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PROGRAMA DE PÓS-GRADUAÇÃO EM ENERGIA E AMBIENTE**

**FRANCISCO IZAIAS DA SILVA AIRES**

**SOLAR-POWERED HYDROGEN PRODUCTION FROM SODIUM  
BOROHYDRIDE HYDROLYSIS USING SUGARCANE BAGASSE  
(*SACCHARUM OFFICINARUM*) AS A GREEN CATALYST**

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Dissertação apresentada ao Programa de Pós-Graduação em Energia e Ambiente, do Instituto de Engenharias e Desenvolvimento Sustentável, da Universidade da Integração Internacional da Lusofonia Afro-Brasileira, como requisito parcial para obtenção do título de Mestre em Energia e Ambiente.

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*A Deus, a mim, a minha mãe Maria  
José, e a minha família.*

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*“O aprendizado contínuo é a chave  
para se manter relevante”*

*John Maxwell*

## ABSTRACT

The quest to replace fossil fuels with clean energy has been driven by the production of more sustainable and metal-free hydrogen. A hydrogen generation system based on the hydrolysis of sodium borohydride ( $\text{NaBH}_4$ ) was developed using catalysts derived from residual biomass, specifically sugarcane bagasse. The successful synthesis of Modified Sugarcane Bagasse (MOB) catalysts with high stability and catalytic activity impregnated with phosphoric acid  $\text{H}_3\text{PO}_4$  was confirmed through characterization techniques, including FTIR, XRD, XRF, TGA, SEM and GC, which verified positive structural modifications. The Taguchi method was applied to optimize the process and reduce the number of experiments. The factors that contributed the most statistically within the model confidence range were time (35 minutes), temperature (60 °C),  $\text{NaBH}_4$  (750 mg) and MOB (300 mg), with  $\text{NaBH}_4$  standing out with 58.87% and the MOB catalyst contributing with 27.03% for hydrogen production. Additionally, the integration with a photovoltaic solar energy grid demonstrated energy feasibility, with a total consumption of 136 kWh to produce 1000 mL of  $\text{H}_2$ . The results show that the MOB exhibits superior catalytic performance for  $\text{NaBH}_4$  hydrolysis, with excellent economic viability. This catalyst is highly promising for practical applications in  $\text{NaBH}_4$  hydrolysis.

**Keywords:** Sugarcane Bagasse, Green Catalyst, Hydrogen Production, Hydrolysis,  $\text{NaBH}_4$ .

## RESUMO

A busca pela substituição de combustíveis fósseis por energia limpa tem sido impulsionada pela produção de hidrogênio mais sustentável e livre de metais. Um sistema de geração de hidrogênio baseado na hidrólise de borohidreto de sódio ( $\text{NaBH}_4$ ) foi desenvolvido usando catalisadores derivados de biomassa residual, especificamente bagaço de cana-de-açúcar. A síntese bem-sucedida de catalisadores de Bagaço de Cana Modificado (MOB) com alta estabilidade e atividade catalítica impregnado com ácido fosfórico  $\text{H}_3\text{PO}_4$  foi confirmada por meio de técnicas de caracterização, incluindo FTIR, XRD, XRF, TGA, SEM e GC, que verificaram modificações estruturais positivas. O método Taguchi foi aplicado para otimizar o processo e reduzir o número de experimentos. Os fatores que mais contribuíram estatisticamente dentro da faixa de confiança do modelo foram tempo (35 minutos), Temperatura (60 °C),  $\text{NaBH}_4$  (750 mg) e MOB (300 mg), com  $\text{NaBH}_4$  se destacando com 58,87% e o catalisador MOB contribuindo com 27,03% para a produção de hidrogênio. Adicionalmente, a integração com uma rede de energia solar fotovoltaica demonstrou viabilidade energética, com um consumo total de 136 kWh para produzir 1000 mL de  $\text{H}_2$ . Os resultados mostram que o MOB exibe desempenho catalítico superior para hidrólise de  $\text{NaBH}_4$ , com excelente viabilidade econômica. Este catalisador é altamente promissor para aplicações práticas em hidrólise de  $\text{NaBH}_4$ .

**Palavras-chave:** Bagaço de Cana-de-açúcar, Catalisador Verde, Produção de Hidrogênio, Hidrólise,  $\text{NaBH}_4$ .

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## 1 INTRODUCTION

In recent years, the pursuit of alternative, sustainable energy solutions that respect the environment has become a major focus across various scientific fields (lian, 2024; Olabi *et al.*, 2023; Requena-Leal *et al.*, 2024; Sikiru *et al.*, 2024). Among the promising options, hydrogen stands out as a high-efficiency fuel with zero pollutant emissions during combustion, offering significant potential to replace fossil fuels (Qazi, 2022; Roy; Pramanik, 2024; Sadeq *et al.*, 2024). Among the existing production methods, the hydrolysis of sodium borohydride ( $\text{NaBH}_4$ ) emerges as an attractive route due to its high hydrogen generation capacity and efficiency, which can exceed 90%, especially when the water-to-borohydride molar ratio is optimized (Liu; Li; Suda, 2009). However, the slow reaction rate of sodium borohydride hydrolysis poses a significant challenge (Mirshafiee; Rezaei, 2024).

$\text{NaBH}_4$  is a stable compound that reacts with water in the presence of an appropriate catalyst, releasing hydrogen in a controlled and efficient manner (Chen, W. *et al.*, 2017; ERTÜRK, 2024; Fang *et al.*, 2020; Jia *et al.*, 2022; Manna *et al.*, 2017). This catalyzed reaction has garnered significant attention, driving the development of new catalytic materials (Li, Jinghua *et al.*, 2020; Muir; Yao, 2011). Traditionally, metallic catalysts have been widely studied, but their high costs and environmental impacts have prompted the search for more sustainable alternatives (Baruah *et al.*, 2024; García-Serna; Piñero-Hernanz; Durán-Martín, 2022). In this context, agricultural residues, such as biomass, have emerged as promising catalytic supports due to their abundance, low cost, and alignment with circular economy principles (Matthews; Seroka; Khotseng, 2024; Omran; Baek, 2022).

Sugarcane bagasse holds great potential in this scenario, as it combines features like high surface area, porous structure, and the presence of acidic functional groups, which favor its use as a support for catalytic materials (Omran; Baek, 2022; Zafeer *et al.*, 2024; Zhang, Ping *et al.*, 2020). Moreover, reusing biomass residues contributes to the valorization of agricultural by-products and reduces environmental impacts, highlighting the importance of integrated solutions that promote sustainability and innovation in the energy sector (Duque-Acevedo *et al.*, 2022; Nguyen; Toan, 2024). Among the catalysts developed to optimize hydrogen production via  $\text{NaBH}_4$  hydrolysis, materials synthesized from lignocellulosic residues stand out (Avcı Hansu, 2024; Patel, Nainesh; Miotello, 2015; Serrano *et al.*, 2024).

These catalysts not only offer high reaction efficiency but also incorporate environmentally advantageous characteristics, such as biodegradability and the possibility of reuse in subsequent cycles (Patel, Nainesh; Miotello, 2015; Serrano *et al.*, 2024). Chemical treatments of these materials, such as activation with acids or bases, enhance their porosity and increase the availability of active sites, which is crucial for maximizing catalytic reactions (Li, Dongxiao *et al.*, 2024; Pan *et al.*, 2024; Xiong *et al.*, 2024). Thus, the use of agricultural residues as a basis for catalysts represents a significant advance toward more sustainable and accessible processes (Díaz *et al.*, 2024; Khan *et al.*, 2021; Nath *et al.*, 2023). In this context, integration with renewable energy sources, such as solar energy, plays a fundamental role (Maka; Alabid, 2022). The photovoltaic minigeneration plant installed at UNILAB's Auroras Campus exemplifies the potential of clean energy in enabling sustainable hydrogen production systems. With its energy generation capacity, this plant provides a solid foundation for calculating the energy consumption required for catalyst synthesis and H<sub>2</sub> production. This approach not only demonstrates the energy feasibility of large-scale hydrogen production but also reinforces the role of renewable energy as a strategic support for sustainable energy transitions, reducing dependence on non-renewable resources and promoting energy autonomy across various sectors.

This study aims to investigate hydrogen production through the hydrolysis of sodium borohydride using sugarcane bagasse-based catalysts. To this end, it first presents an independent assessment of sugarcane bagasse (MOB) as a sustainable alternative for catalysis. The dissertation is written in English and organized into two main articles. Article 2, Sustainable Approaches in Energy Production from Sugarcane Bagasse: A Bibliometric Review, provides a comprehensive literature review that contextualizes the role of sugarcane bagasse in sustainable energy generation. This section examines significant advances in the field, identifies current challenges, and offers valuable insights for future research. Furthermore, the bibliometric analysis highlights recent trends and developments, establishing a solid foundation for the advancement of the study. Article 3, Solar-Powered Hydrogen Production from Sodium Borohydride Hydrolysis Using Sugarcane Bagasse (*Saccharum officinarum*) as a Green Catalyst, focuses on the characterization of the catalyst through experimental studies on lignocellulosic materials. This section introduces the Taguchi “L16” experimental design, which evaluates the optimization of borohydride hydrolysis under the “larger-the-better” performance criterion. With optimized parameters, the catalyst is tested

across six reuse cycles for hydrogen production, confirming its practical feasibility and efficiency. In addition, the study explores the integration of this process with a photovoltaic plant at the Universidade da Integração Internacional da Lusofonia Afro-Brasileira (UNILAB), Auroras Campus, further reinforcing its sustainable applicability. Finally, the dissertation provides an overview of the findings and discusses the contributions of this research to innovation and sustainable hydrogen production.

## **1.2 GENERAL OBJECTIVES**

- Explore the potential of sugarcane bagasse (*Saccharum officinarum*) as an innovative catalyst for hydrogen production through the hydrolysis of sodium borohydride (NaBH<sub>4</sub>).
- *Specific Objectives*
- Synthesize and characterize the sustainable sugarcane bagasse catalyst.
- Verify the efficiency of the sodium borohydride hydrolysis reaction with the catalyst (MOB).
- Evaluate the reuse of the sustainable catalyst in six hydrolysis reaction cycles.
- Determine the optimal reaction conditions for H<sub>2</sub> production using the sugarcane-based catalyst.
- Conduct a review on sustainable approaches to energy production from sugarcane bagasse.

## **2 SUSTAINABLE APPROACHES IN ENERGY PRODUCTION FROM SUGARCANE BAGASSE: A BIBLIOMETRIC REVIEW**

### **Abstract**

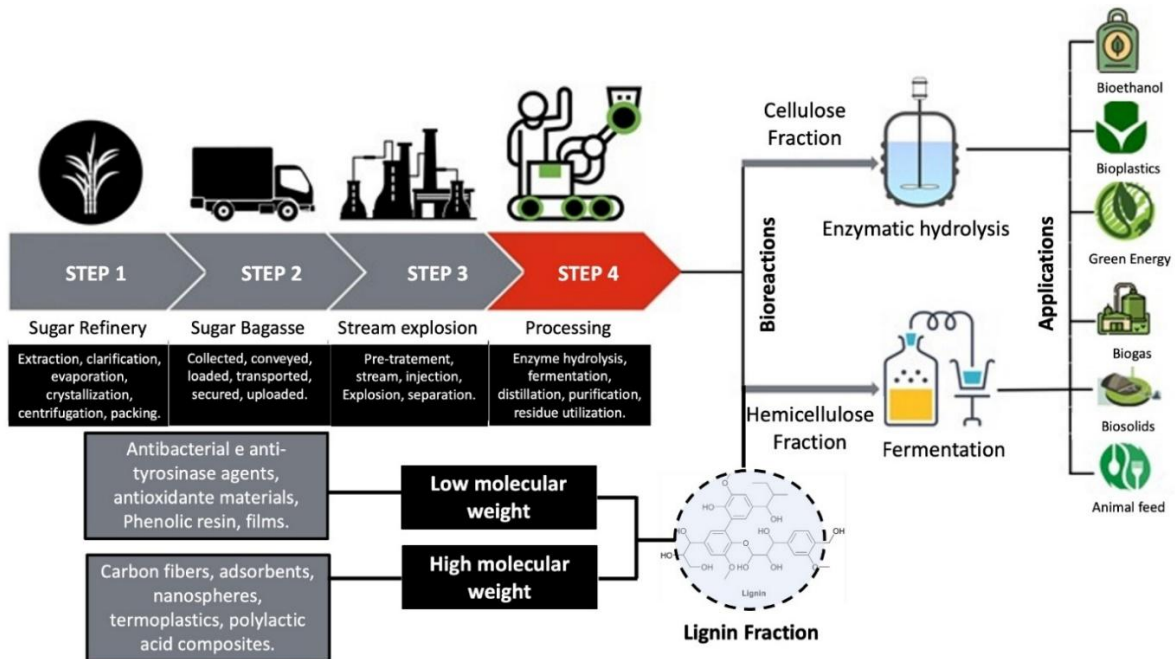
Sugarcane bagasse (SCB), a byproduct of the milling process in sugar and ethanol production, has garnered global attention for its potential in renewable energy research. This study provides a comprehensive bibliometric analysis of global research trends on SCB from 2005 to 2024, based on 26,663 articles from the Web of Science database, refined to 657 publications. The findings highlight Brazil, India, the United States, and China as leaders in SCB-related research and technological advancements. Keyword analysis revealed terms like "sugarcane bagasse," "biomass," and "renewable energy" as dominant, reflecting the expanding scope of SCB applications in sustainable energy research. This study primarily synthesizes existing knowledge from the literature regarding SCB's broader role in renewable energy and sustainability efforts. The analysis underscores the importance of SCB valorization in reducing greenhouse gas emissions and advancing a sustainable energy transition.

**Keywords:** Sugarcane Bagasse (SCB), Biomass Conversion Technologies, Renewable Energy, Energy Production, Advanced bibliometric.

## 2.1 INTRODUCTION

Sugarcane bagasse (SCB) (*Saccharum officinarum*) is a byproduct of the sugar and ethanol production process that is generated during the milling of sugarcane (Ajala *et al.*, 2021; Hiranobe *et al.*, 2024b). This lignocellulosic residue accounts for a significant portion of the total sugarcane biomass produced in the sugar industry (Isikgor; Becer, 2015; Yadav, Pratiksha *et al.*, 2024). Traditionally, SCB has been used as a fuel to generate thermal (Khattab; Watanabe, 2018) and electrical (Dantas; Legey; Mazzone, 2013) energy within the mills themselves, contributing to their energy self-sufficiency (Hofsetz; Silva, 2012) In recent years, however, its potential as a sustainable energy source has gained considerable recognition, spurring research and technological advances aimed at supporting the energy transition (Kabeyi; Olanrewaju, 2023). Despite this, a significant amount of SCB is still wasted or underused (Huang, Jiangfeng *et al.*, 2020a), posing a challenge to maximize its use and implement more efficient, sustainable solutions (Kabeyi; Olanrewaju, 2023; Wani *et al.*, 2023) (Figure 2.1).

**Figure 2.1** Process flow of sugarcane bagasse (SCB): From harvesting and milling to treatment, processing, and bioreactions. The figure also illustrates its industrial applications, including the production of biomaterials, biofuels, and other derivatives from SCB.



Source: Author (2024).

The growing global demand for sustainable energy has intensified efforts to identify alternative and renewable sources, aiming to reduce reliance on fossil fuels and mitigate environmental impacts (Jaiswal *et al.*, 2022; Owusu; Asumadu-Sarkodie, 2016a). In this context, SCB (sugarcane bagasse) has emerged as a promising resource, particularly in sugarcane-producing countries such as Brazil, India, and China (Melati *et al.*, 2017; Owusu; Asumadu-Sarkodie, 2016b). SCB is not only abundant and renewable but also possesses high energy potential, making it suitable for conversion into electricity, biofuels, and other value-added products (Awasthi Shraddha and Mishra, 2024; Faizal *et al.*, 2019; Hassan, Soulayma; Ngo; Ball, 2024). Using SCB contributes to diversifying the energy matrix while promoting economic and environmental sustainability by valorizing this widely available agricultural residue. Therefore, harnessing the potential of SCB is essential for progressing toward a more sustainable and resilient energy future (Bordonal *et al.*, 2018; Hiloidhari *et al.*, 2018).

Chemically, SCB is primarily composed of cellulose, which accounts for approximately 40-50% of its composition and serves as the primary source of fermentable sugar (Mahmud, Md Arif; Anannya, 2021). Cellulose, a crucial biopolymer found in the cellular structures of plants (Rongpipi *et al.*, 2019), provides rigidity and strength to plant cells (Arakawa; DeForest, 2017). It is widely used in the production of cardboard (Jeihanipour *et al.*, 2010), fabric (Rongpipi *et al.*, 2019), and bioethanol (Mikulski; Kłosowski, 2023). Hemicellulose, comprising about 25-35% of SCB (Konde *et al.*, 2021), is a heterogeneous polymer composed of various pentoses (e.g., xylose and arabinose) and hexoses (e.g., glucose and galactose), along with uronic acids (Peng, Feng *et al.*, 2011; Xu, Q. *et al.*, 2011). Hemicellulose forms a matrix around the cellulose fibers, contributing to the flexibility of the cellular structure (Qaseem; Shaheen; Wu, 2021).

