
Moisture	65.75 ± 0.08	98.98 ± 0.02	94.66 ± 0.09	89.10 ± 0.76	%
Total solids	34.25 ± 0.08	1.02 ± 0.02	5.34 ± 0.09	10.90 ± 0.76	%
Total fixed solid	0.70 ± 0.04	0.03 ± 0.01	0.71 ± 0.01	0.17 ± 0.01	%
Total volatile solid	33.55 ± 0.04	0.99 ± 0.00	4.63 ± 0.08	10.73 ± 0.77	%
Alkalinity	n.d.	n.d.	147.25 ± 3.27	n.d.	mg $CaCO_3$ L^{-1}
Ammonium nitrogen	69.16 ± 0.03	29.26 ± 7.98	15.96 ± 0.04	26.60 ± 5.32	mg N-NH_3 L^{-1}
Soluble chemical oxygen demand	9.69 ± 0.25	1.08 ± 0.01	0.23 ± 0.04	1.65 ± 0.01	g O_2 L^{-1}
Total chemical oxygen demand	13.34 ± 0.32	1.44 ± 0.02	0.44 ± 0.02	2.75 ± 0.09	g O_2 L^{-1}
Soluble proteins	0.013 ± 0.001	0.07 ± 0.002	0.036 ± 0.001	0.034 ± 0.002	g L^{-1}
Total phosphorus	0.012 ± 0.004	0.04 ± 0.003	0.03 ± 0.02	0.02 ± 0.003	g L^{-1}

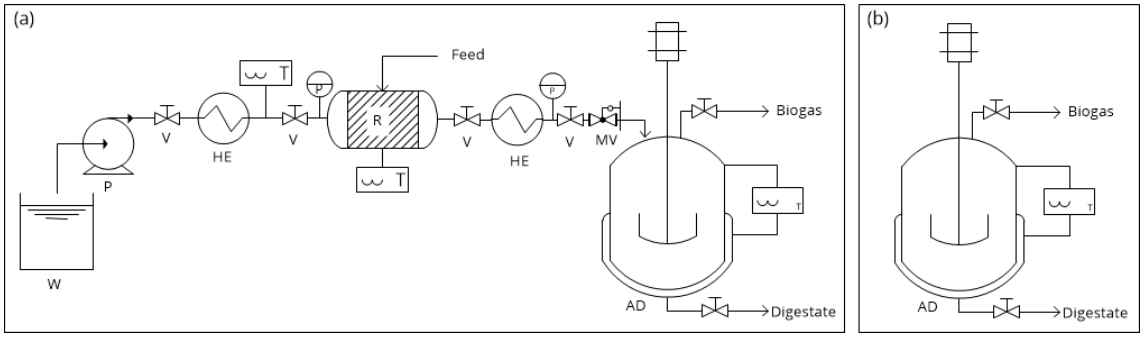
The results are expressed as the mean ± standard deviation. Analysis conducted in triplicate (n=3).

2.2. Subcritical water pretreatment of jabuticaba by-product

The SWP was carried out in a semi-continuous flow-through process **(Fig. 1)**. A hydrolysis reactor with an internal volume of 110 mL was used in the subcritical system. A high-pressure water pump was installed in the system. A preheater and an electric jacket-type heat exchanger (1500 W) insulated by ceramic fiber heated the water flowing into the reactor. The hydrolysis temperature was measured by thermocouples (type K), and the pressure was measured by manometers (0 – 7.500 psi, 0.1 % accuracy). After hydrolysis, the hydrolysate was cooled in a heat exchanger connected to a thermostatic bath. The pressure was adjusted with a micrometer valve.

The operational conditions of SWP were adopted based on previous research [27]. The reactor was fed with jabuticaba by-product (20 g, wet basis) and operated with a hydrolysis temperature of 180 °C, pressure of 15 MPa, and water flow rate of 10 mL min^{-1}. The solvent-to-feed ratio was 22.5 g water g^{-1} jabuticaba by-product. The SWP was conducted for 45 min, and every 5 min, an aliquot of the hydrolysate was collected to perform the hydrolysis kinetics.

Figure 1. Designed bioprocess for subcritical water pretreatment and anaerobic digestion. (a) SWP+AD reactor and (b) AD reactor.



Label: W, water tank; P, high-pressure pump; V, block valves; P, manometer; T, thermocouples; R, subcritical reactor; HE, heat exchanger; MV, micrometric valve; AD, anaerobic digestion reactor.

2.3. Characterization of hydrolysates and solid residues after SWP

2.3.1. pH

A digital pH meter (IonLab, model THS-3E, New York, NY, USA) was used to determine the pH of the hydrolysate during the hydrolysis kinetics. The pH meter was calibrated with buffer solutions before the readings, and the measurements were taken at 25 °C.

2.3.2. Monosaccharides, organic acids, and inhibitors

High-performance liquid chromatography (HPLC) with a refractive index detector (RID) was used to quantify the sugar monomers (monosaccharides), organic acids, and inhibitors. Separation was performed with a Rezex^{TM} column (Phenomenex, model ROA-Organic Acid H+ (8 %), 8 µm, 300 × 7.8 mm, Torrance, CA, USA) with an isocratic flow rate of 0.6 mL min^{-1} of H_2SO_4 (5 mmol L^{-1}) at 60 °C. The RID was maintained at 40 °C. For the HPLC analysis, the hydrolysates were centrifuged (10,000×*g*) and filtered (nylon 0.22 µm). 10 µL of hydrolysate was injected, and the run time was set to 48 min. The concentrations of cellobiose, glucose, fructose, arabinose, formic acid, acetic acid, furfural, and 5-hydroxymethylfurfural (5-HMF) were calculated from the calibration curves of each standard. The analysis was conducted in triplicate, and the results were expressed as g L^{-1}.

2.3.3. Characterization of the solid residue

The solid residue remained in the reactor after SWP was collected and dried (105 °C, 24 h). The remaining solids were quantified by difference considering the initial mass of jabuticaba by-product used in the experiments. The raw biomass and solid residue were analyzed by scanning electron microscopy (SEM) (Tescan Vega 3 microscope) equipped with an energy dispersive X-ray microsound (Penta FET Precision, Oxford Instruments). For SEM analysis, the samples were previous dried (105 ºC, 24) and then fixed to the surface of double-face adhesive tape and coated with a thin gold layer. The visualisation occurred at an excitation voltage of 10 kV.

2.4. Semi-continuous anaerobic digestion of jabuticaba by-product

The AD process was started-up in a 4.3 L stirred tank reactor and operated in semi-continuous mode for 50 days. The following bioprocesses were started-up:

i) AD reactor (control, without pretreatment): substrate composed of 46.3 % wet jabuticaba by-product (1.54 L or 500 g, density of 0.324 g mL^{-1}), 31.87 % inoculum (1.07 L or 874 g, density of 0.812 g mL^{-1}) and 21.83 % water (0.7095 L). The substrate accounted for 3.32 L (77.2 % of the reactor's total volume), with the remaining 0.98 L for headspace (22.8 %). The AD reactor was fed daily with 14.58 g of jabuticaba by-product and 55 mL water.

ii) SWP+AD reactor (process with pretreatment): substrate composed of 52.5 % hydrolysate (1.354 L), 17.5 % wet jabuticaba by-product (0.451 L or 146 g, density of 0.324 g mL^{-1}), and 30 % inoculum (0.774 L or 953.7 g, density of 0.8115 g mL^{-1}). The substrate accounted for 2.58 L (60 % of the reactor's total volume), with the remaining 1.72 L for headspace (40 %). The SWP+AD reactor was fed daily with 14.58 g of jabuticaba by-product and 55 mL water.

The AD and SWP+AD reactors were operated under hydraulic retention times (HRT) of 33.2 and 25.8 days, respectively. The organic load rate (OLR) was 4.32 and 5.57 g O_2 L^{-1} d^{-1} for the AD and SWP+AD reactors, respectively. The volatile solids loading rate (VSR) was 1.47 and 1.89 g TVS L^{-1} d^{-1}, respectively, for the AD and SWP+AD reactors.

The reactors were kept at a mesophilic temperature (36 °C) with a thermostatic bath (Marconi Equipment, model MA184, Piracicaba, SP, Brazil). The pH was maintained between 7 and 8.5 to enable methanogenic processes by adding NaOH (6 mol L^{-1}) to the feed. Mechanical stirrers (Fisatom®, model 715, São Paulo, SP, Brazil) were used for 5 min before sample collection and 5 min after feed to keep the reactors homogenized. The biogas produced in the reactor was collected daily in a Tedlar bag connected to the system (Supelco Analytical, Darmstadt, Germany). The digestate was collected to determine the operational performance.

2.5. Operational performance of the digestate from anaerobic digestion

2.5.1. Physicochemical parameters

The operational performance of the reactors was assessed over 50 days by measuring pH, alkalinity, total nitrogen, ammonia nitrogen, soluble protein, soluble chemical oxygen demand (sCOD), total chemical oxygen demand (tCOD), total solids (TS), total fixed solids (TFS), and total volatile solids (TVS) using the Standard Methods for the Examination of Water and Wastewater [28]. The content of soluble protein was determined according to the method of Bradford [29]. All analyses were performed in triplicate (n=3). The phosphorus content in the digestate was determined according to [30], with modifications. For this, 2.5 g of digestate was solubilized in 25 mL of Mehlich solution (HCl 0.05 mol L^{-1} and H_2SO_4 0.0125 mol L^{-1}) to extract the phosphorous. The solution was stirred for 10 min (125 rpm, 25 °C) and then rested for 24 h before being filtered. The filtered solution (or water as a control) was reacted with 2 mL ammonium molybdate solution containing 300 mg ascorbic acid as a reducing agent. The absorbance was measured in a spectrophotometer at 660 nm after 1 h. The calibration curve was conducted with monopotassium phosphate (KH_2PO_4), and the results were expressed as g phosphorus L^{-1} (g L^{-1}).

2.5.2. Volatile fatty acids (VFA)

The VFA were extracted from the digestate using 5 g of sample in 50 mL of water. The solution was homogenized for 1.0 h (150 rpm, 25 °C) and filtered to remove non-soluble particles. The filtered solution was centrifuged (10.000 × g), and the supernatant was filtered (nylon 0.22 µm). The quantification and separation of VFA were conducted by HPLC-RID according to the method described in **Section 2.3.2**. Concentrations of acetic acid, propionic acid, isobutyric acid, and valeric acid were measured. The VFA was calculated from the

calibration curves of each standard. The analysis was conducted in triplicate, and the results were expressed as g L^{-1}.

2.5.3 X-ray fluorescence (XRF)

The ashes from the TFS analysis of raw jabuticaba by-product and the digestate from AD and SWP+AD reactors (initial and final day) were used to quantify the minerals profile by XRF. Approximately 50 mg of sample was weighed, WAX binder was added, and then the samples were homogenized in a mortar. Another 50 mg of WAX binder was sieved in a plastic cup, after which the mortar containing the sample and the homogenized binder, together with the plastic cup containing the binder, were taken to the hydraulic press (AMEF, model AP-25T) with the tablet of aluminum inserted into the cavity of the press. First, the binder was added, and on top of the binder, the contents of the mortar were added, after which the material was pressed. The tablet with the pressed material was placed in X-ray fluorescence equipment (Panalytical, model Axios 1KW), and then the sample was read. The results are presented as a mass percentage of the elements obtained in the analyses.

2.6. Biogas volume, composition, and methane yield

The biogas produced from the AD and SWP+AD reactors was collected from the Tedlar bag, and the volume was measured daily using a syringe. The volume of biogas was adjusted for standard temperature and pressure conditions (1 atm and 298.15 K). The accumulated biogas volume was calculated by the daily biogas produced.