Hemicellulose is utilized in the production of biofuels (Huang, Ling Zhi *et al.*, 2021), bioplastics (Macedo *et al.*, 2022), and chemicals such as xylitol and furfural (Rao *et al.*, 2023). Lignin, which constitutes 20-30% of SCB, is a complex polymer made up of phenols (Bugg, 2024; Nabipour; Hu, 2022), primarily consisting of monolignol units such as coniferyl, sinapyl, and p-coumaryl alcohol (Amthor, 2003). Lignin provides rigidity and strength to the cell wall, protecting cellulose fibers from microbial degradation and contributing to the structural integrity of plants (Iram; Berenjian; Demirci, 2021; Mariana *et al.*, 2021). It is used in the production of biofuels (Vasile; Baican, 2023), resins (Juhl; Hauschild; Dam-Johansen, 2024),

adhesives (Stewart, 2008), and as an additive in composite materials and concrete (Yao *et al.*, 2022). This chemical composition makes SCB a valuable feedstock for the production of bioenergy and bioproducts.

The importance of SCB as a renewable resource is evident due to its abundance and consistent availability in sugarcane-producing regions (Iwuozor *et al.*, 2023; Norsuraya; Fazlena; Norhasyimi, 2016). The use of SCB in sustainable energy production not only reduces dependence on fossil fuels but also helps lower greenhouse gas emissions (Neamhom; Polprasert; Englande, 2016; Vandenberghe *et al.*, 2022). Additionally, valorizing SCB can bring substantial economic benefits to the sugarcane industry and support the development of clean and efficient technologies (Ortiz-Sanchez *et al.*, 2024; Wani *et al.*, 2023). The SCB has great potential for sustainable energy, with various technologies such as energy cogeneration (Manzini Poli *et al.*, 2022), biogas production via anaerobic digestion (Zongo *et al.*, 2024), advanced biofuel production, and fuel cell generation (Canabarro *et al.*, 2013; Nicodème *et al.*, 2018; Poornima *et al.*, 2024). With the help of bibliometric analysis, new perspectives for future research have emerged to address current knowledge gaps. This analysis seeks to answer the following bibliometric review questions (RQs):

- RQ1: How has scientific research on developing technologies for energy production from SCB evolved?
- RQ2: Which countries/regions have contributed significantly to using sugarcane bagasse, and what are their advantages?
- RQ3: What are the most essential keywords mentioned in the search?
- RQ4: What are the emerging research areas in this field?

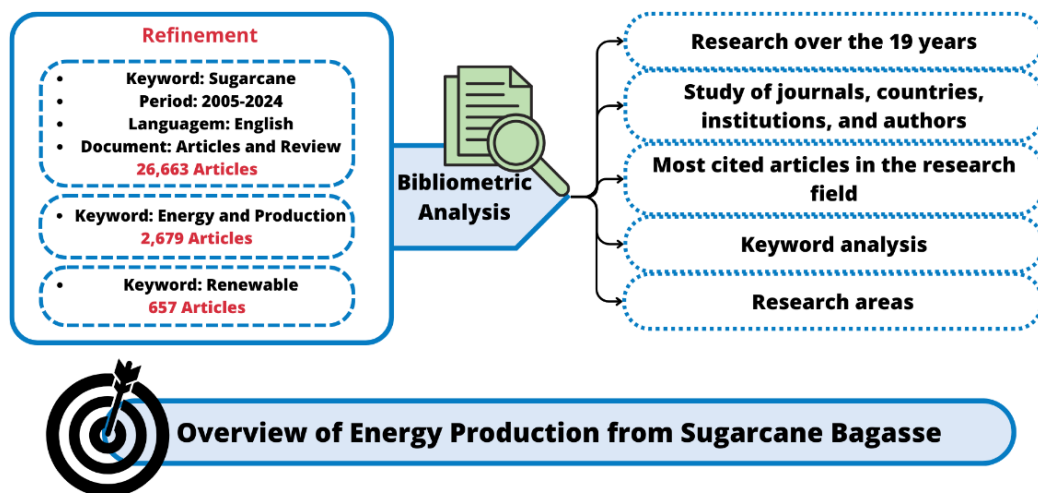
In this context, this study aims to conduct a comprehensive literature review on sustainable approaches to energy production from SCB, using advanced bibliometric analysis to identify key trends, leading countries, research areas, and commonly used keywords. After the bibliometric analysis, an overview of the literature was presented, addressing key topics such as energy conversion methods, future trends and expected innovations, economic aspects, and environmental impacts are presented. Furthermore, this bibliometric review explored case studies, practical applications, and challenges, highlighting successful examples and their implications using SBC.

## 2.2 METHODOLOGY

### 2.2.1 Data Collection

The Web of Science was searched for relevant articles following the methods described in previous studies (Alves *et al.*, 2025; Cavalcante *et al.*, 2024; Aires *et al.*, 2024; Dari *et al.*, 2024; Bizerra *et al.*, 2024; Melo *et al.*, 2023; Ferreira *et al.*, 2025; Simão Neto *et al.*, 2024). Initially, “sugarcane” was used as the primary search term across all research fields, retrieving 26.663 published articles. To refine the dataset for relevance to the bibliometric analysis, topic-specific keywords such as “energy” (4.240 articles), “production” (2.679 articles), and “renewable” (657 articles) were applied. These terms were selected based on their direct relevance to the study’s focus on energy production and sustainability, ensuring alignment with the research objectives. The search was restricted to 2005–2024 and limited to “Articles” and “Review articles” published in English, the dominant language in scientific literature. Specific inclusion criteria were established to ensure data relevance: only publications with at least five citations and five occurrences of key terms were selected as units of analysis. These limits were chosen to filter studies with greater significance and contributions in academic terms, ensuring a robust and representative dataset, as illustrated in Figure 2.2.

**Figure 2.2.** Methodological representation of the main terms used, search criteria and refinement processes on Web of Science, and overview of the main topics.



Source: Author (2024).

In addition, the study is divided into a second part dedicated to the literature review. This section covers the main aspects related to hydrogen production from sugarcane bagasse, including energy conversion methods, future trends, economic aspects, environmental impacts, relevant case studies, main challenges and future perspectives, as well as research and development initiatives. In this way, it offers a comprehensive and up-to-date view on the subject, contributing to the advancement of discussions and the development of sustainable solutions in the energy sector.

### ***2.2.2 Data Visualization***

Bibliometrics employs statistical methods and mathematical calculations to evaluate and measure the production and impact of scientific articles (Basile; Giacalone; Cozzucoli, 2022). By using various analytical tools and techniques, bibliometrics offers valuable insights into the dynamics and development of research fields, playing a crucial role in the management and strategic planning of academic research. VOSviewer (<http://vosviewer.com>), CiteSpace (<https://citespace.podia.com>), and Bibliometrix (<https://www.bibliometrix.org/>) were used for a quantitative, accurate data analysis. This methodology enabled the clear identification of trends and research patterns in the field of energy production from SCB. Combination of these methodological approaches ensures a comprehensive, detailed understanding of the study area, thereby contributing to a deeper grasp of research dynamics and technological development within this sector.

VOSviewer is a widely recognized tool in bibliometric analysis, known for its ability to create visual maps of scientific networks through an intuitive and effective interface (McAllister; Lennertz; Mojica, 2022). According to the VOSviewer manual, the TLS (Total Link Strong) score is calculated based on both the quantity and the quality of citations received by an article or author (Eck; Waltman, 2023). The score is determined by the number of links, with an increase in citation counts leading to a higher score (Alves Martins *et al.*, 2024). Furthermore, it adjusts the value of citations according to the impact of the citing sources, giving more weight to citations from more influential sources. The score also considers the overall strength of the citation network, providing a more comprehensive measure of an article's or author's influence (Damar; Koksalmis, 2024). Thus, the TLS score reflects the

relevance and influence of an article within its citation network. CiteSpace was used in this study for advanced bibliometric analysis, specifically to visualize and analyze trends and patterns in the scientific literature related to sustainable energy production from SCB. It helped create knowledge maps that tracked the evolution of research topics, identified emerging areas, and revealed the intellectual structures of the field.

R serves as a crucial platform for bibliometric analysis, providing a robust and flexible environment for managing and visualizing large bibliographic datasets. With specialized packages like "bibliometrix" and "ggplot2," R facilitates the extraction, cleaning, and analysis of data from sources such as Web of Science and Scopus. Its capability to create interactive graphs and maps improves data interpretation, making it more accessible and intuitive.

## 2.3 RESULTS AND DISCUSSION

### *2.3.1 Evolution of publications and citations in SCB over the last 20 years*

Results obtained in this section address RQ1:

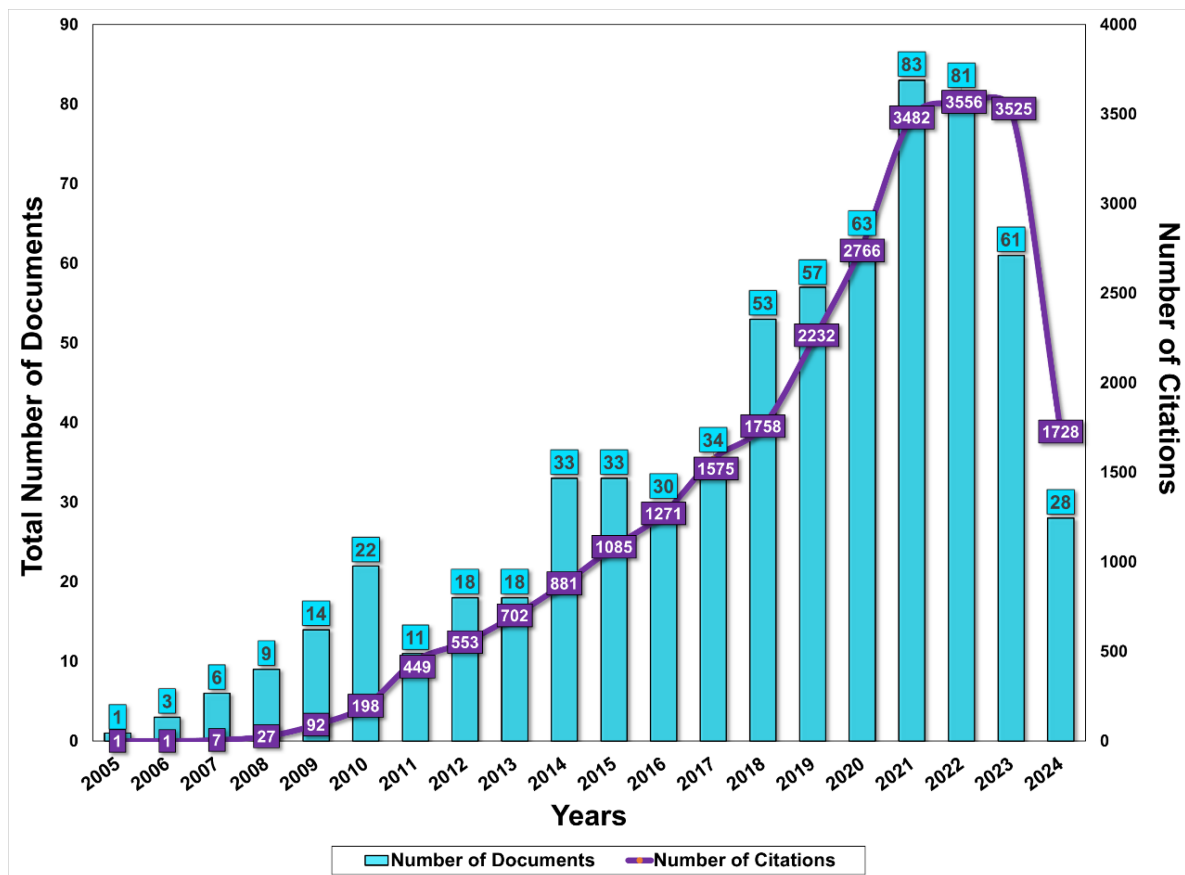
- *RQ1: How has scientific research on the development of technologies for energy production from SBC evolved?*

Figure 2.3 combines bars and lines to illustrate the annual evolution of the number of documents and citations in studies related to energy production from SCB, spanning the period from 2005 to 2024. Light blue bars represent the number of documents, while purple line reflects the number of citations. This visualization reveals a significant jump in academic production in 2010, followed by a slight decrease, and then a notable peak in 2021, marking the highest volume of document production within the analyzed timeframe.

However, despite the high number of citations, there is a sharp decline in document production from this point onwards. This trend may indicate a potential decrease in the relevance or impact of recent publications. The peaks observed in 2010 and 2021 can be attributed to several factors. The spike in document production in 2010 could be driven by

significant advances in the field, increased research funding, or renewed interest in the topic due to global events or relevant discoveries. The peak in 2021, on the other hand, could be associated with an increase in international collaboration, greater access to research technologies, or the urgent need to address contemporary challenges that require more in-depth scientific investigation.

**Figure 2.3** Annual scientific production and citations indexed on Web of Science from 2005 to 2024, related to energy production from sugarcane bagasse (SCB).



**Source:** Author (2024).

However, despite the high number of citations, there is a sharp decline in document production from this point onwards. This trend may indicate a potential decrease in the relevance or impact of recent publications. The peaks observed in 2010 and 2021 can be attributed to several factors. The spike in document production in 2010 could be driven by significant advances in the field, increased research funding, or renewed interest in the topic due to global events or relevant discoveries. The peak in 2021, on the other hand, could be

associated with an increase in international collaboration, greater access to research technologies, or the urgent need to address contemporary challenges that require more in-depth scientific investigation.

On the other hand, the decline in paper production after 2021, despite the high number of citations, may suggest a saturation point in the field, indicating that core questions have been largely explored. Alternatively, this decline may reflect a shift in research focus towards emerging areas, particularly the increasing use of artificial intelligence and machine learning to optimize processes. These technological advances not only offer new perspectives but also redefine existing paradigms, signaling a continuous evolution in the scope of scientific research. This shift makes it imperative that scholars adapt their approaches by integrating these innovations to promote a more holistic and dynamic development within the field. Furthermore, the Covid 19 pandemic may also reflect changes in publishing practices, with an increasing emphasis on research quality rather than quantity of publications. The years 2020, 2021, and 2022 had the highest number of citations, with peaks of 2,766, 3,482, and 3,556 citations, respectively. These peaks suggest that papers published in these years were particularly influential and widely referenced in subsequent research, underscoring their significant impact on the academic community.

### ***2.3.2 Collaboration network between countries/regions***

Results in this section respond to RQ2:

- *RQ2: Which countries/regions have contributed significantly to the use of sugarcane bagasse, and what are their advantages?*

Figure 2.4(A) presents a network visualization generated using CiteSpace, a tool specialized in analyzing scientific literature and citation patterns. In this visualization, each circle represents a country, with the circle's size indicating the level of research activity or data associated with that country. The lines connecting the circles illustrate collaborations or relationships between countries, with the colors of the circles and lines varying according to the years covered, as indicated in the legend on the left, spanning from 2008 to 2021. On the other hand, Figure 2.4(B) combines a circular bar chart with a chord diagram. The outer ring is

segmented by different countries, with the width of each segment representing the volume of data or publications from each country. This figure provides a clear view of the global distribution of research and the international collaboration networks within the field.

Brazil stands out as the world's largest producer of sugarcane, excelling in sugar and ethanol production. In 2010, the country recorded an impressive production of 719.1 million tons, accounting for 43% of global production (Hofsetz; Silva, 2012; Travaini *et al.*, 2013). Sugarcane bagasse in Brazil is primarily used for electricity generation, supplying energy to mills (Hofsetz; Silva, 2012). The vast availability of this biomass as a byproduct of the sugar and ethanol industry drives initiatives for renewable energy, such as the cogeneration of electricity in mills, biofuel production, and the creation of higher-value products, such as bioplastics and green chemicals (Bizzo *et al.*, 2014).

Brazil has consistently invested in renewable energy, promoting innovation; however, it faces the challenge of managing bagasse disposal. Studies, such as those conducted by (Mubarak *et al.*, 2024). point to the potential of renewable and biodegradable materials, such as natural fibers and biopolymers, to replace petroleum-based plastics, thus reducing environmental impacts such as carbon footprints and waste disposal. Sugarcane bagasse is seen as a promising resource, which could be used as an adsorbent for heavy metals and as reinforcement in polymers (Mubarak *et al.*, 2024).

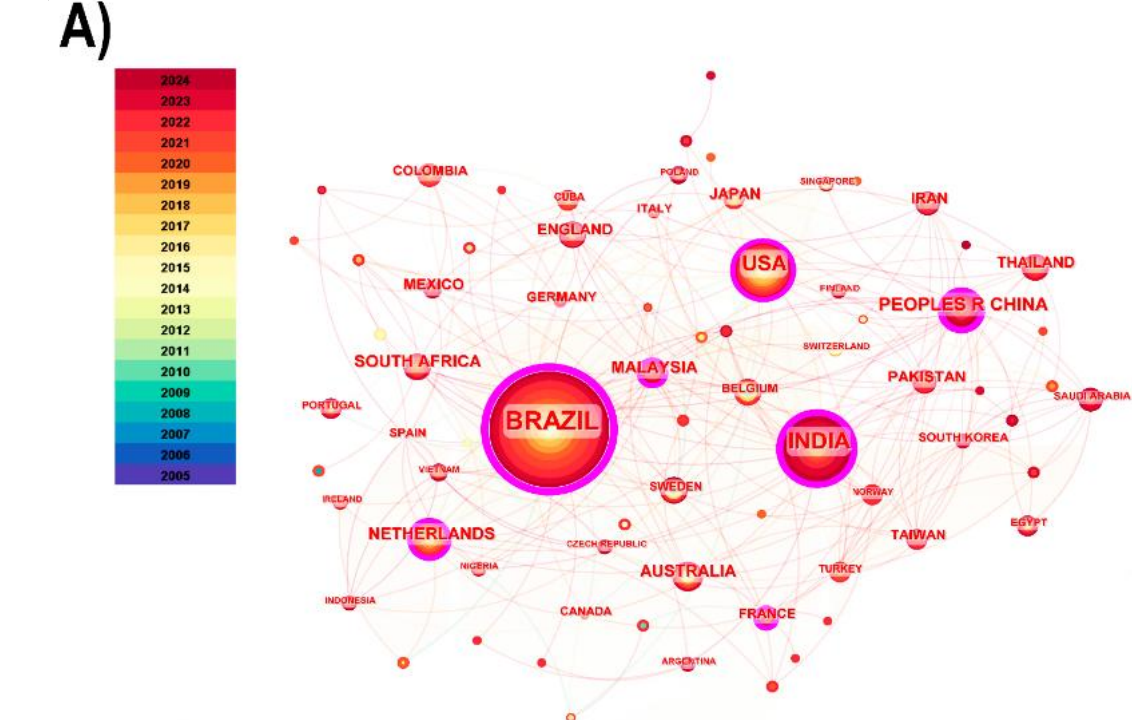
India, the second-largest sugarcane producer globally, faces unique challenges that shape its approach to research on bagasse. Historically, bagasse has been widely used in the paper and pulp industry due to the growing national demand for these products (Kasiviswanathan KS, 1998; Rainey; Covey, 2016; Savastano *et al.*, 2016). The Indian government has directed its policies and incentives towards the development of biogas and bioethanol, foreseeing a significant market for these biofuels in the future.

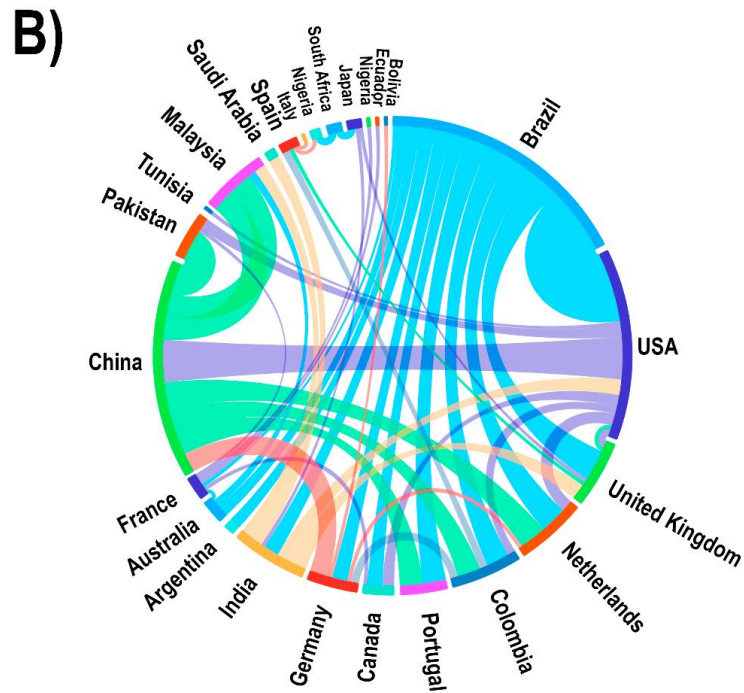
Chemical compounds such as xylitol, succinic acid, and lactic acid have been recognized as important products for the implementation of biorefineries in the country (Konde *et al.*, 2021). In China, one of the advantages is that research on SBC is driven by national circular economy strategies and carbon neutrality goals (Mathews; Tan, 2011). Although sugarcane cultivation is not as extensive as in Brazil, the country heavily invests in cutting-edge technologies, such as the use of bagasse in the production of nanocellulose, biochar, and

biohydrogen (Cheng; Zhu, 2016; Huang, Jiangfeng *et al.*, 2020a, b; Mahmud, Md. Zobair Al; Mobarak; Hossain, 2024).

In the United States, the use of SBC for energy production is highly relevant, particularly in states such as Louisiana, where it is grown on a large scale (Pimentel; Patzek, 2007). It has an important environmental characteristic that is considered an advantage, as the carbon dioxide CO<sub>2</sub> emissions from its combustion are offset by the carbon captured during the growth of the cane (Mann *et al.*, 2016a). The combustion of bagasse is also used for steam and electricity generation in ethanol plants, which reduces dependence on fossil fuels, minimizing agricultural waste (Pimentel; Patzek, 2007). Furthermore, research initiatives have explored the conversion of bagasse into valuable bioproducts, such as bioplastics (Hiranobe *et al.*, 2024a).

**Figure 2.4** International collaboration on (SCB) research. A) Collaboration clusters over time, highlighting Brazil, USA, and India. B) Collaborating countries visualized through a chord diagram





**Source:** Author (2024).

### 2.3.3 Network of Most Searched Keywords

The information contained in this section addresses the third QR:

- *RQ3: What are the most important keywords mentioned in the search?*

Figure 2.5(A) presents the results of a network analysis conducted using VOSviewer©, identifying four major clusters. The analysis was based on data obtained from the Web of Science, with a minimum occurrence of five for each keyword as the counting criterion. Out of a total of 3,326 keywords, 256 met this criterion. In Figure 2.5(A), the analysis highlights a critical blue cluster that is central to the network. This cluster is anchored by the keyword “sugarcane bagasse,” which is closely linked to other key terms such as “bioethanol,” “ethanol-production,” and “fermentation,” indicating the primary focus of the study. SCB is a residual material generated during the sugarcane juice extraction process, which is typically carried out using two main methods: diffusion and milling (Fioranelli; Bizzo, 2023). After the juice is extracted, the remaining SCB is usually utilized in the industry, often as fuel in boilers to

generate steam. However, numerous studies have demonstrated that SCB can also be effectively used in bioethanol production, thereby enhancing the efficiency of the entire process through the reuse of this byproduct (Parascanu *et al.*, 2021; Toscano Miranda *et al.*, 2021).

The second most prominent cluster, represented in yellow, centers around the keyword “biomass,” which is the second most cited term in the analysis. This cluster is linked to keywords such as “biochar,” “combustion,” and “fuel.” Numerous studies explore various applications for different types of biomasses and SCB is no exception. Among the possibilities studied, the production of biochar stands out. Biochar is a charcoal-like substance produced through the pyrolysis of biomass, and SCB is considered an excellent option for developing this energy product (Sharma, Pramila *et al.*, 2024). The third significant cluster is led by the keyword “renewable energy,” represented by the green color. This cluster is linked to other important keywords such as “biogas production,” “hydrogen production,” and “electricity.” This connection highlights the ongoing research into new renewable sources that enable better utilization of biomass for energy production, with a focus on generating biogas, producing hydrogen, and subsequently applying these for electricity generation. The fourth key cluster, represented by the main keyword “sugarcane,” is strongly associated with terms such as “energy,” “bioenergy,” and “life-cycle assessment.”

Figure 2.5(B) shows a network visualization map focused on the period from 2020 to 2024, highlighting keywords to track trends over time. The analysis reveals shifting research interests within the field: In 2020, the keywords “sugarcane” and “energy” were more heavily researched, reflecting a broad focus on the potential of SCB as a renewable energy source. In 2022, the keyword “bioethanol” gained prominence, indicating a growing focus on the use of biomass, particularly SCB, for bioethanol production. Between 2023 and 2024, there was a noticeable shift in research trends towards the effective use of SCB, highlighting the need to explore new methods and techniques to optimize the use of biomass for energy production. This keyword evolution analysis provides a critical perspective on the evolution of sugarcane biomass research. It highlights not only the growing interest in specific technologies, such as bioethanol production, but also the field’s adaptation to emerging demands for sustainability and efficiency in the use of renewable resources.

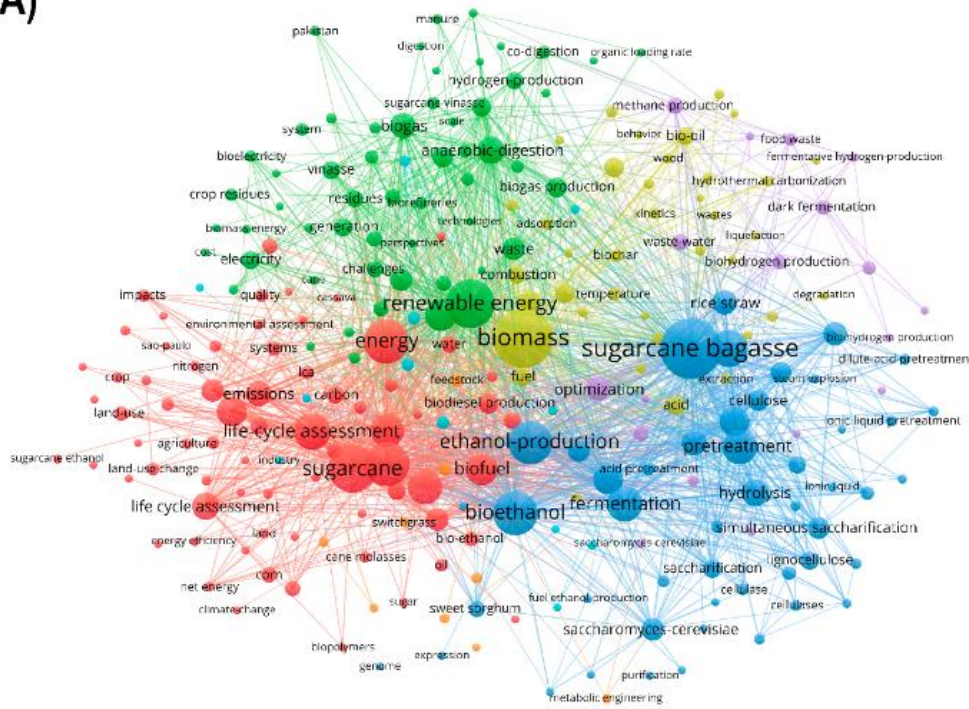
Considering the comprehensive analysis from the network visualizations, several key areas of interest emerge for future research discussions. Firstly, the increasing focus on

“sugarcane bagasse” and related keywords such as “bioethanol,” “ethanol production,” and “fermentation” highlights its significant potential in bioethanol production. Future studies could aim to optimize conversion processes to improve yield and efficiency. Additionally, the “biomass” cluster points to the potential of using SCB for biochar production.

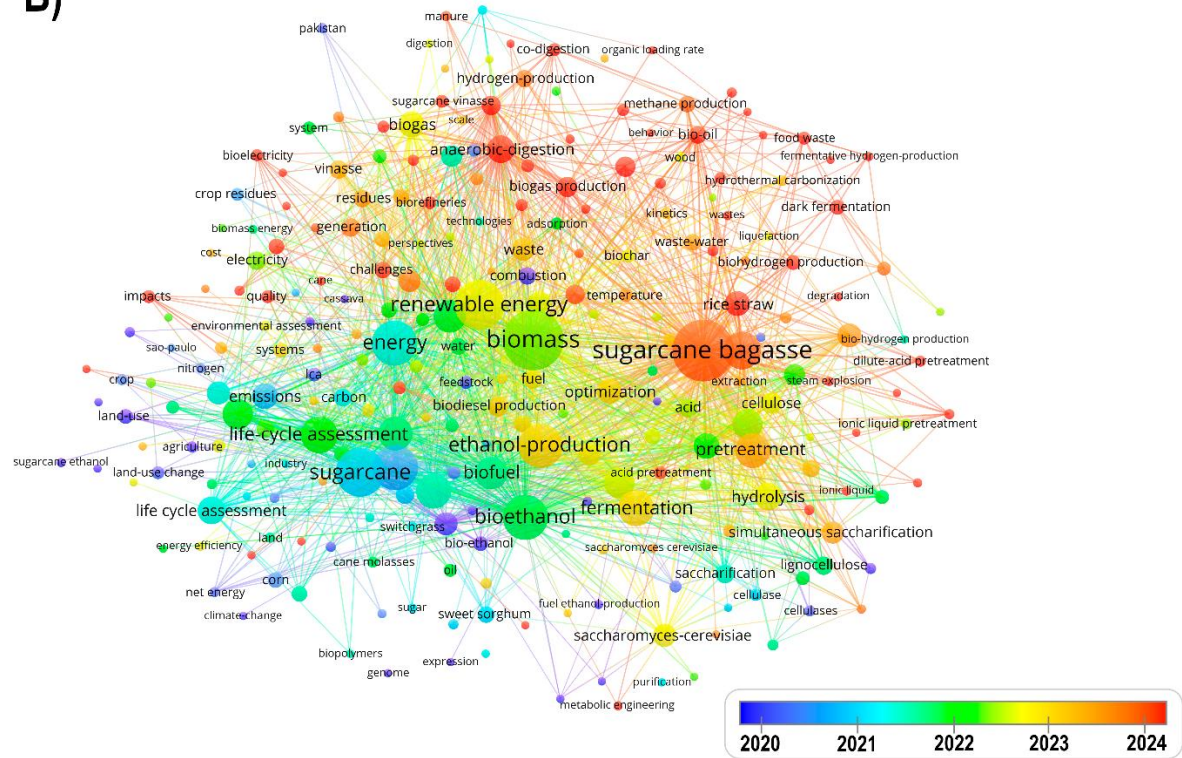
Research into the pyrolysis process and ways to optimize it for producing high-quality biochar could provide valuable insights. Moreover, the cluster around “renewable energy” shows growing interest in biogas and hydrogen production. Investigating how SCB can be integrated into these systems could result in more sustainable energy solutions. Finally, the shift in keyword prominence from “sugarcane” and “energy” to “bioethanol” and “sugarcane bagasse” suggests evolving research trends. Future discussions should focus on these trends, emphasizing innovative approaches to fully harness the energy potential of sugarcane byproducts while ensuring sustainability and efficiency.

**Figure 2.5** Map of the main keywords and their connections, considering a minimum of 5 (five) occurrences. A) Keyword mapping based on different clusters. B) Annual average mapping of keyword occurrences.

A)



B)



Source: Author (2024).

Table 2.1 lists the twenty most frequent keywords in the dataset, with “Sugarcane bagasse” (164), “Biomass” (158), “Renewable energy” (112), “Sugarcane” (108), and “Energy” (95) being the top five. The high total link strength of these keywords underscores their centrality and interconnectedness in the research network, reflecting a strong focus on sustainable energy and biomass utilization. This highlights the critical role of SCB and related biomass in advancing renewable energy technologies, emphasizing the need for continued innovation.

Based on the Bibliometrix analysis, relationships between countries, keywords, and university affiliations (n=10) were observed, as shown in Figure 2.6. Brazil is the leading country, closely linked to the top 10 keywords. The University of São Paulo demonstrates significant engagement among institutions, connecting with nearly all primary keywords. Dominant terms like “biomass,” “renewable energy,” and “sugarcane” highlight an intense

research focus on sustainability. This visualization underscores the global emphasis on renewable energy and SCB's critical role.

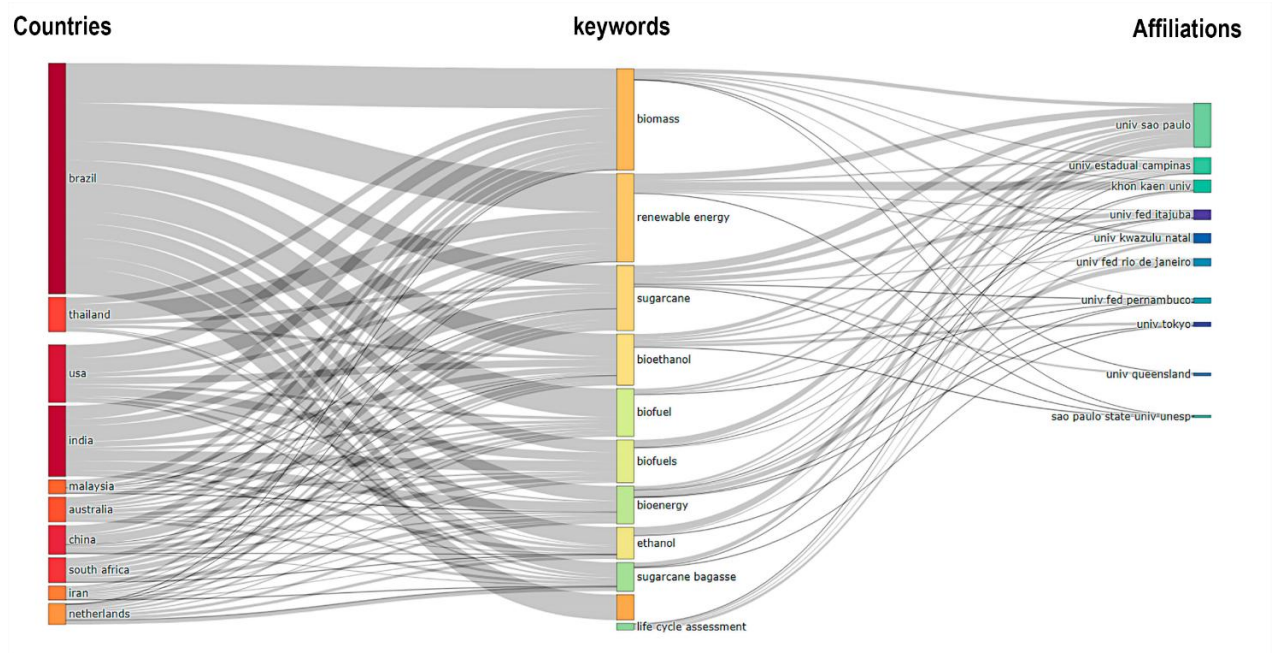
**Table 2.1** Ranking 20 Keywords with Highest Frequency in Analyzed Articles.