For the quantification of biogas composition, a gas chromatograph (GC) with a thermal conductivity detector (TCD) (Shimadzu®, model GC 2014, Kyoto, Japan) was used. Approximately 0.5 mL of biogas was collected from the reactor's headspace and injected into the GC-TDC. A micropacked column (length of 6 m and internal diameter of 3 mm)

(ShinCarbon, ST 50/80 mesh) was used to determine the composition of oxygen (O_2), hydrogen (H_2), methane (CH_4), and carbon dioxide (CO_2). The following chromatographic conditions were employed: injection port and detector temperatures were set to 200 °C; the GC column temperature was initially set to 50 °C (held for 3 min) and then increased by 5 °C min^{-1} to 180 °C (held for 5 min); and N_2 was used as the carrier gas (35 mL min^{-1}, 5 bar). The quantification was determined by the relative area of each compound.

The experimental methane yield was determined according to **Eq. 1**.

$$Methane\ yield\left(\frac{L\ CH_4}{kg\ TVS_{added}}\right) = \frac{V_{biogas} \times CH_4}{TVS} \qquad (1)$$

where V_{biogas} is the accumulated volume of biogas (L), CH_4 is the percentage of methane in the biogas (%), and TVS is the content of volatile solids added in the reactor.

2.7. Potential for bioenergy recovery and avoided GHG emissions

The potential for electrical and thermal energy recovery from the methane-rich biogas was assessed assuming that the biogas is burning in a cogenerator, according to **Eqs. 2** and **3**.

$$Electricity\ \left(\frac{MWh}{ton}\right) = \frac{Q_{biogas} \times\ LCV_{CH_4}\ \times\ C_m\ \times\ \eta_e\ \times CF}{M_{Jabuticaba}} \qquad (2)$$

$$Heat\ \left(\frac{MJ}{ton}\right) = \frac{Q_{biogas} \times\ LCV_{CH_4}\ \times\ C_m\ \times\ \eta_e}{M_{Jabuticaba}} \qquad (3)$$

where $M_{Jabuticaba}$ is the mass of jabuticaba by-product used during the 50 days of the experiment; Q_{biogas} is the volume of biogas produced during the 50 days of AD (m^3 of biogas); LCV_{CH_4} is the lower calorific value of methane (35.59 MJ m^{-3}); C_m is the percentage of methane in biogas (%); η_e is the engine efficiency (%), assumed to be 40 % for electric energy and 50 % for thermal energy; and CF is the conversion factor from MJ to MWh (1 MWh = 3600 MJ).

GHG emissions are avoided when electricity from the national grid is replaced with electricity from a local renewable source (e.g., from methane). A similar approach can quantify

the avoided GHG from heat, where the biogas produced can replace natural gas (non-renewable fuel) in boilers. The quantification of avoided GHG emissions was calculated according to **Eqs. 4** and **5**.

$$Avoided\ GHG_{electricity} = EF_{CO_2-Electricity} \times Electricity \qquad (4)$$

$$Avoided\ GHG_{heat} = EF_{CO_2-Heat} \times Heat \qquad (5)$$

where $EF_{CO_2-Electricity}$ is the emission factor of CO_{2eq} for the 2019 Brazilian national electric energy generation, assumed to be 0.075 t CO_{2eq} MWh^{-1} [31], and EF_{CO_2-Heat} is the emission factor of heat energy (0.056 tCO_{2eq} GJ^{-1}), assuming a replacement of natural gas for biogas in the boiler [32].

2.8. Global energy balance of the bioprocesses

The global energy balance of the AD and SWP+AD processes was evaluated based on the experimental results obtained in this study. The energy balance was utilized to determine and quantify the amount of energy consumed, accumulated, transformed into another form, and lost during the process [33]. For the SWP, the first rule of thermodynamics was used to achieve energy balance, considering a steady state with constant pressure and no shaft work [34]. The variations in kinetic and potential energy were not considered. During SWP, mass (M) is constant in the process, and the heat required in the subcritical reactor (Q) is the difference in enthalpy (H) (**Eq. 6**)**.** Furthermore, the enthalpy can be determined using the mixture's specific heat in the reactor. It was able to use the specific heat of water (Cp*), which is 4.178 kJ kg^{-1} K^{-1} at 25 °C, after considering the mass of water greater mass of jabuticaba by-product [33]. A pressure pump was employed to regulate the pressure during hydrolysis, and it was assumed to be constant in the current operation. In addition, the subcritical reactor keeps the pressure

constant over time while raising the water temperature from 25 to 180 °C. Thus, enthalpy was calculated based on **Eq. 7**.

$$\frac{Q}{M} = H_2 - H_1 \qquad (6)$$

$$H\left(\frac{kJ}{kg}\right) = C_p^*\left(\frac{kJ}{kg \cdot K}\right) \times T(K) \qquad (7)$$

2.9 Application of digestate for the germination of *Lactuca sativa*

A germination experiment was carried out with the digestate obtained from the AD and SWP+AD reactors after 50 days. The phytotoxicity test identifies the agronomic quality of digestate for use as an agricultural substrate [35,36]. For the germination test, the digestate was diluted to different concentrations in deonized water (0.1, 0.5, and 1 g L^{-1}) followed by homogenization (150 rpm, 30 min, 25 °C). The homogenized solution was vacuum filtered using qualitative filter paper, and the fileted solution was used for the germination experiment. Briefly, 10 seeds of iceberg lettuce (*Lactuca sativa*) were placed in 11 cm Petri dishes containing 5 mL of the different digestate solutions soaked in filter paper. The control was conducted with deionized water. The seeds were incubated in the dark for 7 days at 20 °C, which is the optimal germination temperature for Iceberg lettuce [37]. Tests were conducted in five repetitions.

The number of germinated seeds and the length of the roots were measured to determine the germination index (**Eq. 8**) [38] and the inhibition percentage (**Eq. 9**).

$$Germination\ Index\ (\%) = \frac{NGS_{Digestate} \times ARL_{Digestate}}{NGS_{Control} \times ARL_{Control}} \times 100 \qquad (8)$$

$$Germination\ Inhibition\ (\%) = \frac{NGS_{Control} - NGS_{Digestate}}{NGS_{Control}} \times 100 \qquad (9)$$

where $NGS_{Digestate}$ is the number of germinated seeds in the experiment with the digestate; $NGS_{Control}$ is the number of germinated seeds in the control experiment (with water); $ARL_{Digestate}$

is the average length of roots in the experiment with the digestate; and $ARL_{Control}$ is the average length of roots in the control experiment (with water).

2.10 Statistical analysis

All the data were evaluated in triplicate (n = 3), and the results are expressed as the mean ± standard deviation. The data were statistically analyzed using one-way analysis of variance (ANOVA), and the difference between the averages was validated using Tukey's test ($p \leq 0.05$) (Statistica® version 10.0, StatSoft Inc., Tulsa, OK, USA).

3. Results and discussion

3.1. Subcritical water pretreatment of jabuticaba by-product

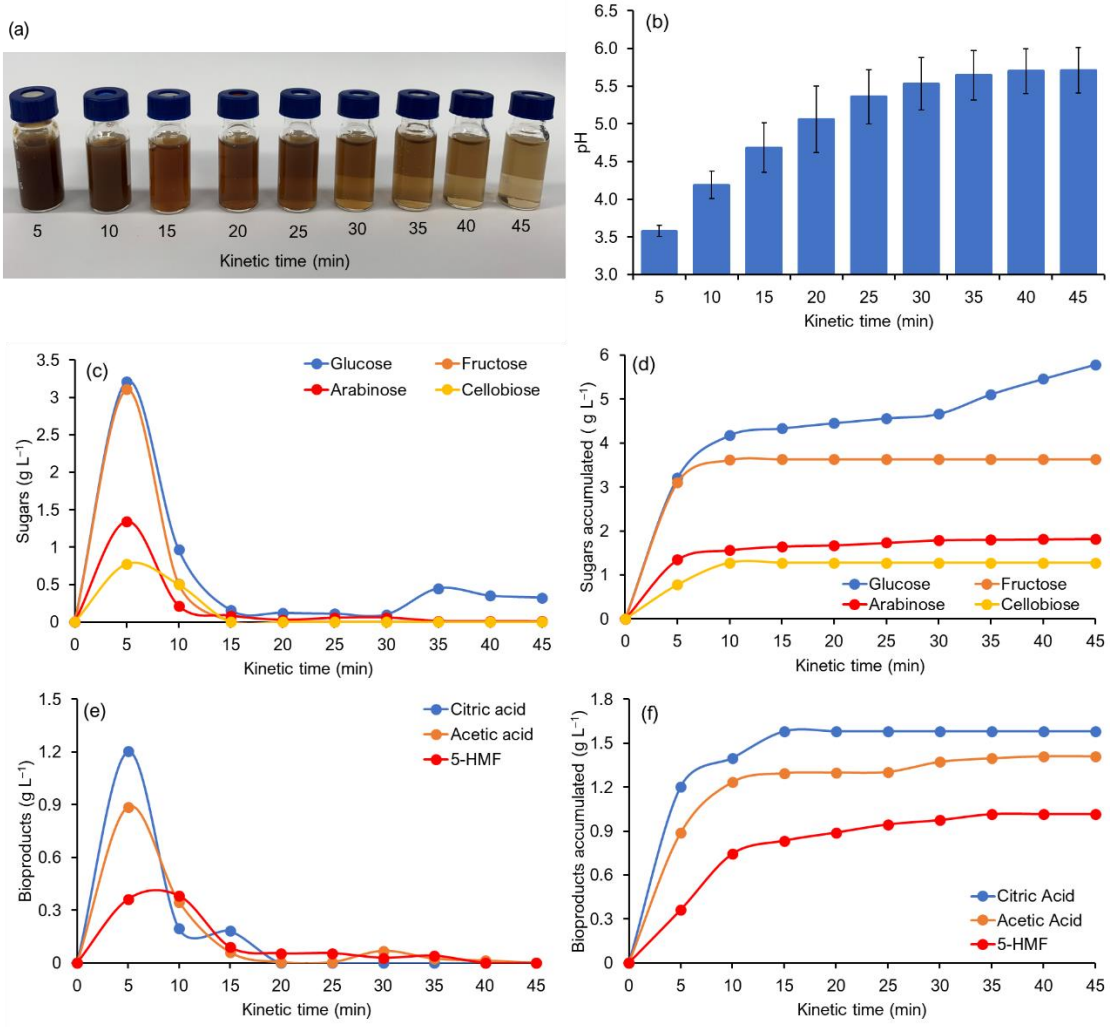
Fig. 2 shows the kinetic profile of pH, sugar, and bioproducts evolution during the SWP of the jabuticaba by-product. From the visual appearance of the hydrolysate (**Fig. 2a**), it was observed that the first points of the hydrolysis kinetics resulted in a hydrolysate with a more concentrated color. The pH of the hydrolysate was acidic, ranging between 4.69 and 5.71 **(Fig. 2b)**. The pH values are within those found in the literature where it undergoes a slight increase during the hydrolysis kinetics [39], with similar behavior to a previous study on the hydrolysis of jabuticaba peels at different temperatures [27].

The release of sugars is the expected phenomenon from the SWP, since high temperature and pressure can promote the degradation of lignocellulose into monosaccharides. The hydrolysate obtained had a high amount of monosaccharides released during the kinetics (**Fig. 2c** and **2d**). At the end of the pretreatment, the hydrolysate was composed of glucose (5.78 g L^{-1}), fructose (3.63 g L^{-1}), arabinose (1.82 g L^{-1}), and cellobiose (1.28 g L^{-1}). Glucose was the primary monosaccharide, as it comes from the hydrolysis of hydrolytically accessible cellulose and hemicellulose [40,41]. The sugar composition obtained in this study was similar to a

previous study on the hydrothermal pretreatment of jabuticaba by-product, where glucose, fructose, arabinose, and cellobiose were the monosaccharides obtained [27]. Moreover, using subcritical water technology, some studies obtained glucose as the major monosaccharide using orange peel [42] and sugarcane bagasse [43] as feedstocks.