<b>Ranking</b>	<b>Keywords</b>	<b>Occurrence</b>	<b>TLS</b>	<b>Rank</b>	<b>Keyword</b>	<b>Occurrence</b>	<b>TLS</b>
<b>1</b>	Sugarcane bagasse	164	1065	<b>11</b>	Fermentation	43	404
<b>2</b>	Biomass	158	977	<b>12</b>	Biofuels	43	135
<b>3</b>	Renewable energy	112	677	<b>13</b>	Pretreatment	40	313
<b>4</b>	Sugarcane	108	619	<b>14</b>	Bioenergy	38	242
<b>5</b>	Energy	95	610	<b>15</b>	Bagasse	36	284
<b>6</b>	Ethanol	95	604	<b>16</b>	Biofuel	34	251
<b>7</b>	Bioethanol	93	623	<b>17</b>	Bioethanol production	32	206
<b>8</b>	Ethanol-Production	91	654	<b>18</b>	Enzymatic-Hydrolysis	29	238
<b>9</b>	Lignocellulosic Biomass	69	475	<b>19</b>	Sustainability	29	162
<b>10</b>	Life-cycle Assessment	66	478	<b>20</b>	Life cycle assessment	29	153

**TLS: Total link strength**

**Source:** Author (2024).

**Figure 2.6** Three-field plot with its relationships between countries, keywords, and affiliations.



**Source:** Author (2024).

CiteSpace cluster analysis identified six distinct themes, each characterized by a unique set of keywords. Three of these themes were particularly relevant to the study. Figure 2.7(A) provides a timeline visualizing these prominent themes, displaying key keywords clustered together and their frequency over time. This graphical representation, generated through CiteSpace data analysis, allows for tracking the evolution of these themes and the interaction between keywords across the years. The identified themes and their associated keywords are described below:

1. Lignocellulosic Biomass: This theme focuses on keywords such as “sugarcane bagasse,” “bioethanol production,” “cellulose,” and “enzymatic hydrolysis.” It emphasizes the role of biomass in the study of lignocellulosic materials, which are crucial for producing biofuels and sustainable chemicals. These plant-derived materials offer an increasingly viable alternative to conventional fossil fuels, making this transition essential for promoting planetary sustainability.
2. Lignocellulosic materials: Characterized by terms such as “renewable energy,” “bioenergy,” and “sustainability,” this theme centers on the efficient use of natural resources for clean energy generation. The use of materials such as SCB is vital for

transitioning to a greener energy matrix and reducing dependence on fossil fuels. Additionally, these materials offer a sustainable solution for agricultural waste management, promoting a circular economy and minimizing environmental impacts. In summary, exploring lignocellulosic materials for bioenergy is crucial for a more sustainable future.

3. Land and water management: This theme explores the relationship between water and terrestrial environments, with a focus on the sustainable management of water resources. Research under this theme includes topics such as biodiesel production from agricultural sources, solid waste management, and their impacts on water quality, highlighting the complexity of interactions between these elements. Keywords such as “biodiesel,” “solid waste,” “agriculture,” and “oil” underscore this diversity.
4. Optimizing Bioenergy Processes: This theme encompasses several subtopics, including “methane production,” “life-cycle basis,” “biomass residue,” and “dual kinetic-en.” The focus here is on optimizing the processes involved in bioenergy production. “Methane production” emphasizes efficient biogas generation, while “life-cycle basis” ensures sustainability assessments throughout the production stages. “Biomass residue” highlights the utilization of agricultural waste, and “dual kinetic-en” explores advanced kinetics in these processes. Together, these approaches aim to maximize energy efficiency, minimize environmental impacts, and advance the development of sustainable energy solutions.

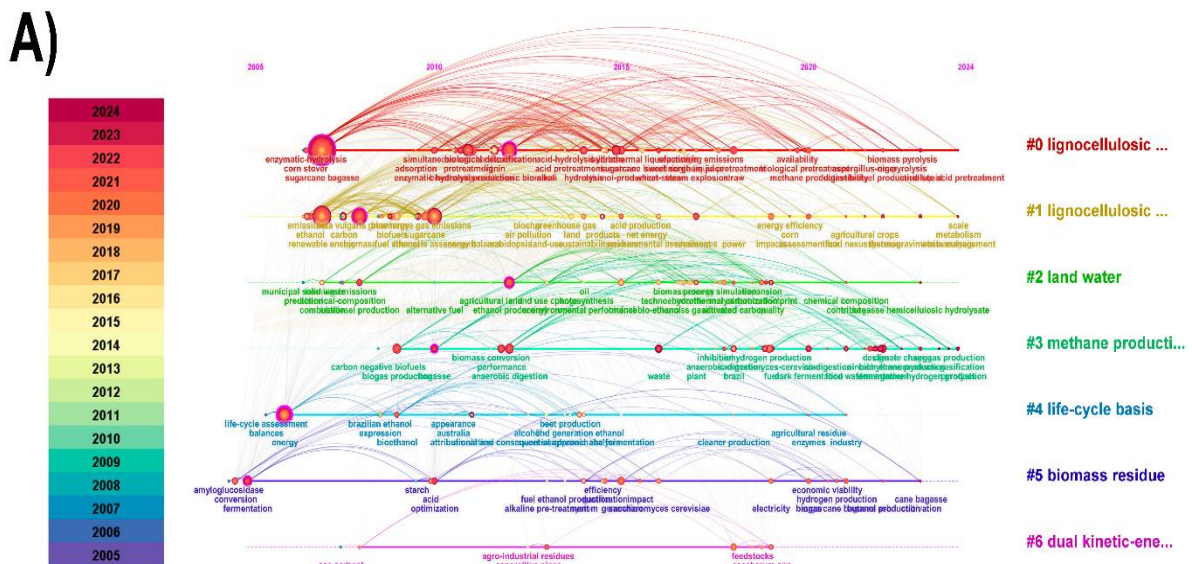
Figure 2.7(B) presents clusters with their respective densities, highlighting the main keywords. The variations in color allow us to identify the periods during which these keywords were most frequently used. For example, the keyword “sugarcane bagasse” is represented in red, indicating its peak frequency between the years 2022 and 2024, as shown in the legend. As mentioned earlier, this increase stems from the growing interest in reusing SCB for energy production. The figure also enables visualization of the keywords that have been consistently repeated over the years.

Figure 2.7(C) shows a three-field graph generated by the Bibliometrix platform, which allows for the analysis of keyword evolution over three different time periods. The plot shows that from 2005 to 2017, keywords were primarily concentrated around “energy” and other terms such as “ethanol production,” and “combustion.” However, there was a shift in the period from

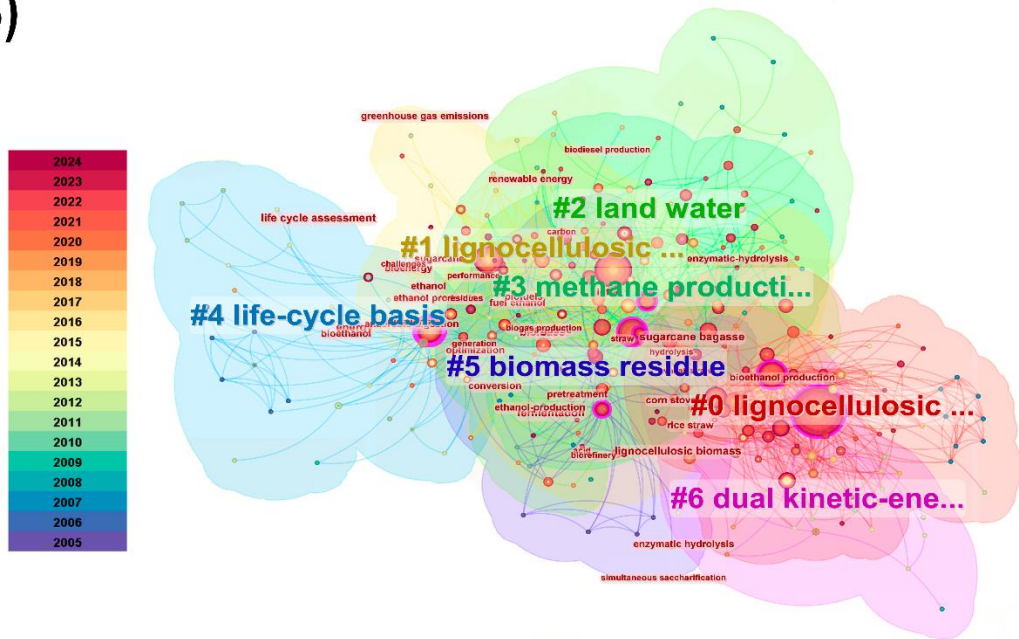
2018 to 2021, with “sugarcane bagasse” and “ethanol production” becoming more prominent. In the most recent period, from 2022 to 2024, the most frequently cited keywords focus on the relationship between “sugarcane bagasse” and “biomass.”

The factors influencing the transition of keywords include technological advancements, public policies aimed at sustainability, the demand for renewable energy sources, and the impact of environmental regulations. Furthermore, changes in research funding and academic interest play a significant role. Future trends are likely to continue prioritizing sustainability and efficiency, with an emphasis on emerging technologies for biomass conversion, green hydrogen production, integration with solar and wind energy, and the development of high-value bioproducts. Continuous bibliometric analysis can help identify emerging themes and guide future research efforts.

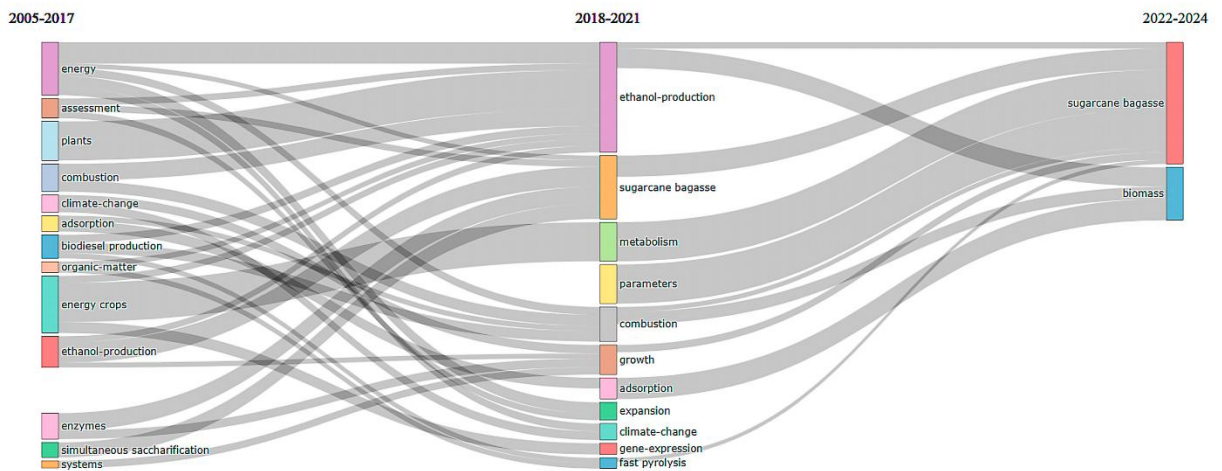
**Figure 2.7** A) Evolution of keywords across clusters. B) Cluster map showing the occurrence density of keywords according to their annual progression. C) Three-field plot displaying correlations between keywords and their temporal evolution.



B)



C)



Source: Author (2024).

### 2.3.4 Ranking of the Most Cited Journals

The most-cited articles primarily focus on the areas of energy fuel and sustainable scientific technologies.

Table 2.2 ranks the ten most influential scientific articles on energy production, highlighting their significance and contribution to the field. The top-ranked article, with 3,879 citations, is titled “Biofuels from microalgae—A review of technologies for production, processing, and extractions of biofuels and co-products,” published in 2010. This article provides an extensive overview of biofuel production technologies from microalgae, including the use of open pond systems and closed photobioreactors, emphasizing the potential of microalgae as a substantial alternative to fossil fuels (Brennan; Owende, 2010). The second most cited article, “Strategies to reduce the global carbon footprint of plastics,” published in 2019, has garnered significant attention for its analysis of the impact of plastics on global greenhouse gas emissions. The article explores various strategies to reduce these emissions, compiling emission data for ten conventional and five bio-based plastics. It examines different scenarios, such as the use of renewable energy and the replacement of traditional plastics with biomass-based alternatives (Zheng; Suh, 2019).

The third-ranked article, titled “Methane production from lignocellulosic agricultural crop wastes: A review in context to second generation of biofuel production,” was published in 2012. This article examines methane production from lignocellulosic agricultural residues through bio methanation, comparing its potential with bioethanol generation (Chandra; Takeuchi; Hasegawa, 2012). The fourth-ranked article, “Sustainable bio-ethanol production from agro-residues: A review” published in 2015, offers a comprehensive exploration of sustainable bioethanol production from lignocellulosic agricultural byproducts. It provides a detailed overview of the processes involved, highlighting key challenges and opportunities within this emerging field (Gupta; Verma, 2015). The fifth-ranked article, “Ethanol as an alternative fuel from agricultural, industrial and urban residues,” published in 2007, investigates the potential of ethanol production from various waste streams. It addresses the critical need for renewable energy sources and detail the key processes involved in transforming these residues into viable biofuels (Prasad; Singh; Joshi, 2007).

The sixth most cited article, “Enhanced hydrogen production from biomass within situ carbon dioxide capture using calcium oxide sorbents,” published in 2008, investigates hydrogen production from biomass via steam gasification, incorporating calcium oxide (CaO) for efficient carbon dioxide capture. The study explores the underlying mechanisms and proposes strategies to optimize this promising process for sustainable hydrogen generation (Florin;

Harris, 2008). The seventh-ranked article, “A review on bio-oil production from biomass by using pyrolysis method,” published in 2012, reviews the thermal conversion of biomass into bio-oil through pyrolysis, focusing on biomass characterization, reactor designs, product formation, and techniques for improving bio-oil quality (Isahak *et al.*, 2012). The eighth-ranked article, “A mini review on renewable sources for biofuel,” published in 2014, examines the three generations of biomass—food-based, lignocellulosic, and algae—emphasizing their roles in meeting the growing global energy demand (Ho; Ngo; Guo, 2014). The ninth-ranked article, “Bioconversion of Sugarcane Biomass into Ethanol: An Overview about Composition, Pretreatment Methods, Detoxification of Hydrolysates, Enzymatic Saccharification, and Ethanol Fermentation,” published in 2012, provides an overview of the conversion of sugarcane biomass into ethanol, detailing each stage of the production process (Canilha *et al.*, 2012). The tenth-ranked article, “Anaerobic digestion of vinasse from sugarcane ethanol production in Brazil: Challenges and perspectives,” published in 2015, highlights the potential of anaerobic digestion to convert vinasse—a byproduct of the production of sugarcane ethanol—into biogas. The study delves into the technical challenges encountered during this process and explores potential solutions (Moraes; Zaiat; Bonomi, 2015). Overall, this ranking of the most cited articles reveals a predominance of review articles, underscoring their critical role in synthesizing knowledge within the field. The most cited papers focus primarily on energy fuels and sustainable scientific technologies, reflecting the importance of these areas in contemporary research.

**Table 2.2** Ranking of the 10 most cited articles related to energy production from sugarcane.

Ranking	Title	Authors	Source	Reviews	Year published	Freq
1	Biofuels from microalgae-A review of technologies for production, processing, and extractions of biofuels and co-products (Brennan; Owende, 2010)	Brennan, Liam; Owende, Philip	RENEWABLE & SUSTAINABLE ENERGY REVIEWS	Yes	2010	3,879
2	Strategies to reduce the global carbon footprint of plastics (Zheng; Suh, 2019)	Zheng, Jijia; Suh, Sangwon	NATURE CLIMATE CHANGE	No	2019	582
3	Methane production from lignocellulosic agricultural crop wastes: A review in context to second generation of biofuel production	Chandra, R.; Takeuchi, H.; Hasegawa, T.	RENEWABLE & SUSTAINABLE ENERGY	Yes	2012	544

(Chandra; Takeuchi;  
Hasegawa, 2012)

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4	Sustainable bio-ethanol production from agro-residues: A review (Gupta; Verma, 2015)	Gupta, Anubhuti; Verma, Jay Prakash	RENEWABLE & SUSTAINABLE ENERGY REVIEWS	Yes	2015	496
5	Ethanol as an alternative fuel from agricultural, industrial and urban residues (Prasad; Singh; Joshi, 2007)	Prasad, S.; Singh, Anoop; Joshi, H. C.	RESOURCES CONSERVATION AND RECYCLING	No	2007	440
6	Enhanced hydrogen production from biomass with in situ carbon dioxide capture using calcium oxide sorbents (Florin; Harris, 2008)	Florin, Nicholas H.; Harris, Andrew T.	CHEMICAL ENGINEERING SCIENCE	No	2008	407
7	A review on bio-oil production from biomass by using pyrolysis method (Isahak <i>et al.</i> , 2012)	Isahak, Wan Nor Roslam Wan; Hisham, Mohamed W. M.; Yarmo, Mohd Ambar; Hin, Taufiq-yap Yun	RENEWABLE & SUSTAINABLE ENERGY REVIEWS	Yes	2012	404
8	A mini review on renewable sources for biofuel (Ho; Ngo; Guo, 2014)	Ho, Dang P.; Huu Hao Ngo; Guo, Wenshan	BIORESOURCE TECHNOLOGY	Yes	2014	320
9	Bioconversion of Sugarcane Biomass into Ethanol: An Overview about Composition, Pretreatment Methods, Detoxification of Hydrolysates, Enzymatic Saccharification, and Ethanol Fermentation (Canilha <i>et al.</i> , 2012)	Canilha, Larissa; Chandel, Anuj Kumar; dos Santos Milessi, Thais Suzane; Fernandes Antunes, Felipe Antonio; da Costa Freitas, Wagner Luiz; Almeida Felipe, Maria das Gracias; da Silva, Silvio Silverio	JOURNAL OF BIOMEDICINE AND BIOTECHNOLOGY		2012	318
10	Anaerobic digestion of vinasse from sugarcane ethanol production in Brazil: Challenges and perspectives (Moraes; Zaiat; Bonomi, 2015)	Moraes, Bruna S.; Zaiat, Marcelo; Bonomi, Antonio	RENEWABLE & SUSTAINABLE ENERGY REVIEWS		2015	278

Source: Author (2024).

### 2.3.5 Main Areas

The data obtained in this section addresses the fourth RQ:

- *RQ4: What are the emerging research areas in the field?*

Because of the ongoing advancements in energy conversion technologies by using SCB as a feedstock, evaluating prominent research areas to identify trends and gaps in the field is crucial (Paudel *et al.*, 2024; Sarker; Azam; Bonanomi, 2017; Wani *et al.*, 2023). Figure 2.8(A) highlights the promising research areas and the corresponding percentage of published articles in each area. Figure 2.8(B) illustrates the distribution of published articles in different research areas.

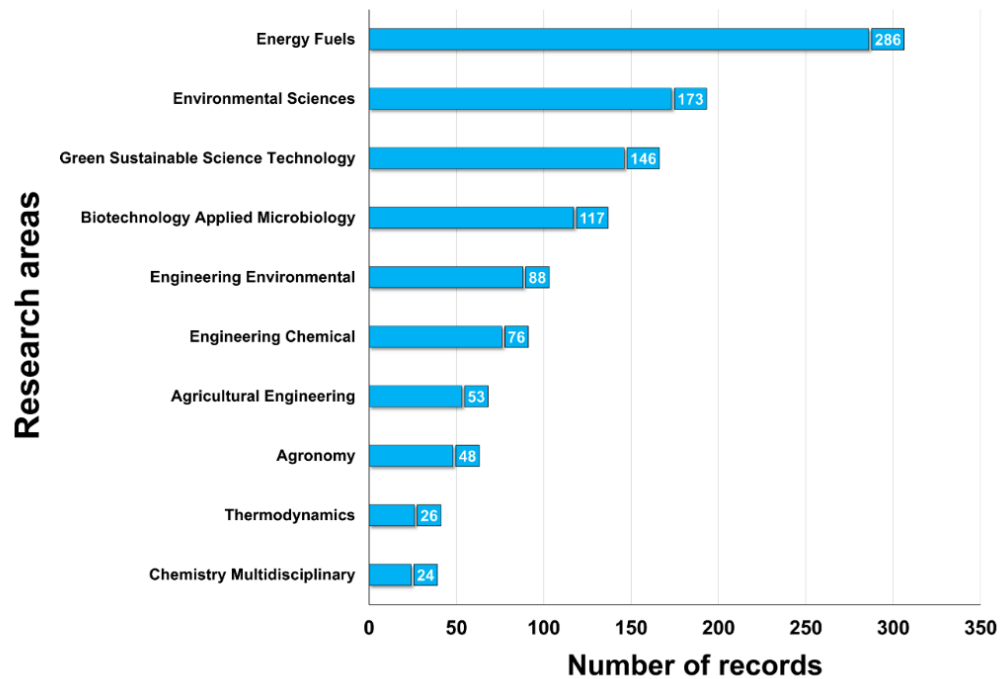
The analysis reveals that the “energy fuels” category has 286 publications, accounting for 28% of the articles in the chosen database. This indicates the significant role that advancements in the synthesis of liquid fuels play in this field. Among the notable works published in this area is “Potential for Bioenergy Production from Sugarcane in China,” published in 2014. This article reviews the sugarcane industry in China, identifies underutilized resources such as sugarcane fiber and bagasse, discusses policies that could support renewable energy production, and outlines priorities for future research (Ling *et al.*, 2014).

Following “energy fuels,” the “environmental sciences” category has 173 documents, representing 17% of the articles in the evaluated database. A significant contribution in this area is the study by Tsiropoulos *et al.* (2014), which conducted a comparative analysis of ethanol production in Brazil and India (Tsiropoulos *et al.*, 2014). The present study highlights the significant collaboration involved in using different feedstocks in diverse social contexts and underscores its relevance to the environmental sciences research area.

The research areas with the lowest number of publications are “Agronomy,” “Thermodynamics,” and “Chemistry Multidisciplinary,” with 28, 26, and 24 publications, respectively. In contrast, a more promising area is “Green Sustainable Technology Science,” which has 146 publications, accounting for 14% of the articles used to build the database. In this field, Gupta and Verma. (2014) published a study evaluating the environmental compatibility of bioethanol production from agricultural waste (Gupta; Verma, 2015).



B)



Source: Author (2024).

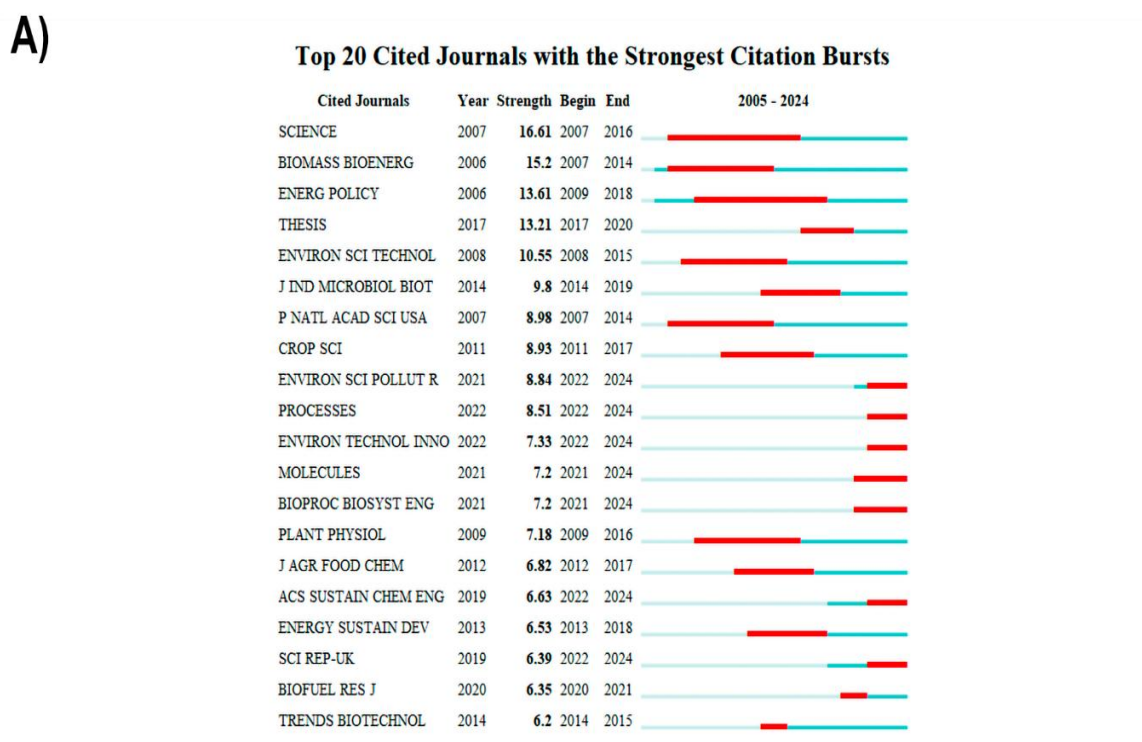
### 2.3.6 Ranking of the Main Journals

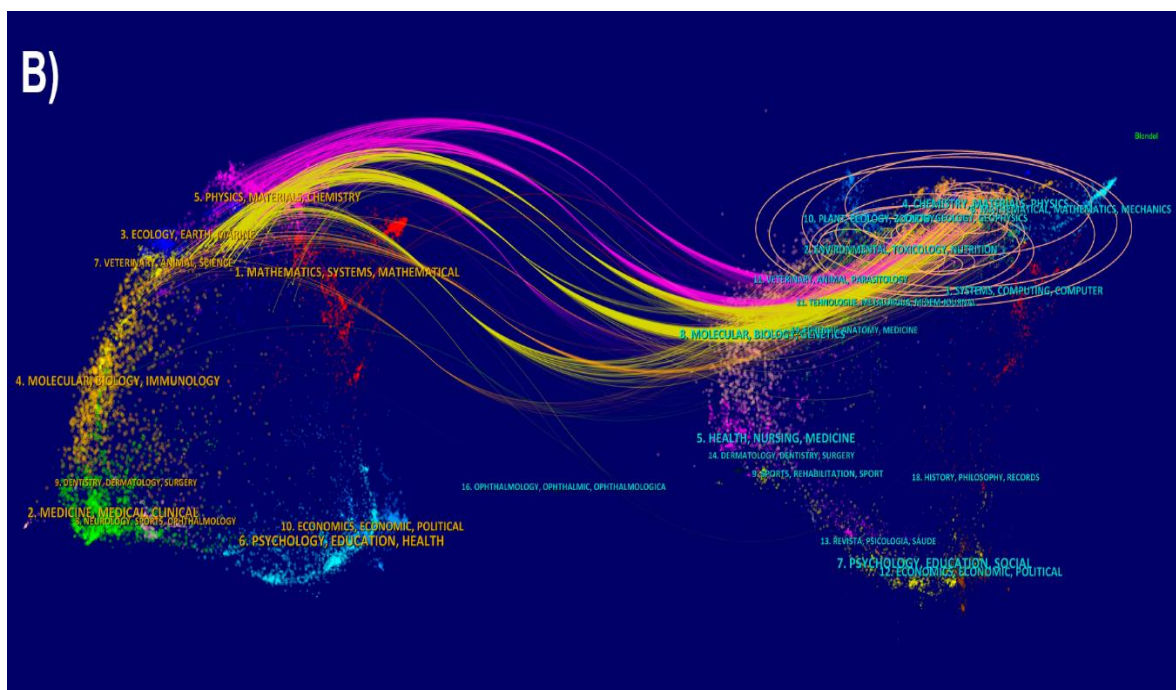
A critical metric for assessing research impact is the identification of the most frequently cited journals. This study analyzed the top 20 journals by citation ranking, focusing on their Impact Factor (IF). Figure 2.9(A) illustrates these journals, with red indicating periods of high citation frequency and blue representing lower citation activity. The journal *Science* (IF: 16.61), particularly its 2007 publication, stands out for its high citation relevance in the field during its early years. *Biomass and Bioenergy* (IF: 15.2) ranks second, showing a significant citation peak after initial low activity and maintaining stable citations thereafter. *Energy Policy* (IF: 13.61), with influential work published in 2006, holds the third position. These journals highlight key contributions and trends in the field over time. Similar to *Biomass and Bioenergy*, *Energy Policy* had a lower number of citations initially, but the citation data indicate a consistently high and stable citation rate in the years that followed (Rodrigues *et al.*, 2024).

Figure 2.9(B) presents an overlay of the evolution of journals focused on energy production from SCB between 2005 and 2024. The map illustrates citation paths within various study areas and the correlations between them. Fields such as Physics, Materials Science, and Chemistry are particularly prominent in the context of biomass energy production and are connected to related disciplines such as Molecular Biology and Immunology (Cavalcante *et al.*, 2024).

This analysis provides a bibliometric perspective on the impact of these journals within the broader field of sustainable energy production, highlighting the annual relevance of each journal. This connection allows researchers to assess the impact and relevance of their work and gain insights into the directions the scientific community is taking regarding the conversion of SCB into green energy (Dari *et al.*, 2024).

**Figure 2.9** (A) Top 20 journals with the highest impact by number of citations, highlighting the year, impact factor, and the span of citation activity. (B) Dual-map overlay of journals (blue-named) in hydrogen research, generated using CiteSpace. Labels highlight different research aspects within the field.





Source: Author (2024).

## 2.4 HYDROGEN PRODUCTION FROM SUGARCANE BAGASSE

Researchers are developing more efficient electrocatalysts for the hydrogen evolution reaction (HER), thus increasing hydrogen production as a clean energy source. (Qi *et al.*, 2024), showed that the NiCoP/CoP<sub>2</sub>-SCBC (Sugarcane Bagasse Carbon) composite outperforms other catalysts in HER. This is due to the hydroxyl groups in SCBC, which help distribute metal ions and increase Ni<sup>2+</sup>, accelerating the reaction. Additionally, SCBC introduces defects and phosphorus, boosting active sites. The study emphasizes the role of biomass-derived carbon in supporting non-precious metal catalysts for HER (Qi *et al.*, 2024).

According to Ji *et al.* (2021), a catalyst composed of MoS<sub>2</sub> (molybdenum disulfide) supported on carbon derived from SBC was proposed as a low-cost and highly efficient alternative for hydrogen production. The catalyst was synthesized through calcination and hydrothermal processes, resulting in MoS<sub>2</sub> nanosheets with good conductivity and exposed active sites. Tests showed that the MoS<sub>2</sub>/SCBC composite exhibited excellent performance in the (HER), with high efficiency, opening a new approach to sustainable electrocatalysis.

Another approach to consider is the biological conversion of SBC into H<sub>2</sub> through anaerobic fermentation (Rashidi *et al.*, 2024). In this process, microorganisms degrade the sugars present in the bagasse, releasing hydrogen as one of the metabolic products (Damiri *et al.*, 2024). Other studies focus on hydrogen production, such as the work by Chen *et al.* (2023), where dark fermentation with (*Clostridiales*) and pretreated SBC was enhanced by adding food waste to optimize the C/N ratio and biochar's with iron and manganese (Fe·Mn·SB) to intensify the process. These modifications resulted in a 113.75% increase in hydrogen production and a 32.48% improvement in hydrogen yield.

The study demonstrated an efficient process and emphasized the role of metallic biochar's, offering a foundation for future research (Chen, Sheng-Jie *et al.*, 2023). The use of SBC to produce H<sub>2</sub> represents an excellent energy alternative, reducing greenhouse gas emissions and providing value to waste from the ethanol and sugar production chain (Vargas; Flórez-Orrego; de Oliveira Junior, 2023).

## 2.5 METHODS OF ENERGY CONVERSION AND TECHNOLOGIES

This section presents the leading technologies, providing an overview of energy conversion methods, which include direct combustion, gasification, pyrolysis, fermentation, and anaerobic digestion. Table 2.3 summarizes the advantages and disadvantages of each process. The subsequent topics further enrich the discussion, offering additional details.

**Table 2.3** Advantages and disadvantages of energy conversion methods.

Method	Advantages	Disadvantages
<b>Direct Combustion</b>	<ul style="list-style-type: none"> <li>- Use of wood residues (e.g., sawdust) for energy generation.</li> <li>- Reduces net carbon emissions compared to fossil fuels.</li> <li>- High efficiency and simplicity of operation.</li> </ul>	<ul style="list-style-type: none"> <li>- Operational issues such as clumping, fouling, slagging, and corrosion.</li> <li>- Limited biomass availability.</li> <li>- Inefficiency in biomass harvesting for low-carbon electricity production.</li> </ul>
<b>Gasification</b>	<ul style="list-style-type: none"> <li>- Conversion of challenging biomass (e.g., fibrous bagasse)</li> </ul>	<ul style="list-style-type: none"> <li>- Complex feedstock characterization.</li> <li>- High costs and logistical challenges.</li> </ul>

	<ul style="list-style-type: none"> <li>into usable syngas.</li> <li>- Economically viable compared to naphtha and diesel.</li> <li>- High efficiency with combined cycle technologies.</li> </ul>	<ul style="list-style-type: none"> <li>- Need for catalyst development and gas treatment.</li> </ul>
<b>Pyrolysis</b>	<ul style="list-style-type: none"> <li>- Production of bio-oil, biochar, and biogas from agricultural residues.</li> <li>- Reduction of greenhouse gases.</li> <li>- Sustainable utilization of agricultural waste.</li> </ul>	<ul style="list-style-type: none"> <li>- High equipment costs.</li> <li>- Lack of supportive policies, reducing competitiveness with fossil fuels.</li> </ul>
<b>Fermentation</b>	<ul style="list-style-type: none"> <li>- Efficient production of bioethanol using starch- or sugar-rich biomass (e.g., sugarcane in Brazil).</li> <li>- Flexible methods such as submerged and solid-state fermentation.</li> </ul>	<ul style="list-style-type: none"> <li>- High energy demand for pH control and heating in submerged fermentation.</li> <li>- Challenges in controlling moisture and temperature in solid-state fermentation.</li> </ul>
<b>Anaerobic Digestion</b>	<ul style="list-style-type: none"> <li>- Production of biogas (methane) as a substitute for natural gas.</li> <li>- Utilization of organic waste as a clean energy source.</li> <li>- Co-digestion can improve biogas yield and methane content.</li> </ul>	<ul style="list-style-type: none"> <li>- Reliance on multiple substrates for optimization.</li> <li>- Complex physical and chemical properties of organic waste can hinder methane production efficiency.</li> </ul>

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**Source:** Author (2024).