The formation of bioproducts (organic acids and inhibitors) can occur during the SWP of lignocellulosic biomass (**Fig. 2e** and **2f**). In this study, citric acid (1.58 g L^{-1}) and acetic acid (1.99 g L^{-1}) were the organic acids obtained. The formation of organic acids occurs due to hydrolysis being carried out at high temperatures, which causes the decomposition of glucose and fructose into organic acids [44]. In addition, the jabuticaba by-product presents a high concentration of organic acids (1.38 - 1.48 g g^{-1}) in its composition [45], and during SWP, these compounds can be released into the hydrolysate. Additionally, a low concentration of 5-HMF (1.02 g L^{-1}) was obtained during SWP of the jabuticaba by-product. 5-HMF is an inhibitor of fermentation processes [46]. The formation of 5-HMF occurs due to the high temperature of hydrolysis, and it is usually formed by the dehydration of the six-carbon sugars formed by the degradation of cellulose [47–49].

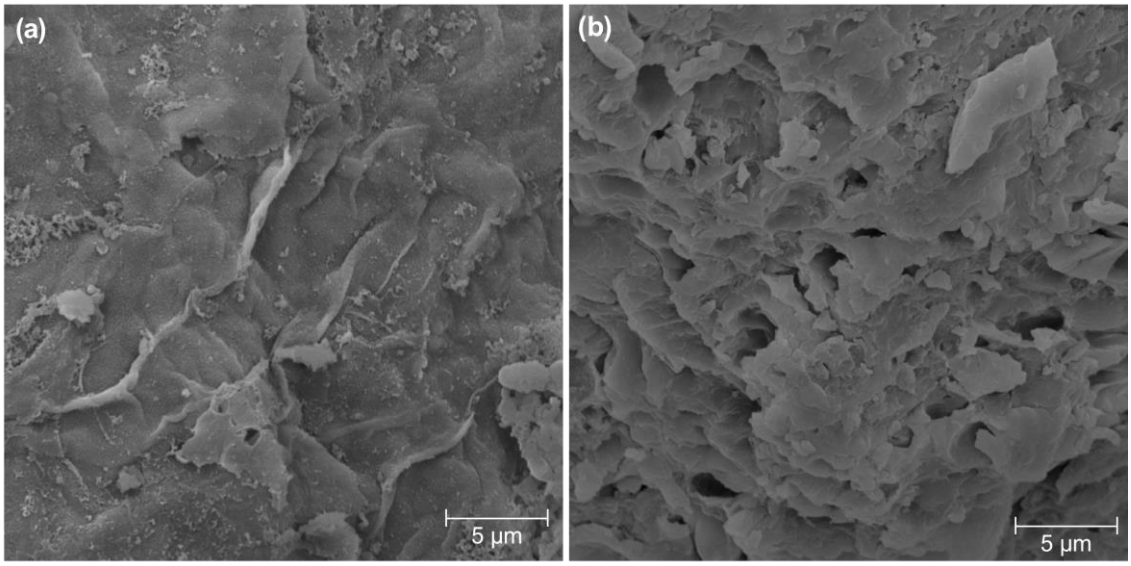
Figure 2. Kinetic profile during the SWP of jabuticaba by-product. (a) Visual appearance of the hydrolysate. (b) pH. (c) Sugars (non-accumulated). (d) Sugar (accumulated). (e); bioproducts (non-accumulated). (f) bioproducts (accumulated).



The residual solids that remained in the reactor after pretreatment was measured and characterized by SEM. In this study, the initial biomass loaded in the reactor was reduced at 83.83 ± 2.05 %. That is, the initial dry mass of 6.7 g (equivalent to 20 g in wet basis) of jabuticaba by-product was reduced to 1.08 ± 0.11 g (dry mass). This fact demonstrates that SWP acts in the biomass and converts the cellulose, hemicellulose, and lignin into a hydrolysate. From the SEM analysis of the raw biomass (**Fig. 3a**) and the solid residue after SWP (**Fig. 3b**), it was possible no observe that the raw biomass presented an uniform structure with a flat surface. After the SWP, the remained biomass presented some pores, which can be

an indicative of changes in the physical structure of the biomass, suggesting that the SWP breakdown the lignocellulosic structure.

Figure 3. Scanning electron microscopy of the (a) raw jabuticaba by-product and (b) solid residue after SWP.



Finally, the pretreatment of jabuticaba with subcritical water technology can be an alternative to produce a hydrolysate containing high concentrations of sugars, especially glucose and fructose. The hydrolysate showed a low concentration of organic acids and 5-HMF, demonstrating that the hydrolysate can be used for anaerobic fermentative processes.

3.2. Characterization of raw materials

The initial characterization of the raw materials is summarized in **Table 1**. The pH value deserves special attention, as it plays an essential role in AD. The pH of the jabuticaba by-product (3.25 ± 0.01) and the hydrolysate obtained from SWP (4.17 ± 0.03) were acids. When applying these feedstocks in AD, the pH should be adjusted to the ideal range to promote

methanogenic reactions [50]. Concerning TVS, the raw jabuticaba by-product presented 33.55 ± 0.04 %, and after SWP, the hydrolysate presented a removal yield of 97 % of TVS. In addition, no alkalinity was detected in the raw material, hydrolysate, and feed. The SWP reduced the ammonia nitrogen, soluble proteins, sCOD, tCOD, and phosphorus of the jabuticaba by-product. The high sCOD (9.69 g O_2 L^{-1}) and tCOD (13.34 g O_2 L^{-1}) of the jabuticaba by-product demonstrate that this feedstock may be suitable for AD since methanogenic microorganisms consume organic matter and produce methane. From the characterization of raw materials, it can be observed that the pretreatment effectively converted the jabuticaba by-product into smaller organic molecules, making it possible to apply this hydrolysate into biotechnological processes for bioenergy recovery.

3.3. Operational performance of AD reactors

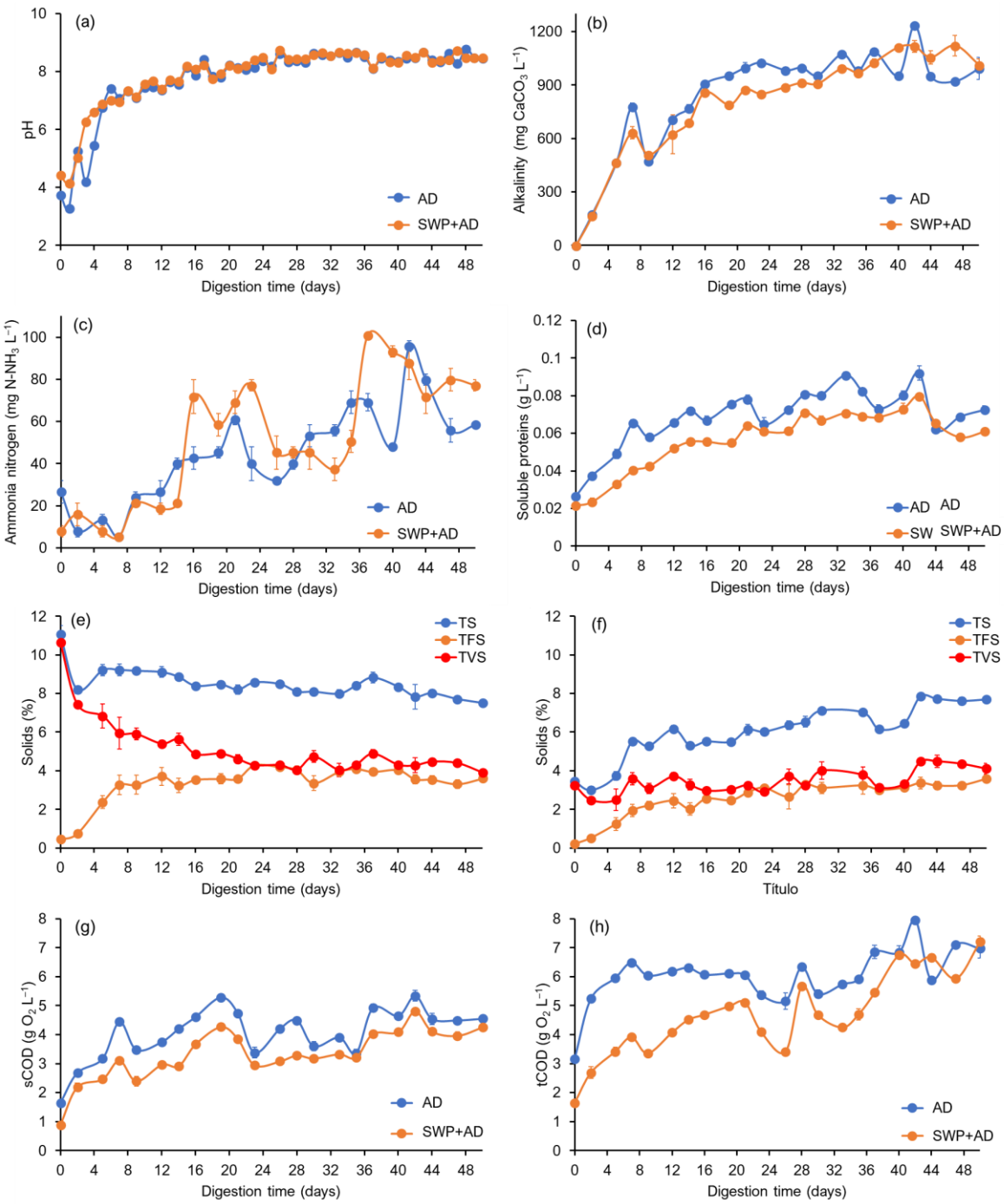
The effectiveness of the jabuticaba by-product with and without pretreatment on operating parameters, biogas generation, and bioenergy recovery was evaluated by characterization of AD and SWP+AD reactors. **Table 2** shows the reactor characterization on the initial and final days of AD, enabling an overview of the impact of SWP on AD performance. **Fig. 4** shows the changes in pH, alkalinity, ammonia nitrogen, solids, and COD during the semi-continuous AD. A deep discussion was conducted of each operational parameter to observe the effect on biogas production and digestate quality.

Table 2. General parameters recorded during the semi-continuous AD and SWP+AD of jabuticaba by-product.

Parameters	AD reactor		SWP+AD reactor		Unit
	Day 0	Day 50	Day 0	Day 50	
pH	3.75 ± 0.09^{b}	8.47 ± 0.12^{a}	4.43 ± 0.05^{B}	8.48 ± 0.21^{A}	–
Total solids	11.08 ± 1.30^{a}	7.5 ± 0.06^{b}	3.46 ± 0.35^{B}	7.69 ± 0.03^{A}	%
Total fixed solid	0.45 ± 0.03^{b}	3.6 ± 0.03^{a}	0.21 ± 0.06^{B}	3.58 ± 0.03^{A}	%
Total volatile solid	10.63 ± 1.33^{a}	3.9 ± 0.09^{b}	3.25 ± 0.40^{B}	4.11 ± 0.00^{A}	%
Alkalinity	n.d.	992.75 ± 19.00	n.d.	1011.75 ± 23.75	mg $CaCO_3$ L^{-1}
Ammonium nitrogen	26.6 ± 1.28^{b}	58.52 ± 5.32^{a}	7.98 ± 2.66^{B}	77.14 ± 10.64^{A}	mg NH_3 L^{-1}
Soluble chemical oxygen demand	1.64 ± 0.47^{a}	4.55 ± 0.00^{b}	0.89 ± 0.13^{B}	4.26 ± 0.12^{A}	g O_2 L^{-1}
Total chemical oxygen demand	3.16 ± 0.04^{a}	6.97 ± 0.01^{b}	1.64 ± 0.07^{B}	7.21 ± 0.09^{A}	g O_2 L^{-1}
Soluble proteins	66.03 ± 0.26^{a}	28.78 ± 0.09^{b}	23.93 ± 0.45^{B}	27.35 ± 0.54^{A}	g L^{-1}
Total phosphorus	0.05 ± 0.01^{a}	0.02 ± 0.01^{a}	0.04 ± 0.01^{B}	0.01 ± 0.01^{A}	g L^{-1}

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

Figure 4. Operational parameters during the semi-continuous AD and SWP+AD of jabuticaba by-product. (a) pH. (b) Alkalinity (mg $CaCO_3$ L^{-1}). (c) Ammonia nitrogen (mg N-NH_3 L^{-1}). (d) Soluble proteins (g L^{-1}). (e) Solids (%) for the AD reactor. (f) Solids (%) for the SWP+AD reactor. (g) Soluble chemical oxygen demand (g O_2 L^{-1}). (h) Total chemical oxygen demand (g O_2 L^{-1}).