## 2.6 FUTURE TRENDS AND INNOVATIVE TECHNOLOGIES

Implementing and optimizing anaerobic digestion technologies for various waste streams, such as sewage sludge and sugarcane processing waste, are anticipated trends that will improve energy sustainability (Oliveira *et al.*, 2024). Scariot, Dal-Bó, and Arrieche *et al.* (2024). Propose using different agricultural residues, including coffee husks and SCB, to expand the biomass base and enhance the environmental sustainability of energy production systems. Advanced biomass combustion and processing technologies will be essential to optimize efficiency, reduce costs, and make agricultural waste economically viable (Scariot; Dal-Bó; da Silva Arrieche, 2024).

Anekwe *et al.* (2024) anticipate the development of new catalytic materials with enhanced efficiency and durability, enabling more economical and sustainable processes. Implementing automated and AI-controlled systems to monitor and optimize conversion processes in real time is essential for improving overall efficiency. Supportive government policies, including subsidies and tax incentives, will be crucial for this development (Anekwe *et al.*, 2024). Balakrishnan *et al.* (2024) highlighted the importance of advancements in biogas production technologies, particularly developing high-efficiency anaerobic reactors, to maximize energy production from vinasse. Using vinasse as a biofertilizer and a biogas source aligns with circular economy principles by promoting nutrient recycling and waste reduction. Government incentive policies will be crucial for the growth and consolidation of the biogas and biofertilizer sector derived from vinasse (Balakrishnan, 2024).

SCB cogeneration is a highly efficient and advanced energy solution widely used in several sugarcane-producing nations, including Brazil, Mauritius, and India (Arshad; Ahmed, 2016). This technology has the potential to meet up to a quarter of the current energy needs of the world's leading sugarcane-producing countries (Khoodaruth, 2014). Cogeneration serves as the foundation for maximizing energy utilization from SCB (Mann, 2016b). In this process, SCB is burned to produce steam in high-pressure and high-temperature boilers (Khatri; Pandit, 2022). The steam then drives turbines to generate electricity. Additionally, the waste heat produced during electricity generation is recovered and used to heat industrial processes, thereby increasing the system's overall efficiency (Purohit; Michaelowa, 2007). Improvements to the Rankine cycle, the thermodynamic cycle used in cogeneration systems, have further enhanced the efficiency of converting heat to electricity (Alokika *et al.*, 2021).

Combustion is the most prevalent method for converting fossil fuels and biomass into valuable thermal energy. This process is utilized in various applications, from basic cooking to operating sophisticated ultra-high-pressure boilers for electricity generation (Peres; Rosa; de Morais, 2021). Biogas, a carbon dioxide and methane mixture, can be produced through the anaerobic digestion of SCB and other sugarcane residues (Formann *et al.*, 2020). This biogas can then be used to generate electricity or heat, enhancing and supporting other energy production technologies (Abo *et al.*, 2019). Two-stage, continuous-flow digestion reactors have been developed to improve the efficiency of biomass conversion (Santosh *et al.*, 2017). Additionally, some current technologies can capture the CO<sub>2</sub> released during the combustion

of SCB, thereby reducing greenhouse gas emissions (Milão; Araújo; de Medeiros, 2021). Captured CO<sub>2</sub> can be repurposed to produce chemicals or injected into advanced oil recovery processes, which increases the sustainability of the energy production cycle (Silveira; Costa; Santos, 2023).

Using energy conversion technologies derived from SCB and its byproducts marks a significant advancement toward more economical and sustainable energy production (Ahmad *et al.*, 2021; Reshmy *et al.*, 2021). By integrating various technologies, it is possible to optimize energy output, reduce emissions, and promote a more efficient and sustainable use of available resources (Ajala *et al.*, 2021). This integrated approach improves the economic viability of bioenergy production and plays a crucial role in mitigating climate change and advancing a more sustainable energy future.

## **2.7 ECONOMIC AND COMMERCIAL ASPECTS**

Around 565 million tons of SCB are generated annually worldwide (To; Seebaluck; Leach, 2018). To promote the use of SCB in sustainable energy production, governments have implemented various policies and incentives (Deepchand, 2002; To; Seebaluck; Leach, 2018). These include subsidies and tax credits that reduce the initial costs of installing bagasse-to-energy technologies. Additionally, low-interest financing programs encourage companies to invest in clean energy (To; Seebaluck; Leach, 2018). In countries like Brazil, renewable energy policies and incentive targets have been established to encourage companies to adopt cleaner energy sources, thereby creating a market for energy from SCB (Lima, M A *et al.*, 2020).

The earliest technical-economic studies on SCB processing plants found in the database, primarily focused on biofuel generation, specifically second-generation ethanol. (Cavalett *et al.*, 2012) indicate that autonomous plants (independent and self-sufficient operations) yield a higher return on investment compared to attached plants (integrated with other facilities). Additionally, fixed plants (dedicated to a single process) outperformed flexible ones (capable of handling multiple processes), although flexibility is crucial for productivity in the business sector. (Dias *et al.*, 2010) conducted an economic evaluation of bioethanol generation from SCB in an autonomous distillery, revealing that using waste as fuel in cogeneration systems could significantly increase profit margins.

The excess energy generated during the processes was considered for commercialization (Dias *et al.*, 2010). (Dantas; Legey; Mazzone, 2013) compared the cost and feasibility of different technologies for converting SCB into electrical energy. The main finding was that electricity generation through biomass combustion was the most economically viable technology then. The study also explored other biomass conversion technologies, including gasification (Fawad; Qyyum; Ul Jabbar, 2024), pyrolysis (Toscano Miranda *et al.*, 2021), and anaerobic fermentation (Dari *et al.*, 2024) for energy production from SCB. While these technologies have remained unchanged since then, there are predictions of significant improvements in electricity generation from SCB by 2030. However, other technologies have yet to reach full maturity (Agrawal; Kumar, 2023; Ajala *et al.*, 2021; Kusuma *et al.*, 2023).

Plants and biorefineries that utilize biomass have the potential to integrate energy generation into their operations and sell excess energy to the electricity grid through long-term purchase contracts with distributors, thereby achieving stable revenue streams (Louw *et al.*, 2023; Singh, Jaswinder, 2015). (Rial *et al.*, 2024), highlighted the growing integration of bioenergy into the global energy landscape, primarily driven by supportive government policies that promote sustainable biofuel production and consumption. International cooperation is crucial for sharing knowledge on best practices, cutting-edge technologies, and strategies to minimize environmental impact. Unified policies among nations can further encourage global biofuel trade and establish shared sustainability criteria, helping to standardize and strengthen the global bioenergy sector (Rial *et al.*, 2024).

Considering economic, commercial, and policy aspects, future discussions should focus on integrating international policies to enhance global efforts and improve the efficiency of sustainable energy production from SCB. Analyzing the long-term economic impacts of these policies on local and global economies is crucial. Additionally, exploring the feasibility of emerging technologies and their applications can drive innovation.

## **2.8 ENVIRONMENTAL IMPACTS AND SUSTAINABILITY**

The use of SCB for energy production can significantly reduce greenhouse gas emissions. By repurposing this agricultural residue to generate energy, reliance on fossil fuels is decreased, lowering the release of large amounts of CO<sub>2</sub> (Fioranelli; Bizzo, 2023). Using

biomass, such as SCB, results in net zero emissions because the carbon released during combustion is reabsorbed by plants during their growth, unlike fossil fuels, which add additional CO<sub>2</sub> to the atmosphere (Anukam *et al.*, 2016; Jamora *et al.*, 2019). The sugarcane industry primarily utilizes SCB to generate electricity. This byproduct serves as an energy source in sugar and ethanol plants, helping to reduce greenhouse gas emissions by replacing fossil fuels with renewable biomass (Bizzo *et al.*, 2014). This substitution lowers emissions associated with fossil fuel combustion, which has a significantly higher carbon footprint (Bordonal *et al.*, 2024), while creating new economic opportunities (Chipfupa; Tagwi, 2024).

Brazil, the world's largest producer of sugarcane, accounts for approximately 40% of global production. In the 2022/2023 harvest alone, the country cultivated around 8.3 million hectares of this crop (Bordonal *et al.*, 2024). This impressive production volume solidifies Brazil's position in the global market and presents a significant opportunity for environmental sustainability. Electricity generated from SBC and straw has the potential to substantially mitigate greenhouse gas emissions, with the capacity to reduce emissions from the electricity sector by up to 13%, potentially avoiding up to 72 million tons of CO<sub>2</sub> equivalent per year (Souza; Dias, 2021).

SCB, produced in large quantities by the sugar industry, is often underutilized and represents the most abundant agricultural waste (Candido; Godoy; Gonçalves, 2017). While it is commonly used as a fuel, SCB has the potential to be employed in various other applications, such as feedstock for paper, biofuel for energy, fibre for fabrics, and the production of nanoparticles (Mahmud, Md. Arif; Anannya, 2021; Mohlala *et al.*, 2016). These diverse uses make SCB a valuable material for creating products with higher added value (Iwuozor *et al.*, 2022). Enhancing the management and use of SCB can significantly reduce waste and environmental impacts (Iwuozor *et al.*, 2023). One of the main challenges developing countries face is the accumulation of organic waste (Micheal; Moussa, 2021). Energy production from SCB presents a promising solution to this problem (Sheshdeh *et al.*, 2024). By harnessing this waste for energy, an integrated approach is adopted that promotes sustainability and aligns directly with Sustainable Development Goals (SDGs) 7 and 12 (Molina *et al.*, 2023).

## 2.9 CASE STUDIES AND REAL-WORLD APPLICATIONS

Historically, SCB was viewed as waste that needed to be discarded. However, over time, the perception of SCB shifted as its broader energy potential became evident, transforming it from an undesirable waste into a valuable energy source (Botha; von Blottnitz, 2006). The growing focus on sustainable alternatives has further driven the effort to maximize the energy potential and sustainability of not only SCB but also other parts of the sugarcane plant, such as leaves. Innovative approaches, such as the combination of alkali-acid treatment and microwave technology, have proven effective in removing lignin from SCB, thereby increasing its energy yield (Binod *et al.*, 2012). Other methods, including steam explosion (Pitarelo *et al.*, 2016) and liquid hot water (LHW) treatment, have also been reported to improve the hydrolysis of sugarcane (Nana *et al.*, 2018).

Using plant biomass for energy is cost-effective and technologically feasible, with pellets and briquettes providing a sustainable and economical solution for utilizing this abundant resource (Silva *et al.*, 2021). Selecting the optimal pellet size based on specific application needs optimizes process and maximizes benefits (Erlich *et al.*, 2005). SCB demonstrates remarkable versatility beyond energy production, and its applications extend to feedstock for adsorbents, ion exchange resins, briquettes, ceramics and concrete. These diverse uses highlight the potential of SCB to address global energy challenges and strive for socioeconomic progress and environmental sustainability (Ajala *et al.*, 2021).

In the current energy landscape, ethanol is a major player among bio-based products, leading global production of fossil fuel alternatives. Bioethanol, in particular, powers an industry valued at approximately \$58 billion per year, with global production estimated at 86,000 kton/year. Notably, nearly half of the sugar produced worldwide is allocated to make this vital biofuel (Asgher; Ahmad; Iqbal, 2013).

The presence of lignin and the wide range of chemical constituents across different feedstocks pose significant obstacles to developing a standardized and efficient pretreatment method (Chakraborty *et al.*, 2024). The biochemical conversion process faces several critical challenges. These include enhancing enzyme activity while reducing their cost, achieving efficient co-fermentation of different sugar types, minimizing capital investment, accelerating

reaction times, and developing robust strategies for managing harmful compounds generated during pretreatment (Renu *et al.*, 2014).

## 2.10 CHALLENGES AND FUTURE PERSPECTIVES

Implementing advanced technologies for converting SCB is a vital area of research for those focused on renewable energy and its derivatives. By transforming SCB into biofuels and other high-value products, these technologies significantly improve the energy industry's environmental sustainability and economic viability. The economic feasibility of this technology hinges on various factors, including the costs associated with plant implementation, the efficiency of conversion processes, and the market price of energy (Arcentales-Bastidas; Silva; Ramirez, 2022; Fava; Alves; Romanelli, 2025; Rogala *et al.*, 2023). These factors can render more established alternatives more economically appealing.

The availability of bagasse is influenced by the sugarcane harvest cycle, along with logistical challenges related to transportation and storage, which can affect the consistency of supply (Cangussu; Vieira; Maia, 2023; Yamaji *et al.*, 2024). Furthermore, the lack of targeted public policies, subsidies, and tax incentives to encourage the energy use of agro-industrial residues hampers the competitiveness of this technological pathway in comparison to other energy sources (Granco; Caldas; De Marco, 2019; Lima, Mendelson *et al.*, 2020; Quereshi *et al.*, 2022). Addressing these challenges necessitates a collaborative effort among the public sector, private enterprises, and research institutions, all aimed at developing integrated and sustainable solutions (Ba; Nair; Kedia, 2024; Bai *et al.*, 2024). Incorporating these factors is crucial for a thorough analysis of the sustainability and viability of the proposed technology. Table 2.4 delineates various challenges faced across multiple sectors—including economic, logistical, regulatory, and competitive domains—concerning the expansion of sugarcane bagasse utilization for energy production.

**Table 2.4** Assessment of economic challenges, logistics and supply, policy and regulation, market and competitiveness.

Category	Barrier	Implications	Possible Solutions
Economic	High implementation and operational costs	Difficulty competing with more established energy sources	Tax incentives, green financing, technological optimization
Logistics and Supply	Seasonal availability and challenges in transport/storage of bagasse	Irregular supply and increased operational costs	Integrated planning with sugar mills, improved storage technologies, logistic efficiency
Policy and Regulation	Lack of specific public policies and incentives	Low attractiveness to investors and limited integration into national energy strategies	Development of regulatory frameworks, inclusion in bioenergy support programs
Market and Competitiveness	Energy cost may be uncompetitive compared to conventional sources	Reduced economic viability of projects	Creation of regulated energy markets, internalization of environmental externalities

**Source:** Author (2024).

Future research should prioritize integrating biogas production technologies with other renewable energy sources, as emphasized by (Balakrishnan *et al.*, 2024). In addition, using vinasse as both a biofertilizer and a source of biogas is a promising approach to promoting the circular economy. These strategies diversify the portfolio of high-value products and improve the sustainability of industrial processes (Balakrishnan *et al.*, 2024).

According to Brown *et al.* (2024), the variability in SCB composition complicates the standardization of conversion processes. A significant challenge is the need for specific pretreatments to remove lignin and hemicellulose without degrading the sugars, which are essential for subsequent biofuel production. Furthermore, scaling these processes to industrial levels presents additional difficulties, such as handling solids and feeding biomass into high-pressure reactors (Brown *et al.*, 2024).

The economic viability of hydrogen production from biomass depends on several factors, including the availability of low-cost biomass and government incentives. According to Vinu *et al.* (2024), carbon materials derived from SCB exhibit low electrical conductivity, which limits their electrocatalytic performance. The low cycle stability of metal hydroxide-based materials negatively impacts their potential for large-scale application in supercapacitors. Additionally, the low capacitance of metal silicate electrodes further hinders their ability to achieve superior performance (Vinu *et al.*, 2024).

Gautam *et al.* (2024) highlighted the significant technical challenges in efficiently converting SCB into hydrogen using microbial electrolysis cells (MECs). One major issue is the internal resistance of single-chamber systems, which results in a gas mixture that requires advanced technologies for effective hydrogen separation and purification. Additionally, the lack of efficient catalysts and the reliance on costly electrode materials, such as precious metals, can significantly increase the operational costs of MECs, making the technology economically challenging for large-scale implementation (Gautam *et al.*, 2024).

(Machado; Abreu, 2024) highlighted that optimizing first- and second-generation ethanol supply chains is a complex task that requires a careful balance between reducing costs and meeting resource demands. Additionally, integrating Nexus principles—focused on the interconnectedness of water, energy, and food systems—into biofuel supply chain management is particularly challenging due to the need for resource efficiency and sustainable production (Machado; Abreu, 2024).

## **2.11 RESEARCH AND DEVELOPMENT**

Yadav, Pratiksha *et al.*, 2024 conducted detailed investigations into key pretreatment methods to reduce lignin content and increase the accessibility of cellulose and hemicellulose for enzymatic hydrolysis. Combined approaches, such as ultrasound-assisted ionic solvents, have shown promising results in enhancing these processes. Developing integrated biorefinery processes that efficiently convert SCB into various high-value products, including bioethanol, biobutanol, and enzymes, is crucial (Yadav, Pratiksha *et al.*, 2024).

Ali *et al.* (2024) emphasizes the urgent need to develop genetically modified microorganisms or microbial consortia capable of withstanding adverse process conditions and improving ethanol production efficiency. The authors also stress the importance of developing methods to reduce byproduct inhibition and increase cellulose accessibility to enzymes, which are critical research areas. Additionally, ongoing efforts to optimize operational parameters, such as temperature and pH, during the production process are essential for maximizing efficiency and productivity (Ali *et al.*, 2024).