3.3.1. pH and alkalinity

The pH in a solution demonstrates the concentration of protons (H^+) in the reactor. For biotechnological processes, most microorganisms prefer a neutral pH range [51]. pH is one of the most important parameters influencing organic hydrolysis and acidogenesis [52], affecting many aspects of AD, such as the microbial community and metabolic pathways [53]. **Fig. 4a** shows the pH values of the AD and SWP+AD reactors during 50 days of digestion. The two reactors had very similar behavior. Both oscillated in the first days of AD due to the predominance of hydrolysis and acidogenesis phases. During the hydrolysis phase, the enzymes of hydrolytic bacteria convert carbohydrates, proteins, and lipids into sugars, amino acids, and fatty acids, respectively. These compounds are transformed into VFA during the acidogenic phase, where there is formation and accumulation of organic acids, resulting in a drop in pH [54].

From day 0 until day 14, the pH ranged between 3.75 and 7.57 in the AD reactor. In contrast, for the SWP+AD reactor, the pH ranged between 4.43 and 7.68, which favors the hydrolysis of lignocellulosic compounds from the jabuticaba by-product [55]. From 15 days of AD, the methanogenesis phase was predominant in both reactors, as the pH ranged from 7.5 to 8.7. To maintain the pH values around the optimal range for methane production, 369 mL of NaOH (6 mol L^{-1}) was needed for the AD reactor, and 328 mL of NaOH (6 mol L^{-1}) was needed for the SWP+AD reactor over 50 days of the experiment in each reactor.

Alkalinity demonstrates the ability of a system to neutralize weak acids, and in AD, this is associated with the buffering capacity in this system. Alkalinity is necessary to maintain a stable pH in the digester to achieve optimal biological activity [56]. **Fig. 4b** demonstrates that alkalinity increased for both reactors (AD and SWP+AD). At the beginning of AD, no alkalinity was detected in the reactors. In the subsequent days of the experiment, there was an almost equal increase in both reactors. Nevertheless, from 12 days on, the alkalinity of the AD reactor

was slightly higher than the alkalinity of the SWP+AD reactor, and from 40 days on, the SWP+AD reactor showed higher alkalinity. On the last day of digestion, the alkalinity was 992.75 mg CaCO3 L^{-1} in the AD reactor and 1011.75 mg $CaCO_3$ L^{-1} in the SWP+AD reactor.

The increase in alkalinity can be explained by the formation of carbonates, bicarbonates, methane, and carbon dioxide [57]. The considerably favorable alkalinity for AD varies from 1000 to 5000 mg $CaCO_3$ L^{-1}, where in this range, the alkalinity has a positive effect on methane production, as the pollutant removal process is accelerated and the buffering capacity increases without inhibiting methanogenesis reactions [56]. In this experiment, the ideal alkalinity range was reached by the AD reactor on day 33 with 1073.5 mg $CaCO_3$ L^{-1}, while the SWP+AD reactor only reached the range on day 37 with 1026 mg $CaCO_3$ L^{-1}. This delay can be explained by the acidic pH of the raw material, which made it difficult to increase the alkalinity.

3.3.2. Ammonia nitrogen and soluble proteins

Fig. 4c presents the results for ammonia nitrogen. The end product of anaerobic fermentation of proteins, urea, and nucleic acids is ammonia, which can be present in the free ammonia form (NH_3) or ammonium (NH_4^+) [50,58]. Ammonia is essential in AD, as it influences microbial growth. Although necessary for AD, excess ammonia can inhibit methanogenesis [59].

In the experiments, the reactors showed regular ammonia nitrogen contents and did not show inhibitory concentrations. This increase is associated with the degradation of nitrogen compounds present in the jabuticaba by-product during hydrolysis. Ammonia nitrogen at the beginning and end of the AD reactor ranged from 26.6 to 58.52 mg N-NH_3 L^{-1}, while SWP+AD ranged from 7.98 to 77.14 mg N-NH_3 L^{-1}. Concentrations below 500 mg N-NH_3 L^{-1} can lead to loss of biomass and, consequently, a reduction in biogas production due to a lack of nitrogenous

nutrients [60]. In both reactors, the ammonia nitrogen values were below and an essential factor in biogas production in both systems.

Proteins in organic matter are converted into soluble forms during AD. From the results of soluble proteins (**Fig. 4d),** it was possible to observe an increase in the concentration of soluble proteins in the digestate. The results showed that the total proteins were converted into soluble proteins. Several groups of microorganisms participate in the degradation of different types of proteins. The proteins in AD are important because there is a high correlation between biogas production and protein degradation [61].

3.3.3. Phosphorus

Phosphorus plays a vital role in ecosystems and is an irreplaceable element for agriculture. The major problem is that phosphorus is a finite and scarce resource [62,63]. The phosphorus obtained in the digestate can be used in agriculture as a fertilizer. Phosphorus is a very important nutrient for the physiological and biochemical processes of plants and is one of the most important nutrients for carrying out the photosynthesis process [64].

In this study, both reactors showed a decrease in phosphorus content during AD. The AD reactor had a phosphorus content that ranged from 0.054 g L^{-1} on day 0 to 0.018 g L^{-1} on day 50, while the SWP+AD reactor ranged from 0.04 g L^{-1} (day 0) to 0.12 g L^{-1} (day 50). The decrease in phosphorus values can be explained by the fact that the microbiota used the amount of available phosphorus, since phosphorus is an essential nutrient for living cells and is important for energy metabolism by adenosine triphosphate and for the constitution of deoxyribonucleic and ribonucleic acids [65]. Finally, the digestate obtained is rich in bioavailable nutrients, one of which is phosphorus, which makes digestate an excellent alternative for agricultural use as a fertilizer for plants [66].

3.3.4. Solids

The TS, TVS, and TFS evolution during AD can be seen in **Fig. 4e** (AD reactor) and **Fig. 4f** (SWP+AD reactor). Initially, the AD reactor presented a TVS of 10.63 %. The TVS decreased significantly in the initial days of digestion, reaching 5.95 % on the 7th day of AD. This decrease can be associated with the high microbial activity in the early stages of the process, where bacteria hydrolyze complex materials, reducing organic matter in the system [58]. The TVS was practically constant from day 16 until the end of the experiment, with an average of 4.4 %. The AD reactor removed 63.4 % of TVS, showing that the AD reactor is advantageous in removing organic matter with untreated jabuticaba by-product and can be an alternative for the adequate management of this agro-industrial by-product.

The SWP+AD reactor was started with a TVS content of 3.25 %. Unlike the AD reactor, which showed a decrease in the TVS value, the SWP+AD reactor showed a small increase in the value. At the end of digestion, the SWP+AD reactor had a content of 4.11 % TVS. This increase was due to the solid feed used in the system, in which a VSR of 1.95 g TVS L^{-1} d^{-1} was used. This feed was used for nutrient supplementation for the methanogenic microbiota.

3.3.5. Chemical oxygen demand

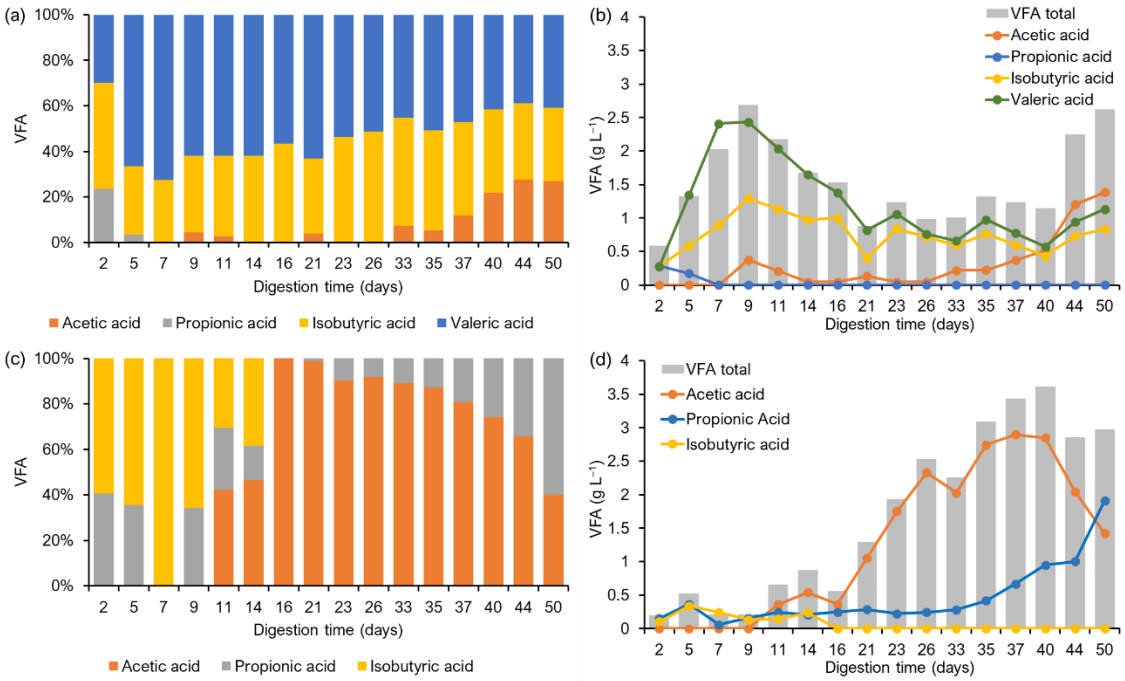
The chemical oxygen demand is one of the most important parameters to verify the efficiency of the AD process regarding the biodegradation of organic matter and methane production [1]. COD also provides the amount of oxygen needed to completely oxidize the organic content of the digestate [50]. In this study, the reactors were evaluated for sCOD (**Fig. 4g**) and tCOD (**Fig. 4h**). The sCOD and tCOD values increased during digestion, which can be associated with the OLR (3.71 g O_2 L^{-1} d^{-1} for the AD reactor and 4.93 g O_2 L^{-1} d^{-1} for the SWP+AD reactor) applied to the feed. The AD reactor started with a sCOD value of 1.64 g O_2 L^{-1} and a tCOD of 3.15 g O_2 $L^{-1,}$ and at the end of the digestion, the values were 4.55 and 6.97 g O_2 L^{-1}, respectively. For the SWP+AD reactor on day 0, the sCOD value was 0.88 g O_2 $L^{-1,}$

and tCOD was 1.64 g O_2 L^{-1}, a lower value than the AD reactor. The results showed lower COD in the SWP+AD reactor on day 50 of digestion. The sCOD and tCOD were 4.25 and 7.20 g O_2 L^{-1}, respectively. There was an increase of 2.8- (AD reactor) and 4.8-fold higher (SWP+AD reactor) in the sCOD, while for the tCOD the increase was 2.2- (AD reactor) and 4.4-fold higher (SWP+AD reactor).

3.3.6. Volatile fatty acids

Fig. 5 shows the production of VFA (acetic, propionic, isobutyric, and valeric acids) during the AD of jabuticaba by-products. VFA are the main intermediate metabolites of the anaerobic process. VFA play an important role in methane production, which is formed due to the conversion of VFA by bacteria during the methanogenic phase [67]. VFA are influenced by some environmental conditions, including pH, organic loading rate, and retention time. Nevertheless, it is worth mentioning that the production of specific VFA depends not only on pH but also on the type of substrate [68]. Although the production of VFA is not solely dependent on pH, it is a key factor in controlling the production of VFA in the acidification process [52].

Figure 5. Production of volatile fatty acids during the semi-continuous AD and SWP+AD of jabuticaba by-product. (a) The percentage of VFA in the AD reactor (%). (b) The concentration of VFA in the AD reactor (g L^{-1}). (c) The percentage of VFA in the SWP+AD reactor (%). (d) The concentration of VFA in the SWP+AD reactor (g L^{-1}).



In the AD reactor **(Fig. 5 a-c),** valeric acid was the most produced VFA, with an average production of 0.7 g L^{-1}, followed by isobutyric acid, with an average of 0.5 g L^{-1}. Although acetic acid is one of the significant VFA in AD processes [52], in this reactor, it started to be produced mainly from the 40th day of digestion. The jabuticaba by-product may have hindered the microbial community from forming acetic acid until the 40th starting day. For the SWP+AD reactor **(Fig. 5 b-d),** acetic acid was the most produced VFA, with an average of 1.27 g L^{-1}, followed by propionic acid (0.37 g L^{-1}). The production of acetic acid tends to increase with increasing pH [52]. In this study, the pH was controlled between 7 and 8.5, which favored a better production of acetic acid during digestion. Acetic and propionic acids were the main products found in alkaline conditions with mixed culture fermentation [69], corroborating the results of the present study.

Traditionally, VFA are produced from petroleum-based sources. Although high-yield and fast-producing, the production of VFA from non-renewable sources and technologies will end up being hampered due to overexploitation and the depletion of fossil resources [70,71]. In a biorefinery concept, some studies evaluated the possibility of using AD technology for the recovery of VFA, being an additional product when compared with the standard process that generates only biogas [72]. For the recovery of VFA, nanofiltration, reverse osmosis, pervaporation, membrane contactors, and membrane distillation are the available technologies for purification and isolation from the digestate [71].

3.3.7 X-ray fluorescence

Table 3 presents the results of the mineral composition of the jabuticaba by-product and the digestate of the AD and SWP+AD reactors. The jabuticaba by-product presented K_2O as the major component, with 60.47 %, followed by P_2O_5 (13.36 %), SO_3 (7.02 %), MgO (6.75 %), and CaO (4.04 %). These compounds represent more than 90 % of the chemical composition of the jabuticaba by-product.

In the beginning of AD, the digestate presented in its chemical composition the K_2O as the major component with 22.90 %, which was already expected since a large concentration of jabuticaba by-products was placed in the reactor. SiO_2 (14.41 %) was the second most abundant compound, followed by SO_3 (14.09 %), P_2O_5 (13.56 %), and Fe_2O_3 (11.93 %), and these compounds correspond to more than 75 % of the chemical composition of the digestate from the AD reactor. For the SWP+AD reactor, the digestate composition on day 0 had SiO_2 (18.95 %). Unlike the AD reactor, the SWP+AD reactor had a small amount of the jabuticaba by-product, so the digestate did not have a large amount of K_2O. The other components were SO_3 (14.90 %), Na_2O (12.54 %), Fe_2O_3 (11.77 %), K_2O (9.97 %) and CaO (7.27 %). These elements

correspond to more than 75 % of the composition of the initial digestate of the SWP+AD reactor.

On the last day of AD (day 50), the digestate from both reactors showed a very similar chemical composition, indicating that the microorganisms acted very similarly in the two reactors at the end of the experiment due to the solid feed. The majority composition of the digestate on day 50 was Na_2O, with 80.32 % for the AD reactor and 81.53 % for the SWP+AD reactor. The second compound with the highest amount was K_2O with 7.63 % and 7.46 %, respectively, for the AD and SWP+AD reactors. The change in the chemical composition of the digestate during AD can be explained by the fact that during digestion, a series of biochemical reactions occur, where microorganisms breakdown organic matter from the substrate [51]. In addition, because it is a biological process with several stages and involves different microorganisms, the use of different trace elements by the microorganisms is expected so that the reactor operates stably [73].

Table 3. Chemical composition recorded during the semi-continuous AD and SWP+AD of jabuticaba by-product.

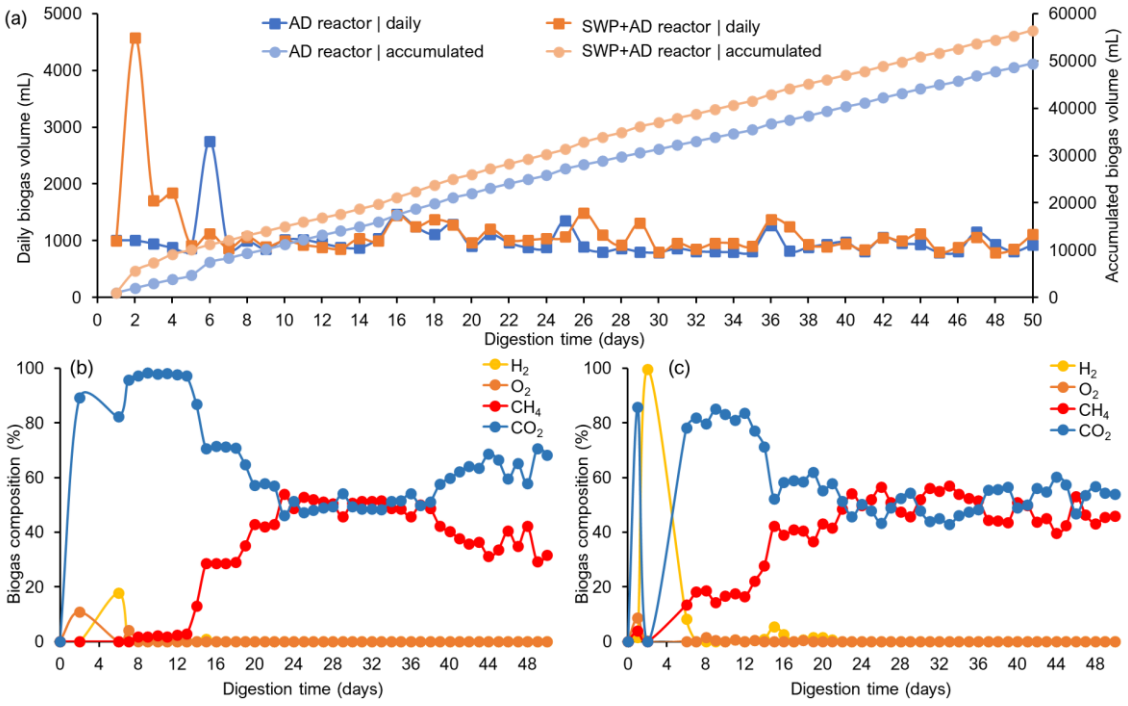
Parameters	Jabuticaba by-product	AD reactor		SWP+AD reactor		Unit
		Day 0	Day 50	Day 0	Day 50	
CaO	4.04	7.72	1.45	7.27	1.17	%
Cl	0.24	0.52	0.13	0.12	0.16	%
CO_3O_4	n.d.	0.02	n.d.	n.d.	0.01	%
Cr_2O_3	n.d.	0.04	0.02	0.04	n.d.	%
CuO	0.06	0.41	0.05	0.36	0.05	%
Fe_2O_3	0.31	11.93	1.74	11.77	1.41	%
K_2O	60.44	22.90	7.63	9.97	7.46	%
MgO	6.75	4.79	1.29	3.29	1.24	%
MnO	0.03	0.11	0.01	0.09	0.02	%
MoO_3	n.d.	0.02	n.d.	0.36	n.d.	%
Na_2O	3.59	n.d.	80.32	12.54	81.53	%
Nd_2O_3	n.d.	n.d.	n.d.	n.d.	0.02	%
NiO	n.d.	0.02	0.01	0.03	n.d.	%
P_2O_5	13.36	13.56	2.38	10.14	2.21	%
Rb_2O	0.10	0.03	0.02	n.d.	0.01	%
SO_3	7.02	14.09	2.12	14.90	1.82	%
SiO_2	0.24	14.41	1.41	18.95	1.53	%
SrO	n.d.	0.01	n.d.	n.d.	n.d.	%
TiO_2	n.d.	0.40	0.04	0.41	0.04	%
Yb_2O_3	0.06	n.d.	n.d.	n.d.	n.d.	%
ZnO	3.51	7.61	1.13	8.45	1.21	%
ZrO_2	0.19	1.32	0.18	1.32	0.13	%

Label: n.d., not detected.

3.4. Production of methane in the anaerobic digestion process

In **Fig. 6,** it is possible to observe the daily and accumulated volume and the biogas composition in the AD and SWP+AD reactors. The daily production of biogas and its composition suffered some oscillations during the AD process. The oscillations of methane production are expected until the stabilization of the reactor because each digestion period is carried out by a different group of microorganisms [74].

Figure 6. Production of methane-rich biogas during the semi-continuous AD and SWP+AD of jabuticaba by-product. (a) Volume of biogas produced (daily and accumulated). (b) Biogas composition (AD reactor). (c) Biogas composition (SWP+AD reactor).



The accumulated biogas production **(Fig. 6a)** was higher in the SWP+AD reactor, with a total production of 56.5 L, while the AD reactor produced 49.6 L. This result demonstrates that the reactor with the pretreatment was more efficient than the reactor without pretreatment for biogas production. The biogas production in the SWP+AD reactor was 13.9 % higher than

that in the AD reactor. Regarding the biogas composition, the AD reactor **(Fig. 6b)** had an initial composition of O_2 (10.82 %) and CO_2 (89.18 %), whereas the SWP+AD reactor **(Fig. 6c)** had a composition of H_2 (1.43 %), O_2 (8.75 %), CH_4 (3.98 %) and CO_2 (85.84 %). Methane production started first in the SWP+AD reactor, with production on day 1. In contrast, methane production in the AD reactor started only on day 7, showing that pretreatment accelerated the start-up of methane production. The highest CH_4 peak for the AD reactor (53.91 %) occurred on day 23, while for the SWP+AD reactor, the highest CH_4 peak was on day 33 (57.01 %). The SWP+AD reactor presented a more stable methane content in the biogas than the AD reactor, demonstrating that the SWP is positive for the solubilization of the biomass components and increasing the production of methane-rich biogas. Some factors that affect the methane composition in biogas, such as OLR, HRT, temperature, pH, substrate composition, particle size, and feed material consistency, are parameters that deserve special attention and must be monitored to produce a stable content of methane in biogas [75–77].

The methane yield for the AD reactor was 42.31 L CH_4 kg^{-1} TVS, while the AD reactor had a much higher yield of 239.04 L CH_4 kg^{-1} TVS. The methane yield increased 5.64-fold higher for the SWP+AD reactor. In the literature, this is the first study on the AD of jabuticaba by-product. Comparing with other feedstocks, macaúba peel (590 L CH_4 kg^{-1} TVS) and açaí processing residue (791.81 L CH_4 kg^{-1} TVS) that received SWP also obtained high methane yields when compared to reactors without pretreatment [78,79]. Finally, the bioprocess designed by combining SWP and AD can be considered an excellent and promising technology for biomass treatment to produce methane-rich biogas.