Zolfagharpour *et al.* (2024) highlighted the importance of investigating various types of metal catalysts and their combinations to enhance the production and quality of bio-oil. Key parameters such as pyrolysis temperature, residence time, and the catalyst-to-biomass ratio require continuous optimization. Further research is essential to identify the optimal conditions that maximize bio-oil yield while minimizing the formation of undesirable byproducts. Additionally, employing advanced techniques such as gas chromatography and mass spectrometry to characterize the chemical composition of bio-oil is crucial for gaining a deeper understanding of reaction mechanisms and improving the production process (Zolfagharpour; Sharafati; Hosseinzadeh, 2024).

## 2.12 CONCLUSION

A thorough review of research and technological advancements has revealed substantial growth in the importance and feasibility of utilizing SCB for energy generation. This growth is primarily driven by the global pursuit of sustainable energy and the shift towards a low-carbon economy. Key findings indicate a significant increase in the use of terms such as “sugarcane bagasse” and “energy production” since 2016, reflecting the rising research interest in these areas. This surge can be attributed to advances in conversion technologies, methods for utilizing agricultural waste, and heightened awareness of the importance of energy sustainability and reducing carbon emissions. The temporal analysis of search terms shows an evolution from general themes to more specific and technically complex topics, highlighting the maturation and increasing complexity of the field. Additionally, our analysis reveals a growing trend toward specialized and diversified research, particularly focusing on improving process efficiency and developing high-value products such as biofuels and biofertilizers. The bibliometric analysis demonstrates not only an expansion in the number of publications but also the sophistication of approaches and the integration of new technologies, indicating a field in constant evolution and adaptation to current technological and environmental demands. However, despite significant progress, substantial challenges remain.

These include the need for improvements in the efficiency of conversion processes and the economic viability of large-scale operations. Furthermore, regulatory issues and the necessity for public incentive policies are crucial for the widespread implementation of these technologies. A continued focus on research and development to enhance technologies for SCB conversion is recommended, along with the promotion of collaborations between academia, industry, and government to overcome existing obstacles. Incentive and financing policies for sustainable energy projects are essential to accelerate the adoption of these technologies. In conclusion, energy production from SCB emerges as a crucial area for the development of clean and sustainable energy technologies. The continuous growth and evolution of research in this domain underscore its relevance and potential to contribute significantly to the global energy transition. However, addressing the existing challenges with a coordinated and strategic approach is imperative to maximize the benefits of this promising sustainable energy source.

### ***3 SOLAR-POWERED HYDROGEN PRODUCTION FROM SODIUM BOROHYDRIDE HYDROLYSIS USING SUGARCANE BAGASSE (SACCHARUM OFFICINARUM) AS A GREEN CATALYST***

#### **Abstract**

The quest to replace fossil fuels with clean energy has been driven by research into more sustainable and metal-free hydrogen production. A hydrogen generation system based on sodium borohydride hydrolysis ( $\text{NaBH}_4$ ) was developed using catalysts derived from residual biomass, specifically sugarcane bagasse. The successful synthesis of Modified Sugarcane Bagasse (MOB) catalysts with high stability and catalytic activity with phosphoric acid  $\text{H}_3\text{PO}_4$  were confirmed through characterization techniques, including FTIR, XRD, XRF, TGA, SEM and GC, which verified positive structural modifications. The Taguchi method was applied to optimize the process and reduce the number of experiments. The factors that statistically contributed the most within the model's confidence range were time (35 minutes), Temperature ( $60\text{ }^\circ\text{C}$ ),  $\text{NaBH}_4$  (750 mg), and MOB (300 mg), with  $\text{NaBH}_4$  standing out at 58.87% and the MOB catalyst contributing 27.03% to hydrogen production. Additionally, the integration with a photovoltaic solar energy network demonstrated energy viability, with a total consumption of 136 kWh to produce 1000 mL of  $\text{H}_2$ . The results show that MOB exhibits superior catalytic performance for  $\text{NaBH}_4$  hydrolysis, with excellent economic feasibility. This catalyst is highly promising for practical applications in  $\text{NaBH}_4$  hydrolysis.

**Keywords:** Sugarcane, Green Catalyst, Hydrogen Production, Hydrolysis,  $\text{NaBH}_4$ .

### 3.1 INTRODUCTION

The need to replace fossil fuels and the negative environmental impacts have driven the search for more sustainable and ecologically friendly energy alternatives (Yadav, Mahendra; Xu, 2012; Zhao *et al.*, 2015; Zhong *et al.*, 2017). To mitigate the harmful effects of gases such as carbon monoxide (CO), carbon dioxide (CO<sub>2</sub>) and the incomplete combustion of hydrocarbons, it is essential to turn to clean energy sources such as solar (Magson *et al.*, 2024), wind (Assareh *et al.*, 2024), biomass (Catumba *et al.*, 2023; Dari *et al.*, 2024) or energy carriers, especially hydrogen (Sahiner; Seven, 2014; Zavala *et al.*, 2025). H<sub>2</sub> gas has emerged as a clean and promising source with the potential to replace fossil fuels (Osman *et al.*, 2024; Yurderi *et al.*, 2015). It also has a high energy density, is non-toxic and its combustion by-product is water (Ashik; Wan Daud; Abbas, 2015; Sarmah *et al.*, 2023).

Among the methods investigated for its production are water electrolysis, methanolysis and hydrolysis of metal hydrides such as LiBH<sub>4</sub> and NaBH<sub>4</sub> (Abbas; Caglar; Kivrak, 2024; Chang; Rajuli, 2024; Shiva Kumar; Lim, 2022). Among these approaches, the hydrolysis of NaBH<sub>4</sub> stands out due to its high hydrogen storage capacity (10.8% by weight), non-toxicity, non-flammability and high purity (Can *et al.*, 2020; Christian; Aguey-Zinsou, 2012; Ekinici *et al.*, 2024; Santra *et al.*, 2019). In addition, its by-product, sodium metaborate (NaBO<sub>2</sub>) is recyclable, soluble in water and environmentally friendly, reinforcing green chemistry (Altaf *et al.*, 2023; Shi *et al.*, 2023). The reaction for this chemical process is shown in Equation (1):



However, hydrolysis of NaBH<sub>4</sub> requires green, efficient and sustainable catalysts to ensure efficient hydrogen production (Gamba *et al.*, 2024; Kadrekar; Patel; Arya, 2020). Fangaj *et al.* (2020) synthesized a green catalyst from apricot kernel shells for the hydrolysis of NaBH<sub>4</sub> (Fangaj; Ceyhan, 2020). The material was treated with different acids and bases, in which 15% phosphoric acid (H<sub>3</sub>PO<sub>4</sub>) stood out for its efficiency, with an activation energy of (30.23 kJ mol<sup>-1</sup>) (Fangaj; Ceyhan, 2020). Bekirogulari (2020) investigated the use of defatted coffee powder impregnated with zinc (Zn) as a reusable catalyst in the alcoholysis reaction of NaBH<sub>4</sub> (Bekirogullari, 2020). The results showed that this material presented ideal conditions, with an impregnation temperature of 75 °C and heat treatment at 1000 °C, demonstrating its superiority

over conventional catalysts (Bekirogullari *et al.*, 2020). In the study carried out by Kaya *et al.* (2020), defatted coffee powder was also used, but in the methanolysis of sodium borohydride ( $\text{NaBH}_4$ ), with acetic acid ( $\text{CH}_3\text{COOH}$ ) as the differential (Kaya *et al.*, 2020). The results obtained indicated a hydrogen production rate of  $3171.4 \text{ mL min}^{-1} \text{ g}^{-1} \text{ cat}$  and an (En) of  $25.23 \text{ kJ mol}^{-1}$ . Zhang *et al.* (2022) improved the dehydrogenation of  $\text{NaBH}_4$  using CoB-anchored graphene nanosheets as a support. The results show a high  $\text{H}_2$  production rate of  $7937 \text{ mL H}_2 \text{ min}^{-1} \text{ g}^{-1}$  and a recyclability of 58% after 10 cycles (Haoran *et al.*, 2023). Other catalysts produced with oxalic acid or doped with rice husk stand out for their high catalytic activity and  $\text{H}_2$  generation rate (Ababaii; Gilani; Pasikhani, 2024; Dudu; Şeker, 2023). These studies motivate the reuse of biomass in green catalysts. However, there is a gap in the literature on the use of local biomass with experimental designs and measurement of solar energy consumption in production, which this study seeks to fill.

Sugarcane (*Saccharum officinarum*) is a tropical plant of the genus *Saccharum*. Cultivated predominantly in tropical and subtropical regions, sugarcane accounts for about 80% of global sugar production, playing a vital role in the sugar industry (Kouzi; Kontro, 2024; Nogueira *et al.*, 2023). Brazil is one of the largest producers of sugarcane, with a harvest of 642.7 million tons in 2019-2020 (Taweengern *et al.*, 2025). However, disposal of sugarcane bagasse in sugar factories represents a challenge, with approximately 0.3 tons of waste being generated (Meghana; Shastri, 2020).

In this context, the innovation of this study lies in the reutilization of sugarcane bagasse as a support material, employing 15% phosphoric acid ( $\text{H}_3\text{PO}_4$ ) as a catalytic species for hydrogen production. The Taguchi method is applied to optimize and reduce the number of reactions. In addition to evaluating catalytic efficiency, the study also examines the elemental composition and morphology using advanced techniques such as XRD, SEM, FT-IR, TGA, XRF, and GC. Furthermore, integrating this approach with clean energy generation systems, such as photovoltaic power plants, enhances the sustainability of the entire production chain by supporting both catalyst production and hydrogen generation. This strategy contributes to Sustainable Development Goal (SDG 7) - Affordable and Clean Energy, by promoting a more sustainable energy matrix, and (SDG 12) - Responsible Consumption and Production, by encouraging waste valorization and the efficient use of natural resources.

## 3.2 METHODOLOGY

### 3.2.1 Materials

Sugarcane bagasse (SBB) was collected in the Baturité Massif region of Ceará, Brazil, from establishments that sell sugarcane juice and from sugar mills. Acid treatment was performed with phosphoric acid ( $\text{H}_3\text{PO}_4$ , 85%; CAS: 7664-38-2, Sigma-Aldrich). For alkaline treatment, hydrogen peroxide ( $\text{H}_2\text{O}_2$ , 35%; CAS: 7722-84-1, Sigma-Aldrich) was used. Sodium hydroxide (NaOH, 98%; CAS: 1310-73-2) and sodium borohydride ( $\text{NaBH}_4$ , 98%; CAS: 16940-66-2) were purchased from Sigma-Aldrich (St. Louis, MO, USA). Ultrapure water (Type I) was obtained using the Solab 71/10 purification system.

### 3.2.2 Methods

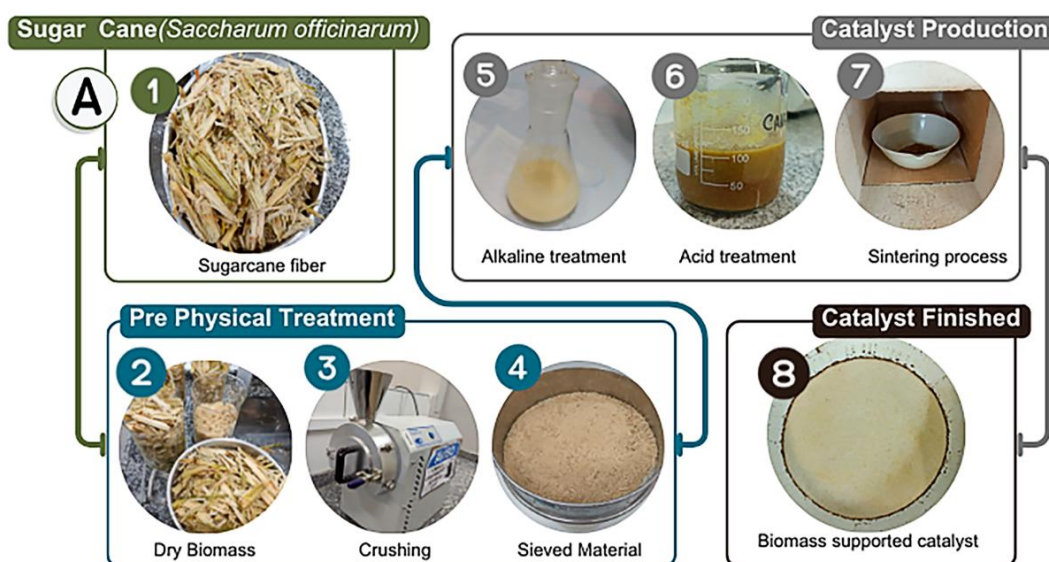
The sugar cane bagasse was subjected to three consecutive washes with ultrapure water over three days and then dried in an oven at 90 °C for 24 hours. The material was then ground in a Willey-type knife mill (SL-31), and the ground powder was sieved through a sieve (28 mesh) to achieve a particle size of 0.60 mm. This procedure was carried out according to the methodology adapted from Rocha et al. (2011) (Rocha *et al.*, 2011).

After the previous treatment, the ground sugarcane bagasse was subjected to alkaline treatment with  $\text{H}_2\text{O}_2$  (4.3% v/v) adjusted to pH 11.5 using 6 mol/L NaOH in order to solubilize the lignin present in the biomass, according to the methodology described by Correa et al. (2013) and De Sousa et al (2016), with some changes (Correia *et al.*, 2013; De Souza, Ticiane C. *et al.*, 2016). This step was carried out to increase the porosity of the support, facilitating the accessibility of phosphoric acid ( $\text{H}_3\text{PO}_4$ ) to the material (Correia *et al.*, 2013). Initially, 10g of sugarcane bagasse was weighed and added to a 125 mL Erlenmeyer flask, with 100 mL of  $\text{H}_2\text{O}_2$ , subjected to an orbital shaker (NT715 - Incubator Shaker, Brazil), operating at a speed of 200 rpm, temperature of 35 °C and time of 6 hours. After this time, the solid material was recovered by vacuum filtration, washed three times with ultrapure water, dried at 60 °C for 24 hours and stored in a desiccator (Santos Lima et al., 2012).

To preparation the most efficient final catalyst (MOB), the material was subjected to treatment with impregnated acid. Following protocols adapted from the literature (Fangaj; Ceyhan, 2020; Kaya, 2020; Saka; Kaya; Bekiroğullari, 2020a). 5 grams of sugarcane bagasse previously treated with an alkaline solution were mixed with 50 mL of 15%  $\text{H}_3\text{PO}_4$  in a 125 mL Erlenmeyer flask. The mixture was stirred in an orbital shaker at 200 rpm and 35 °C for 10

minutes (Fangaj; Ceyhan, 2020). Then, the solution was left to rest for 24 hours to allow complete acid impregnation. After this period, the solid fraction was recovered by vacuum filtration and washed three times with deionized water to remove any acid residue in the sample. Following this step, the material was dried in an oven at 60 °C for 24 hours and transferred to a ceramic container for the sintering stage. This process was carried out in a muffle furnace (EDG F-3000) at 200 °C for 45 minutes (Kaya, 2020). After sintering, the catalyst was washed three times with distilled water and dried in an oven at 60 °C for 6 hours. Finally, the catalyst was stored in a container, ensuring its preservation for later use. Figure 3.1 illustrates the flowchart of the catalyst preparation steps (MOB).

**Figure 3.1** Flowchart of the catalyst production process from sugarcane fiber.



**Source:** Author (2024).

### 3.2.3 Statistical analysis

An experimental design based on the “Taguchi” method was employed, using an L16 ( $4^5$ ) orthogonal array consisting of four levels and five factors. In this method, “L” represents the Latin square, and “16” refers to the number of trials conducted (Simão Neto *et al.*, 2024). Taguchi is a Design of Experiments (DoE) technique aimed at optimizing experimental planning to identify the best optimal parameters (Wei-Hsin *et al.*, 2024; Okolie *et al.*, 2021). Additionally, the Taguchi method allows for the simultaneous consideration of factors affecting system performance (Hamzaçebi, 2020). The design distributed five factors across

four levels to optimize the hydrogen-generated volume. The levels of each factor and the experimental design L16 were randomly distributed by the Taguchi method, indicating that the model assumptions are valid, Table 3.1 and Table 3.2 illustrate the values for each factor.

**Table 3.1** Definition of experimental procedure levels and independent parameter ranges, incorporating the Taguchi method for optimization.

Level	t (min)	NaBH <sub>4</sub> (mg)	MOB (mg)	T (°C)	Stirring rate (RPM)
Level 1 (L1)	15	100.0	50.0	30	120
Level 2 (L2)	25	250.0	100.0	40	130
Level 3 (L3)	35	500.0	150.0	50	140
Level 4 (L4)	45	750.0	300.0	60	150

**Source:** Author (2024).

**Table 3.2** Orthogonal L16 Matrix for Hydrogen Generation Using the Taguchi Method.

Exp	t (min)	MOB (mg)	NaBH <sub>4</sub> (mg)	T (°C)	Stirring rate (RPM)
1	15	150.0	100.0	30	120
2	15	100.0	250.0	40	130
3	15	150.0	500.0	50	140
4	15	300.0	750.0	60	150
5	25	100.0	100.0	50	150
6	25	50.0	250.0	60	140
7	25	300.0	500.0	30	130
8	25	150.0	750.0	40	120
9	35	150.0	100.0	60	130
10	35	300.0	250.0	50	120
11	35	50.0	500.0	40	150

12	35	100.0	750.0	30	140
13	45	300.0	100.0	40	140
14	45	150.0	250.0	30	150
15	45	100.0	500.0	60	120
16	45	50.0	750.0	50	130

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**Source:** Author (2024).