3.5. Bioenergy potential and avoided GHG emissions

AD is a promising technology to produce methane-rich biogas in the context of energy demand and the circular economy. Compared to other technologies, such as incineration, gasification, and pyrolysis, AD causes less air and solid waste pollution [80]. AD can be used to reduce the consumption of fossil fuels by generating energy through methane, resulting in a decrease in GHG emissions [81]. The biogas produced can occur in different ways, such as fuels for vehicular use from the use of biogas, as well as in the generation of electricity and heat from the combustion of biogas [82]. Global energy generation from biogas reached 1,331,949 TJ in 2017, an increase of 57.8 % from 2010 and 367.35 % from 2000 [83].

In this study, considering the biogas produced, methane composition, and jabuticaba by-products mass used during AD, the estimated electrical and thermal energy were evaluated **(Table 4)**. Each ton of jabuticaba by-products submitted to the AD reactor could produce 118.1 kWh of electricity and 531.38 MJ of heat. Furthermore, this value increased to 543 kWh t^{-1} of electricity and 2,443.17 MJ t^{-1} of heat for the SWP+AD reactor, considering only the mass of jabuticaba by-products initially added in both reactors. With the accomplishment of this work, it was possible to obtain an increase in bioenergy production of 4.6-fold higher for electricity and heat, demonstrating that pretreatment is a great option to increase bioenergy production and that this process can be profitable for industrial implementation, reducing energy costs.

The energy and heat generated can be used by industries and even maintain the reactor's temperature. Since excess electricity can be sold to public networks, the use of bioenergy contributes to the mitigation of GHG. [80]. In this study, the methane-rich biogas produced in the AD reactor could mitigate a total of 38.61 kg $CO_{2\text{-eq}}$ t^{-1} (8.85 and 29.76 kg $CO_{2\text{-eq}}$ t^{-1}, respectively, for electricity and heat) **(Table 4)**. SWP increased methane production and avoided GHG emissions, reaching 177.54 kg $CO_{2\text{-eq}}$ t^{-1} (40.72 and 136.81 kg $CO_{2\text{-eq}}$ t^{-1}, respectively, for electricity and heat).

Table 4. Methane yield, potential of electric energy, heat, and avoided GHG emissions for the semi-continuous AD and SWP+AD of jabuticaba by-product.

Parameters	AD reactor	SWP+AD reactor	Unit
Methane yield	42.31	239.04	L CH_4 kg^{-1} TVS_{added}
Electricity	118.1	543.0	kWh t^{-1}
Heat	531.38	2,443.17	MJ t^{-1}
Avoided $GHG_{electricity}$	8.85	40.72	kg $CO_{2\text{-eq}}$ t^{-1}
Avoided GHG_{heat}	29.76	136.81	kg $CO_{2\text{-eq}}$ t^{-1}
Avoided GHG_{total}	38.61	177.54	kg $CO_{2\text{-eq}}$ t^{-1}

Finally, the use of biogas generated in AD and SWP+AD reactors can be used for energy recovery in the jabuticaba processing industry, resulting in financial savings and generating an economic and environmental return. The AD is an alternative for the decentralized production of electric energy, diversifying the energy matrix and reducing GHG emissions.

3.6. Energy balance

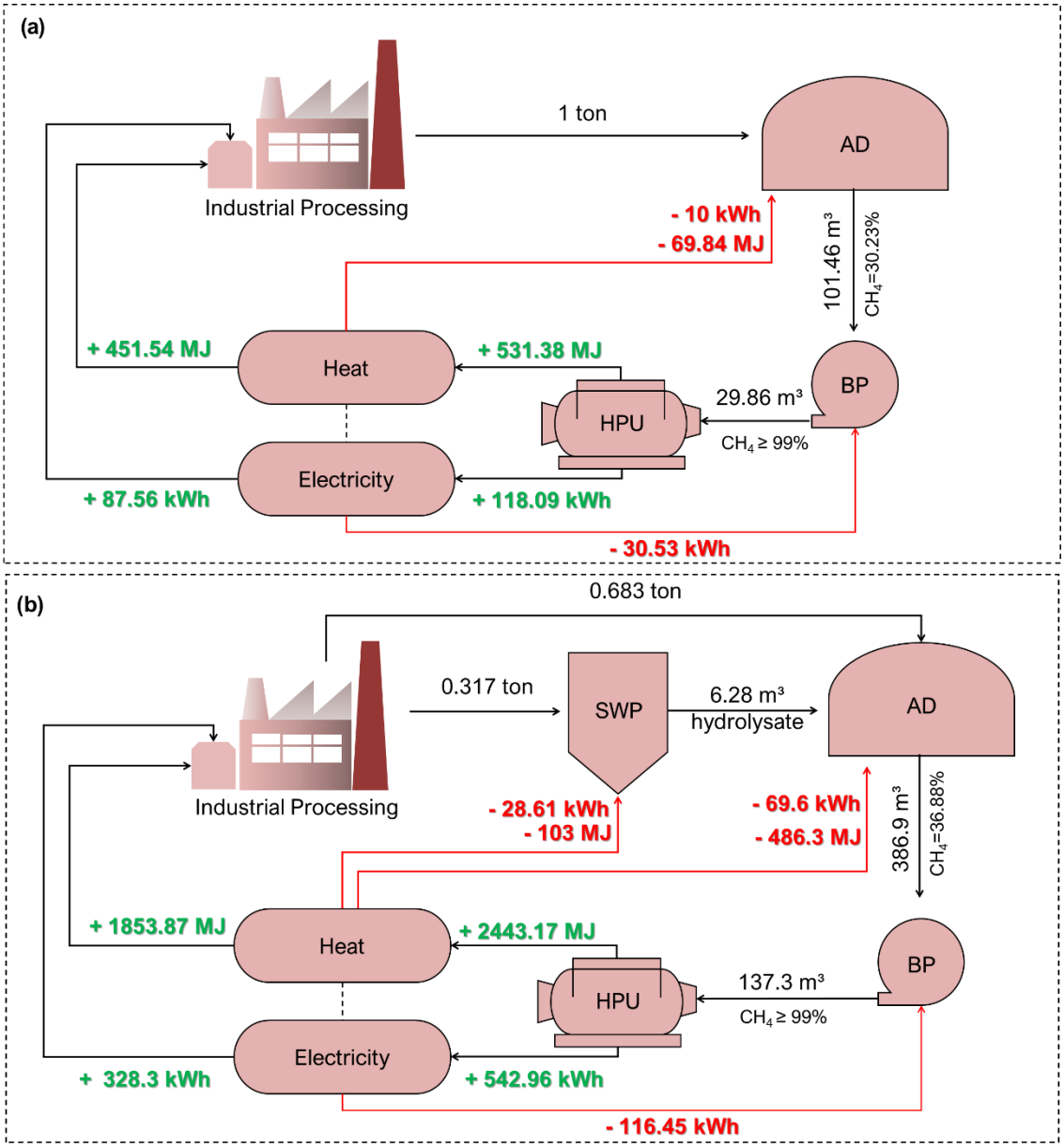
The energy balance was performed considering the input of 1 ton of jabuticaba by-product (**Fig. 7**). The energy consumption in the process was determined to verify the surplus of the electricity and heat generated from the combustion of the methane generated from AD and SWP+AD. The AD of jabuticaba by-product without pretreatment could produce 101.46 m^3 biogas with an average of 30.23 % methane, considering the operational performance described in the AD reactor (**Fig. 7a**). For the application of biogas in a heat and power unit, purification is necessary to remove CO_2, hydrogen sulfide (H_2S), water vapor, and other contaminants that can be obtained in the industrial process of AD.

After purification, a cogenerator can transform methane into electrical and thermal energy. The electric energy generated can be used to supply the energy demand for purification,

which was calculated for the AD process at 30.53 kWh, considering an electrical consumption of 0.301 kWh m^{-3} [84]. In addition, a standard anaerobic reactor with a capacity of 1 ton, 10 kWh of electricity, and 69.84 MJ of heat is required for the mesophilic treatment [85]. In the simulated process operated with 1 ton of jabuticaba by-product, the energy required can be supplied from the self-energy produced. Finally, net electricity (87.56 kWh) and heat (451.54 MJ) can be used in the jabuticaba processing industry to replace the acquisition of national grid energy and natural gas to supply the heat in boilers. In this case, the total avoided GHG emissions are 6.56 kg $CO_{2\text{-}eq}$ for electricity and 25.29 kg $CO_{2\text{-}eq}$ for heat.

For the bioprocess with the adoption of SWP, the energy balance was estimated in the laboratory-scale hydrolysis reactor [34]. The heat required for the pretreatment was estimated at 647.6 kJ kg^{1}. In the process described in **Fig. 7b**, a total of 1 ton of jabuticaba by-products can be used for the pretreatment (0.317 ton) and AD (0.683 ton), considering that it is necessary to feed the process. From the SWP of 0.317 tons, it is possible to produce 6.28 m^3 hydrolysate that will be used in the start-up of the SWP+AD reactor. For the energy balance, 50 % of the energy for SWP was supplied by thermal energy (103 MJ), and the other was provided by electricity (28.61 kWh), both generated in the heat and power unit. In addition, the anaerobic reactor demands 69.6 kWh and 486.3 MJ, respectively, for electricity and heat. In this scenario, using SWP, the energy surplus was estimated at 328.3 kWh electricity and 1853.87 MJ heat. In this process, the avoided GHG emissions are 24.62 kg $CO_{2\text{-}eq}$ for electricity and 103.87 kg $CO_{2\text{-}eq}$ for heat.

Figure 7. Industrial mass and energy balance for the AD of jabuticaba by-product with and without SWP. (a) Process with the adoption of the AD reactor (without pretreatment). (b) Process with the adoption of the SWP+AD reactor (with pretreatment).



Finally, the SWP of jabuticaba by-product followed by AD had a surplus of electricity (3.75-fold higher) and heat (4.1-fold higher) compared with the AD of jabuticaba by-product without pretreatment. The processes studied can contribute to the reduction of the carbon footprint of the agri-food sector since the energy generated can be used in the processing of

jabuticaba. Therefore, the waste management process studied can operate in a circular economy, reducing energy costs, replacing the grid energy and natural gas with biogas, and reducing GHG emissions.

3.7 Application of digestate for the germination of *Lactuca sativa*

The effect of the digestate on lettuce germination was evaluated. The visual appearance of the germinated lettuce with digestate and with water (control) is presented in **Fig. 8.** The application of digestate decreased the germination index with the increase in the amount of digestate applied (**Table 5**).

Figure 8. Germination of lettuce with different concentrations of digestate obtained from the semi-continuous AD and SWP+AD of jabuticaba by-product

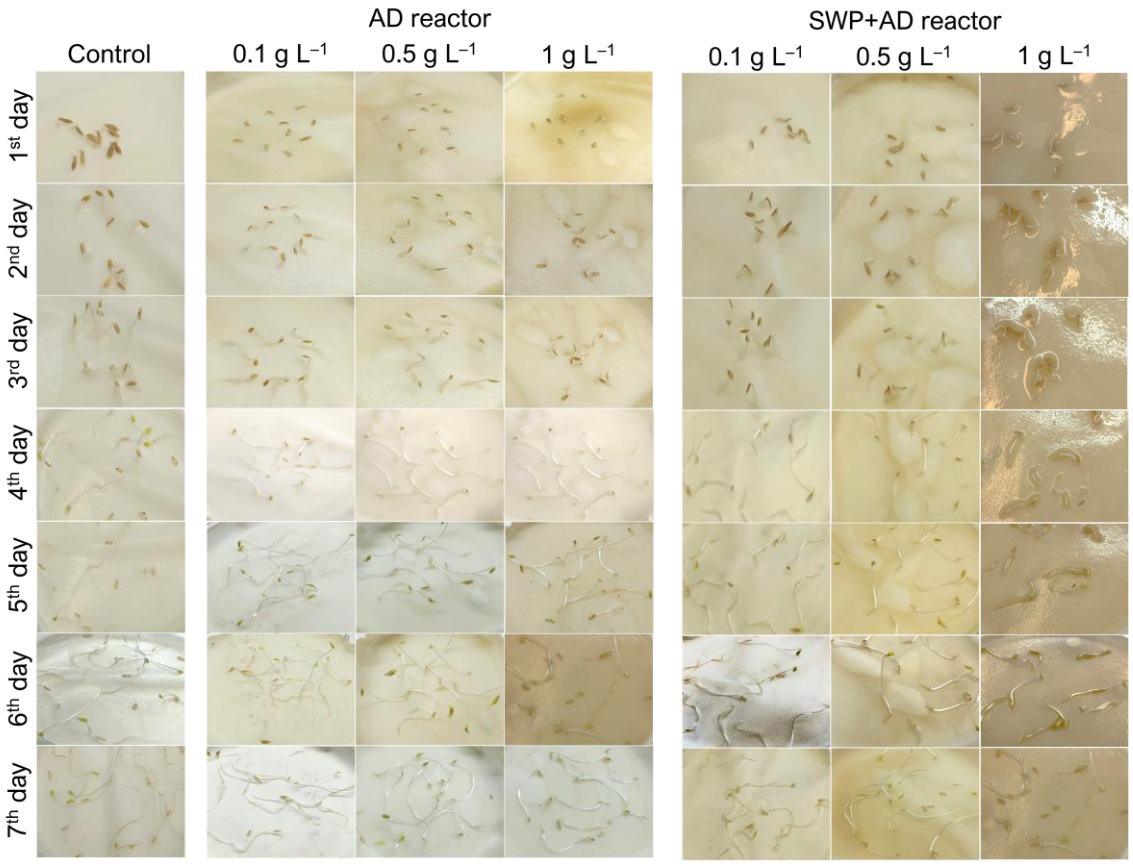


Table 5. Germination index and percentage of inhibition of the digestate obtained at the end of the semi-continuous AD and SWP+AD of jabuticaba by-product.

Parameters	Day	AD reactor			SWP+AD reactor		
		0.1 g L^{-1}	0.5 g L^{-1}	1 g L^{-1}	0.1 g L^{-1}	0.5 g L^{-1}	1 g L^{-1}
Germination index (%)	4	77.79 ± 5.28^{aA}	25.48 ± 1.63^{cA}	18.83 ± 2.80^{dA}	67.46 ± 0.92^{bB}	22.76 ± 0.30^{cA}	23.77 ± 0.40^{cA}
	7	63.73 ± 0.42^{bB}	19.36 ± 2.64^{dB}	13.75 ± 1.02^{eB}	75.32 ± 3.55^{aA}	23.29 ± 1.45^{cA}	17.63 ± 2.73^{dB}
Inhibition (%)	4	22.21 ± 5.28^{cB}	74.52 ± 1.63^{aB}	81.17 ± 2.80^{aB}	32.54 ± 0.92^{bA}	77.24 ± 0.30^{aA}	76.23 ± 0.40^{aB}
	7	36.27 ± 0.42^{cA}	80.64 ± 2.64^{aA}	86.25 ± 1.02^{aA}	24.68 ± 3.55^{dB}	76.71 ± 1.45^{bA}	82.37 ± 2.73^{aA}

Different letters (lowercase for lines and uppercase for the columns) indicate significant differences by Tukey's test at $p \leq 0.05$.

The germination test using digestate from the AD reactor (**Fig. 9a**) and the SWP+AD reactor (**Fig. 9b)** showed that the time for seed germination was associated with the concentration of digestate. The digestate from the SWP+AD reactor had a greater inhibitory effect on the first day when compared with the AD reactor, as the germination index was approximately 10 % (SWP+AD reactor) and 70 % (AD reactor). Even though the digestate from the SWP+AD reactor had a higher inhibitory power, the root lengths of the germinated seeds (**Fig. 9c**) were longer than the roots that used the digestate from the AD reactor. The germination index of digestate from SWP+AD had a slight increase on day 7 compared to day 4, probably due to the lower concentration of digestate, where the presence of inhibitory compounds did not cause growth deceleration over time.

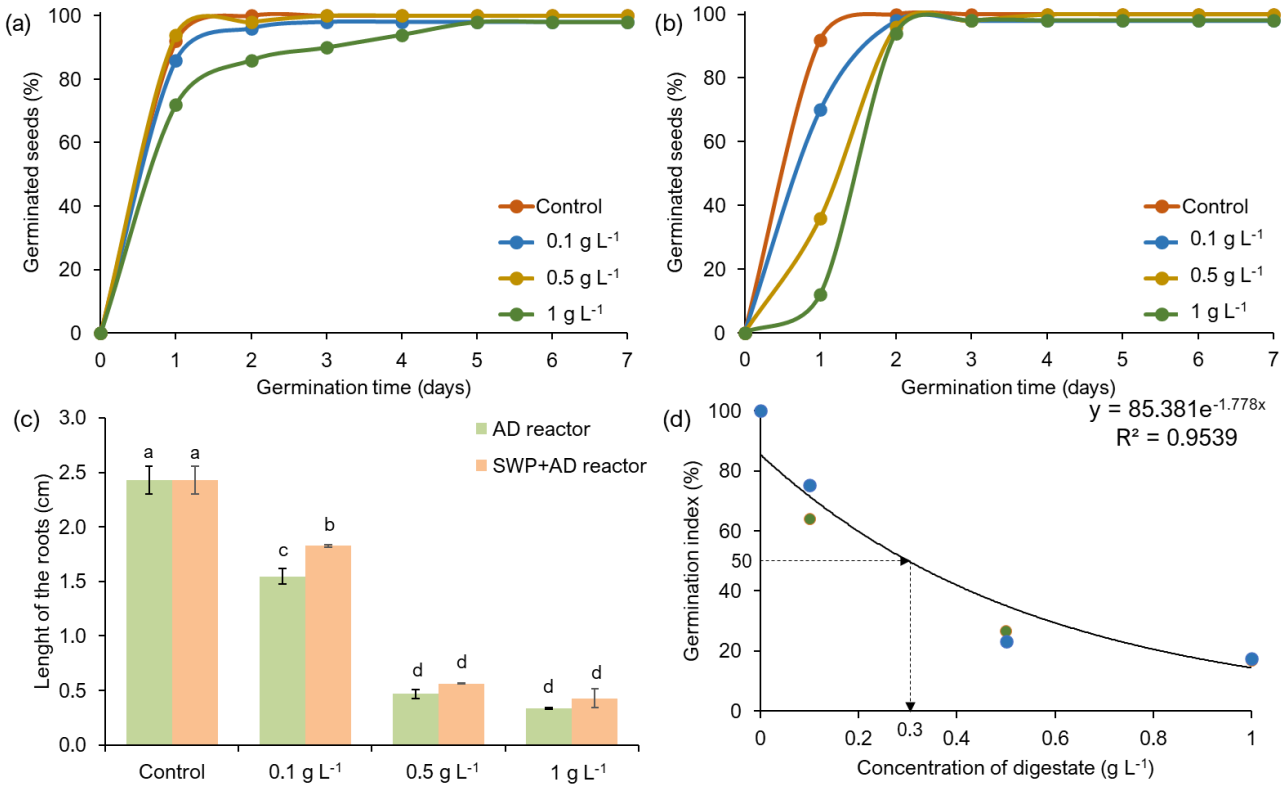
Notwithstanding, a germination index lower than 50 % indicates high toxicity [86]. In this study, the use of 0.5 and 1 g L^{-1} digestate had high toxicity and was not suitable for agricultural application in the germination of lettuce. The inhibition percentage increased for the digestate from the AD reactor, with concentrations higher than 0.5 g L^{-1}, and for the SWP+AD reactor, the same fact was observed for concentrations higher than 1 g L^{-1}. This fact can be explained by the presence of inhibitory compounds that affect lettuce germination, corroborating the literature [35]. The digestate submitted to ultrafiltration reduced its toxicity, increasing the germination rate of watercress (*Lepidium sativum*) [38]. Further technologies should be developed to reduce the toxicity of the digestate and increase the concentration for agricultural application.

The germination index of lettuce was predicted as a function of the concentration of digestate applied (**Eq. 10**).

$$y = 85.381e^{-1.778x} \quad (R^2 = 0.953) \quad (10)$$

The regression analysis demonstrated that to achieve a germination index of 50 % (limit to indicate toxicity), a digestate concentration of 0.3 g L^{-1} can be applied. Therefore, the digestate obtained after AD and SWP+AD can be used up to 0.3 g L^{-1}, without toxic effects.

Figure 9. Variation in germination seeds, length of the germinated roots, and germination index of the seeds of lettuce as a function of dilution levels. (a) Germinated seeds (%) treated with digestate from the AD reactor. (b) Germinated seeds (%) treated with digestate from the SWP+AD reactor. (c) Length of the roots (cm). (d) Regression analysis to predict the germination index as a function of the concentration of digestate.



4. Conclusion

The subcritical water pretreatment of jabuticaba by-product proved to be effective in producing sugars. The hydrolysate showed high concentrations of glucose (5.78 g L^{-1}), fructose (3.63 g L^{-1}), arabinose (1.82 g L^{-1}), and cellobiose (1.28 g L^{-1}). The use of pretreated jabuticaba by-product was excellent for methane generation. The methane production in the SWP+AD reactor (239.04 L CH_4 kg^{-1} TVS) was 5.64-fold higher than that of the AD reactor (42.31 L CH_4 kg^{-1} TVS) without pretreatment. The methane-rich biogas from the AD reactor could produce 451.54 MJ of heat and 87.56 kWh of electricity per ton of jabuticaba by-product, while the SWP+AD reactor could generate 1853.87 MJ of heat and 328.3 kWh of electricity. Furthermore, the avoided GHG emissions were estimated at 177.54 kg $CO_{2\text{-eq}}$ t^{-1} and 38.61 kg $CO_{2\text{-eq}}$ t^{-1} for the SWP+AD reactor and AD reactor, respectively. The digestate generated after the anaerobic process can be applied as a sustainable fertilizer with a concentration up to 0.3 g L^{-1}, without toxic effects on the germination of *Lactuca sativa*. In conclusion, the designed bioprocess combining subcritical water pretreatment followed by anaerobic digestion can be a promising alternative for sustainable waste management and the recovery of bioenergy and fertilizer, advocating a circular economy transition of the agri-food industry.

CRediT authorship contribution statement

Rafael Gabriel da Rosa: Methodology, Investigation, Validation, Writing – original draft. **William Gustavo Sganzerla:** Conceptualization, Methodology, Investigation, Validation, Writing – original draft, Writing – review & editing. **Tiago Linhares Cruz Tabosa Barroso:** Methodology, Investigation, Writing – original draft. **Luiz Eduardo Nochi Castro:** Methodology, Investigation, Writing – original draft. **Mauro Donizetti Berni:** Supervision, Resources, Writing – review & editing, Funding acquisition. **Tânia Forster-Carneiro:** Supervision, Resources, Writing – review & editing, Funding acquisition.

Declaration of Competing Interest

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

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CAPÍTULO 4 – Discussão Geral

4.1. - Sustainable production of bioactive compounds from jabuticaba (*Myrciaria cauliflora*): a bibliometric analysis of scientific research over the last 21 years

A análise bibliométrica demonstrou que os trabalhos realizados utilizando a jabuticaba como matéria-prima, elucidaram que a Tecnologia em Ciência de Alimentos foi a principal área de pesquisa, e as palavras-chave mais frequentes no campo estavam associadas a antocianinas, demonstrando que há uma tendência em determinar o método ideal de extração desse composto bioativo da jabuticaba.

Para esta discussão realizou-se a análise bibliométrica sobre a jabuticaba (*Myrciaria cauliflora*) nos últimos 21 anos (2000 a 2021) para identificar perspectivas para a produção sustentável de compostos bioativos a partir de seus subprodutos.

O software bibliométrico *VosViewer*© foi utilizado para analisar o banco de dados da *Web of Sciencie*©, onde o mesmo executou mapas através dos dados obtidos. Ao todo foram publicados 255 artigos e 5 revisões relacionadas a pesquisas sobre jabuticaba nos últimos 21 anos. O trabalho fornece uma tendência na produção de compostos bioativos (ácido gálico, cianidina, malvidina, peonidina, petunidina, pelargonidina, e muitos outros) em diferentes rotas tecnológicas sustentáveis (extração assistida por ultrassom, extração supercrítica de dióxido de carbono e extração líquida pressurizada) utilizando subprodutos da jabuticaba como matéria-prima.

A partir do estudo foi possível verificar que a jabuticaba possui um grande potencial para a extração de compostos bioativos de importância farmacêutica, e que os estudos atuais são focados na sua produção. Com isso, pode-se avaliar a aplicação da jabuticaba e direcionar novos estudos em áreas poucas exploradas utilizando esta matéria-prima, como por exemplo a sua utilização para a realização da digestão anaeróbia visando a produção de biogás rico em

metano, área inexplorada utilizando a jabuticaba e seus resíduos como substrato para a digestão anaeróbica.

4.2. Sustainable bioprocess combining subcritical water pretreatment followed by anaerobic digestion for the valorization of jabuticaba (*Myrciaria cauliflora*) agro-industrial by-product in bioenergy and biofertilizer

O estudo mostrou um bioprocesso sustentável que combina pré-tratamento de água subcrítica, seguido de digestão anaeróbica semi-contínua (DA) para valorização do subproduto agroindustrial jabuticaba (*Myrciaria cauliflora*) em bioenergia e biofertilizante. Para o trabalho utilizou-se como parâmetros operacionais para a realização do pré-tratamento com água subcrítica, as seguintes condições: temperatura de 180 °C, pressão de 15 MPa, 10 mL min^{-1} de vazão de água, solvente para alimentação de 22,5 g^{-1} e 45 min de tempo cinético. Após a hidrólise, o hidrolisado foi colocado em reator anaeróbio e operado por 50 dias em modo semi-contínuo sob condições mesófilas e metogênicas, assim como o reator controle também (sem pré-tratamento). A composição de açúcares obtidas no hidrolisado foram a glicose (5,78 g L^{-1}), frutose (3,63 g L^{-1}), arabinose (1,82 g L^{-1}) e celobiose (1,28 g L^{-1}).

Os resultados demonstram que houve um aumento no rendimento do metano (239,04 L CH_4 kg^{-1} TVS) no reator pré-tratado, quando comparado com o reator controle sem pré-tratamento (42,31 L CH_4 kg^{-1} TVS). O trabalho confirma que o reator anaeróbio com o pré-tratamento com água subcrítica aumenta a capacidade de geração de eletricidade e calor, na comparação ao reator sem o pré-tratamento, sendo capaz de gerar 451,54 kWh t^{-1} de eletricidade e 1853,87 MJ t^{-1} de calor, evitando uma emissão de GEE no total de 177,54 kg de CO_2^{-eq} t^{-1}, enquanto o reator controle é capaz de gerar 328,3 kWh t^{-1} de eletricidade e 328,3 MJ t^{-1} de calor, evitando um total de GEE de 38,61 kg de CO_2^{-eq} t^{-1}.

O biofertilizante proveniente do digestato da digestão anaeróbia, foi testado como biofertilizante e pode ser utilizado até uma concentração de 0,3 g L^{-1}, sem causar nenhum efeito inibitório na germinação da alface (Lactuca sativa).

Finalmente, o bioprocesso sustentável torna-se uma alternativa para o tratamento do subproduto da jabuticaba em uma estrutura de economia circular, pois pode gerar bioenergia e biofertilizantes agrícolas que possam reduzir as emissões de gases de efeito estufa (GEE), diminuindo os efeitos do aquecimento global, bem como a poluição ambiental na indústria alimentícia.

CAPÍTULO 5 – Conclusão Geral

O estudo avaliou um bioprocesso combinando a hidrólise em água subcrítica, seguida de reatores anaeróbios em regime semi-contínuo do subproduto (cascas + sementes) industrial da jabuticaba para recuperar bioenergia e fertilizante agrícola. Para a avaliação do desempenho dos reatores de digestão anaeróbia utilizou-se os parâmetros operacionais de geração de biogás, composição de metano e ácidos graxos voláteis. Os objetivos principais deste estudo foram a recuperação da bioenergia a partir de biogás gerado na digestão anaeróbia do subproduto industrial da jabuticaba, verificar a redução de emissões de GEE e aplicação do digestato como biofertilizante na capacidade de germinação da alface.

A revisão bibliométrica sobre pesquisas de jabuticaba (*Myrciaria cauliflora*) nos últimos 21 anos (2000-2021), foi realizada para identificar as perspectivas para a produção sustentável de compostos bioativos e identificar as rotas tecnológicas verdes para gerar novos produtos biológicos a partir de subprodutos da Jabuticaba. As principais conclusões foram:

- ✓ A jabuticaba e seus subprodutos (cascas + sementes) apresentam grande potencial biológico, como antioxidante, antimicrobiano, anti-inflamatório, antidiabético, entre outras propriedades funcionais. Os subprodutos constituem um ramo de mercado, pois implicam em causas ambientais, econômicas e sociais no conceito de economia circular;

- ✓ As cascas de jabuticaba podem ser recicladas para produzir bioprodutos com uso de tecnologias verdes emergentes, visando a produção de compostos bioativos de alta qualidade. No entanto, uma investigação mais aprofundada é necessária para abordar adequadamente a adoção de biotecnologias para alcançar bioprodutos economicamente viáveis;

- ✓ A revisão sistemática revelou novas tendências e tecnologias para a recuperação de compostos bioativos através de 255 artigos e 5 revisões nos últimos 21 anos (2000-2021). As áreas de pesquisa mais predominantes foram Tecnologia em Ciência de Alimentos, Química e Agricultura;

- ✓ As técnicas de extração de compostos bioativos nas suas diferentes rotas tecnológicas sustentáveis da jabuticaba indicam a extração assistida por ultrassom, extração supercrítica de dióxido de carbono e extração de líquido pressurizado aquelas mais empregadas.

Realizou-se o estudo para a recuperação de bioenergia a partir de biogás rico em metano, GEE evitados e aplicação de digestato como biofertilizante agricola. Os resultados da aplicação de hidrólise em água subcrítica do subproduto agroindustrial da jabuticaba previamente a digestão anaeróbia indicaram as seguintes conclusões:

- ✓ O estudo demonstrou que a hidrólise com água subcrítica do subproduto da jabuticaba, como pré-tratamento a digestão anárobica, mostrou-se eficaz na produção de açúcares;

- ✓ O hidrolisado continha concentrações de glicose (5,78 g L^{-1}), frutose (3,63 g L^{-1}), arabinose (1,82 g L^{-1}) e celobiose (1,28 g L^{-1});

- ✓ O uso do subproduto agroindustrial de jabuticaba pré-tratado obteve um ótimo desempenho para a geração de metano;

- ✓ A produção de metano no reator pré-tratado foi de 239,04 L CH_4 kg^{-1} TVS, sendo 5,64 vezes maior que a do reator controle (42,31 L CH_4 kg^{-1} TVS), sem o pré-tratamento;

- ✓ O reator sem pré-tratamento pode produzir 453,54 MJ de calor e 87,56 kWh de eletricidade por tonelada de casca de jabuticaba;

- ✓ Já o reator com pré-tratamento pode gerar 1853,87 MJ de calor e 328,3 kWh de eletricidade;

- ✓ Para o reator com pré-tratamento a quantidade de gases de efeito estufa evitdos podem ser de 177,54 kg de CO_2^{-eq} t^{-1}, enquanto no reator controle podem ser evitadas 38,61 kg CO_2^{-eq} t^{-1};

- ✓ Na concentração de 0,3 g L^{-1} , o biofertilizante proveniente do digestato da digestão anaeróbica pode ser aplicado sem efeito tóxico na germinação da alface (Lactuca sativa), pois essa foi a concentração máxima onde não houve efeito inibitório no crescimento da alface.

- ✓ Uma transição da economia circular pode ser defendida utilizando o bioprocesso que combina o pré-tratamento de água subcrítica seguido da digestão anaeróbica, tornando

assim uma ótima alternativa para a gestão consciente de resíduos, transformando-os em bioenergia e fertilizantes.

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ANEXO 1

DECLARAÇÃO DE AUTORIZAÇÃO DE USO DE CONTEÚDO

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Sustainable production of bioactive compounds from jabuticaba (Myrciaria cauliflora): A bibliometric analysis of scientific research over the last 21 years

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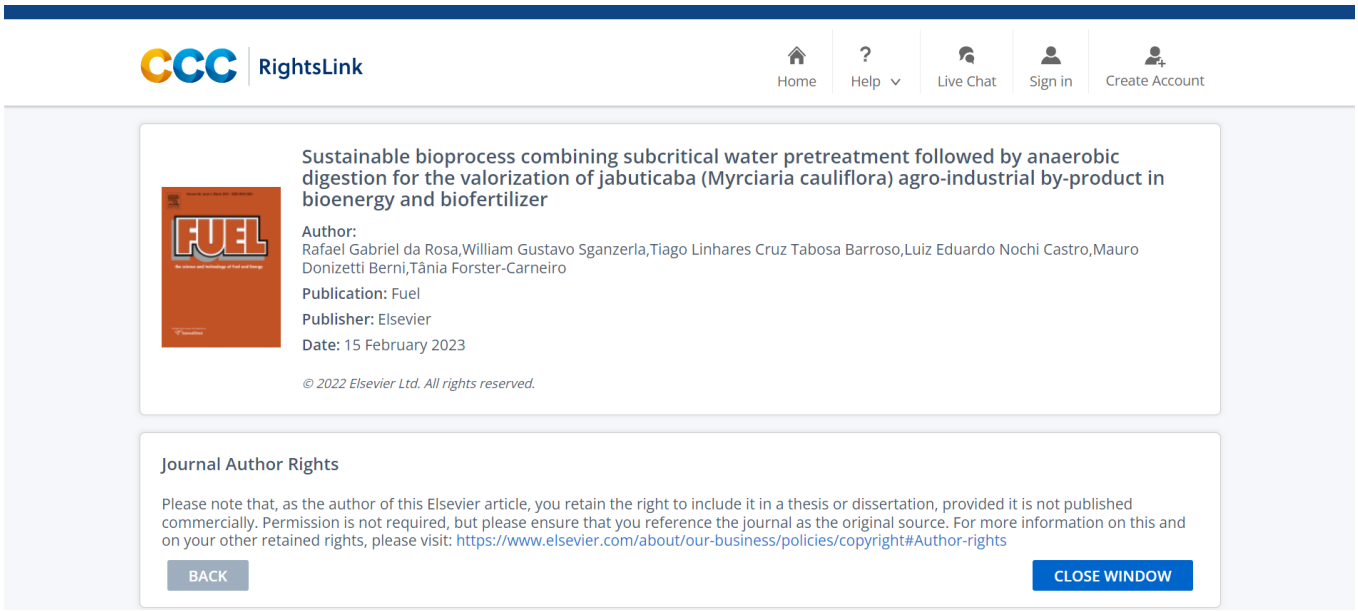

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Sustainable bioprocess combining subcritical water pretreatment followed by anaerobic digestion for the valorization of jabuticaba (Myrciaria cauliflora) agro-industrial by-product in bioenergy and biofertilizer

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