### 3.2.4 Material characterization

#### 3.2.4.1 Scanning Electron Microscopy

Scanning Electron Microscopy (SEM) was conducted using the QUANTA 450 FEG microscope. The samples were prepared with carbon tape fixation and silver coating, performed using the Quorum QT150ES equipment. During the analysis, an electron beam with a voltage of 20 kV was used, allowing for detailed images of the sample surface to be obtained (de Menezes *et al.*, 2024).

#### 3.2.4.2 X-ray fluorescence

X-ray Fluorescence (XRF) analysis was performed using the SHIMADZU EDX-7000 equipment, operating in an air atmosphere and with a 10 mm collimator. The samples were analyzed under two voltage and current settings: 50 kV and 88  $\mu$ A for the energy range of 0 to 40 keV and 15 kV and 381  $\mu$ A for the 0 to 20 keV range. Each configuration had an acquisition time of 30 seconds, allowing for qualitative and quantitative identification of the elements present in the sample, including Cu, K, Si, S, Rh, Ca, Mn, Fe, Tb, Zn, Eu, Ar, among others (Melo *et al.*, 2024a).

#### 3.2.4.3 Fourier Transform Infrared Spectroscopy

Fourier Transform Infrared (FTIR) spectroscopy was used to investigate the activation of the support. The analyses were conducted using a Perkin Elmer FT-IR/NIR FRONTIER spectrophotometer equipped with an attenuated total reflectance (ATR) accessory with a zinc selenide (ZnSe) crystal. The measurements were performed with a resolution of 4  $\text{cm}^{-1}$ , averaging 32 scans and covering the spectral range from 4000 to 500  $\text{cm}^{-1}$  (Priya; Kennedy; Anand, 2024).

#### 3.2.4.4 X-ray diffraction

X-ray diffraction (XRD) analysis was performed on a X'Pert MPD powder X-ray diffractometer (Panalytical), using CuK $\alpha$  radiation ( $\lambda = 1.54059 \text{ \AA}$ ). The experimental conditions included operation at 40 kV and 30 mA, with scanning in the  $2\theta$  range of  $20^\circ$  to  $100^\circ$ , using the Brentano-Bragg geometry in continuous mode, a scan speed of  $0.5^\circ/\text{min}$ , and a step size of  $0.02^\circ$  ( $2\theta$ ). The diffraction patterns were analyzed and interpreted through Rietveld structural refinement using Maud $^\circledR$  software. The crystallite size of the samples was calculated based on the Scherrer equation.

#### *3.2.4.5 Thermogravimetric Analysis (TGA)*

The thermal analysis of both pure and treated biomass was evaluated through thermogravimetric analysis (TGA). The tests were conducted using Mettler Toledo TGA/SDTA851e analyzer, with samples weighing approximately 10 mg. The process was carried out under a continuous nitrogen ( $\text{N}_2$ ) flow of 50 mL/min, with controlled heating at a constant rate of  $10^\circ\text{C}/\text{min}$ , covering a temperature range from  $30^\circ\text{C}$  to  $800^\circ\text{C}$  (Li, Jinjian *et al.*, 2025; Melo *et al.*, 2024b).

#### *3.2.5 Characterization of Generated Gas*

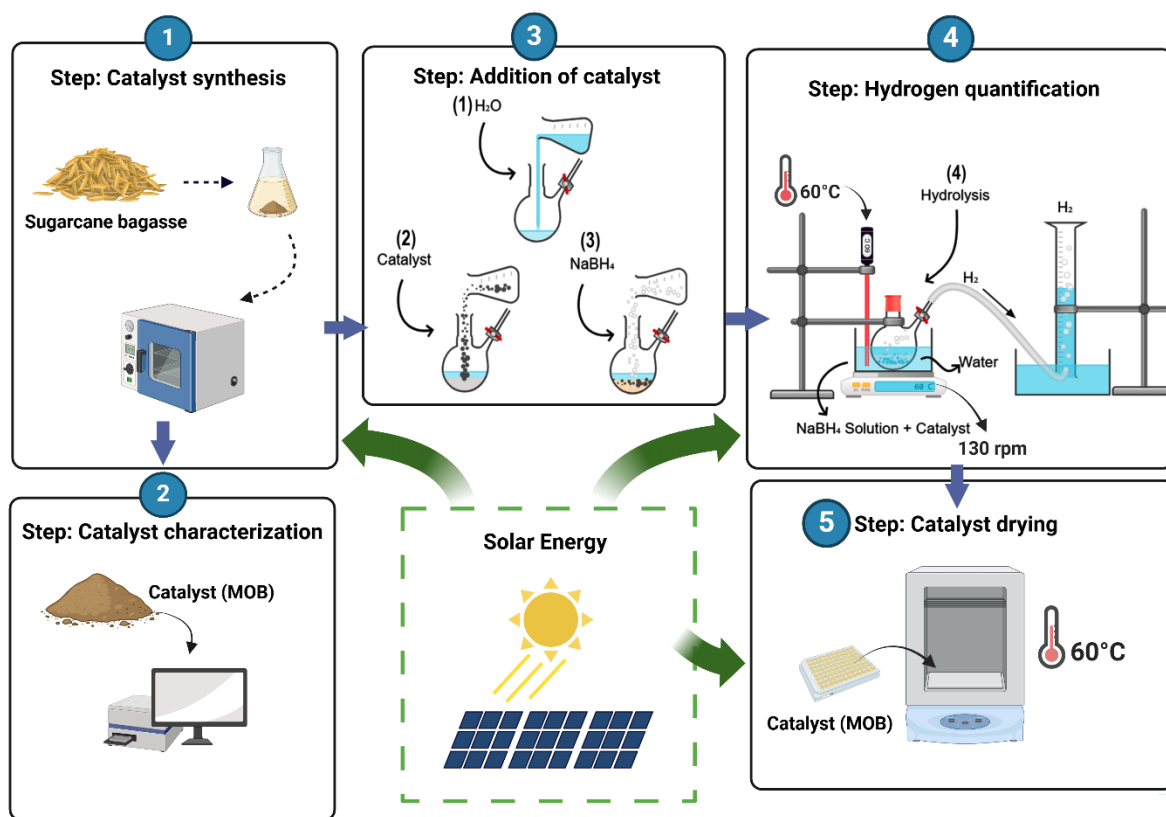
The gas sample was collected in a 5L Tedlar Gas Sampling Bag and then characterized using an Agilent 990 Micro Gas Chromatography (MGC) equipped with a high-precision Thermal Conductivity Detector (TCD). The system has two independent column channels optimized for different separations: a 20-meter Molesieve column and a 10-meter CP-PoraPLOT U column. The process was performed using Argon as the carrier gas.

#### *3.2.6 Experimental setup (water displacement)*

The quantification of hydrogen generated during hydrolysis was performed using the water volume displacement method (Srivastava *et al.*, 2024). The general scheme of the work, (Figure 3.2), presents the steps followed in the study. In the first step, the catalyst was synthesized using methodologies previously described in the previous sections. In addition, the equipment used in the process operated with solar energy, making the system more sustainable. In the second step, the catalyst was characterized using advanced techniques to confirm the treatment performed with phosphoric acid ( $\text{H}_3\text{PO}_4$ ). The third step consisted of adding the catalyst to the reaction system. Initially, 10 mL of deionized water was added to the 250 mL volumetric flask (1), followed by the introduction of the catalyst (MOB) into the flask (2). Then,

the mixture was completed with the addition of NaBH<sub>4</sub> (3), then submerged in a heated water bath on a magnetic stirrer (K40-1820H, 220V, KASVI). In the fourth stage, complete hydrolysis of NaBH<sub>4</sub> (4) occurred, in which the volume of gas generated was quantified, the H<sub>2</sub> gas flows to the graduated column, displacing the water and allowing measurement. This reaction followed the conditions optimized by the Taguchi method, considering factors such as temperature, NaBH<sub>4</sub> concentration, catalyst mass, reaction time and revolutions per minute. After H<sub>2</sub> production, the catalyst was washed with deionized water to remove the NaBO<sub>2</sub> formed in the reaction, then dried in an oven at 60°C and reused in future cycles.

**Figure 3.2** Scheme of volumetric measurement of gas by water displacement in a reaction system.



**Source:** Author (2024).

### 3.2.7 Activation Energy

The performance of the catalytic system in the hydrolysis of sodium borohydride (NaBH<sub>4</sub>) was investigated by measuring the reaction rate at different temperatures and constructing the corresponding Arrhenius plot. The hydrogen evolution rate was measured

between 30 and 60 °C with a heating ramp of 10 °C.min<sup>-1</sup>. The apparent activation energy (E<sub>a</sub>) was calculated using the Arrhenius equation (Eq. 3.2), based on the kinetic curves of hydrogen generation at various reaction temperatures (30, 40, 50 and 60 °C)

$$\ln k = \ln A - \frac{E_a}{RT} \quad (3.2)$$

In the Arrhenius equation, (A) is the pre-exponential factor, (T) is the hydrolytic temperature in Kelvin, and (R) is the universal gas constant (8.314 J mol<sup>-1</sup> K<sup>-1</sup>) (Xu, Fengyan *et al.*, 2024). A lower E<sub>a</sub> value indicates that the reaction occurs more efficiently, requiring less energy. In the context of a biomass-supported catalyst, a reduced E<sub>a</sub> value suggests the catalyst is more active and effective in promoting the reaction, which is desirable for optimizing catalytic process performance.

### 3.2.8 Hydrogen Generation Rate (HGR)

The evaluation of the hydrogen generation rate during the catalytic hydrolysis of NaBH<sub>4</sub> was based on measuring the volume of hydrogen produced over time using the formula presented in Equation (3.3). In this equation, the volume of hydrogen generated, represented by the variable V (in millilitres), is divided by the product of the total reaction time (T, in minutes) and the mass of the catalyst used (M, in grams). Thus, the hydrogen generation rate (HGR) is expressed in units of millilitres per minute per gram (mLH<sub>2</sub>.min<sup>-1</sup>.g<sup>-1</sup>) (Fengyan *et al.*, 2024).

$$HGR = \frac{V}{T \times M} \quad (3.3)$$

### 3.2.9 Photovoltaic Solar Energy Consumption to Power the System

The experiments in this study were conducted at the Biomass Laboratory of the Institute of Engineering and Sustainable Development, powered by a photovoltaic mini generation plant located on the Aurora Campus of UNILAB in Redenção, Ceará, Brazil (Latitude: 4.217876° S, Longitude: 38.713731° W). The plant (Figure 3.3) has been operational since December 2018 and comprises 762 Canadian Solar 60 CS6U-330P photovoltaic modules and 6 ABB PRO 33.0-TL-OUTD-SX-400 inverters, with a total installed capacity ( $P_{pv}$ ) of 251.4 kWp. To estimate

the plant's average monthly generation ( $MG_{pv}$ ) e used Eq. (3.4), which considers panel power, the average daily horizontal solar irradiation ( $Ir_{sun}$ ) as provided by the Brazilian Solar Energy Atlas (CRESESB, 2017) (PEREIRA *et al.*, 2017), system efficiency ( $\eta$ ), approximately 80%, and the number of days in a month. The EMBRASUL RE8000 electrical energy analyzer was used to measure the energy consumed, in kWh, by the equipment used in the synthesis and catalytic treatment, as well as the H<sub>2</sub> generation process.

**Figure 3.3** Rooftop solar plant at the Auroras Campus (UNILAB).



**Source:** Author (2024).

$$MG_{pv} = P_{pv} \times Ir_{sun} \times \eta \times 30 \quad (3.4)$$

### 3.3 RESULTS AND DISCUSSIONS

#### 3.3.1 Process optimization using the Taguchi method (L16)

Table 3.3 expands on the results of the Taguchi design for three samples in aqueous solution: the reference value (Control), corresponding to the reaction of pure NaBH<sub>4</sub> without a catalyst; pure sugarcane bagasse (BAG) with NaBH<sub>4</sub>; and the modified sugarcane bagasse

catalyst (MOB) with NaBH<sub>4</sub>. In these reactions, the volume of hydrogen produced was measured. The amount of sodium borohydride used was observed to be the most influential parameter in hydrogen generation, especially in the MOB sample, which positively impacted the experiment. This is due to sodium borohydride's high efficiency in releasing large volumes of H<sub>2</sub> when decomposed in water in the presence of a catalyst (Akkaş; Gürü, 2021; Bu *et al.*, 2023).

Experiments 4, 7, and 8 produced significant hydrogen volumes, using 750, 500, and 750 mg of NaBH<sub>4</sub>, respectively, generating higher volumes than the other 16 experiments. The Control and BAG samples showed lower volumes due to the lack of a catalyst. Experiment 7, with 300 mg of catalyst and 500 mg of NaBH<sub>4</sub>, produced 616.6 mL of H<sub>2</sub> in 25 minutes at 30 °C. Experiments 4, 7, and 8 produced significant hydrogen volumes, using 750, 500, and 750 mg of NaBH<sub>4</sub>, respectively, generating higher volumes than the other 16 experiments.

The Control and BAG samples showed lower volumes due to the lack of a catalyst. The best catalytic performance was observed in Experiment 7, where 300 mg of catalyst was used in a reaction with 500 mg of NaBH<sub>4</sub> in an aqueous medium, releasing 616.6 mL of H<sub>2</sub> in 25 minutes at 30 °C. It is worth noting that Experiments 4 and 8 also produced considerable hydrogen volumes (403.6 and 318.6 mL, respectively) (Table 3.3), both conducted at 60 °C. Finally, it is essential to emphasize that obtaining a satisfactory hydrogen volume requires a significant amount of NaBH<sub>4</sub> and an adequate catalyst to ensure maximum reaction yield, as shown in Table 3.3.

**Table 3.3** Experimental results and the signal-to-noise (S/N) ratio obtained using the Taguchi L16 design included the reference value, modified bagasse-based catalyst (MOB), and raw bagasse (BAG). Each experimental level was conducted in triplicate to ensure errors remained within standard limits, thus ensuring the accuracy and reliability of the results.

Exp	Control (mL)	MOB (mL)	S/R (MOB)	BAG (mL)	S/R (BAG)
1	6.0	34.3 ± 0.6	30.7	14.0	22.9
2	30.0	50.6 ± 0.4	34.0	20.0	26.0

3	125.0	105.1 ± 1.0	40.4	65.0	36.2
4	345.0	403.6 ± 0.6	52.1	235.0	47.4
5	59.0	54.6 ± 1.1	34.7	11.0	20.8
6	230.0	140.0 ± 0.5	42.9	20.0	26.0
7	13.0	616.6 ± 0.6	55.8	198.0	45.9
8	60.0	318.6 ± 1.5	50.0	220.0	46.8
9	155.0	64.0 ± 2.6	36.1	5.0	13.9
10	160.0	240.3 ± 0.6	47.6	40.0	32.0
11	170.0	213.8 ± 0.8	46.6	20.0	26.0
12	63.0	196.4 ± 0.9	45.8	127.0	42.0
13	51.0	110.0 ± 1.0	40.8	9.0	19.0
14	50.0	150.5 ± 0.5	43.5	59.0	35.4
15	575.0	165.1 ± 0.8	44.3	5.0	13.9
16	200.0	245.5 ± 0.5	47.8	5.0	13.9

Exp: Number of experiments, MOB: Modified Sugarcane Bagasse, BAG: Raw Sugarcane Bagasse, S/N: Signal-to-Noise.

**Source:** Author (2024).

### 3.3.2 Statistical analysis of data

The Taguchi method uses the Signal-to-Noise (S/N) ratio to evaluate each experimental parameter (Cavalcante *et al.*, 2024; Hisam *et al.*, 2024; Xydas *et al.*, 2005). Three S/N criteria are provided: (1) nominal is best, (2) more minor is better, and (3) more prominent is better (Hikmat; Rostam; Ahmed, 2021; Pundir; Chary; Dastidar, 2018). This study used the criterion (larger is better) to maximize the relationships based on the volume of hydrogen produced. Therefore, the larger is better ratio was evaluated according to the mathematical equation below (3.5):

$$S/N = -10 \log \left( \frac{1}{n} \sum_{i=1}^n \frac{1}{y_i^2} \right) \quad (3.5)$$

Where  $n$  is the number of repetitions of the experiment, and  $y_i$  represents the response of the volume of hydrogen generated in the experiment. The S/N ratio was used to predict the optimal conditions for obtaining the maximum volume of H<sub>2</sub>, as estimated by equation (3.6).

$$\overline{S/N} = \frac{\bar{s}}{N} + \sum_{i=1}^n \left( \frac{s}{N_i} - \frac{\bar{s}}{N} \right) \quad (3.6)$$

$N_j$  is the S/N ratio at the optimum point for each factor,  $S'/N$  is the average S/N ratio, and  $n$  is the number of significant factors in the experiment. Table 3.4 and Figure 3.4 show the average S/N values and delta values, which rank the importance of each parameter. The mass of NaBH<sub>4</sub> and the mass of the modified catalyst (MOB) were the most significant factors, with delta values of 13.36 and 9.33, respectively. These factors are crucial for maximizing H<sub>2</sub> production and are key to the experiment's success.

**Table 3.4** (ANOVA) Ranking of the S/N parameters that influence the hydrogen generation rate using the MOB catalyst as the catalyst.

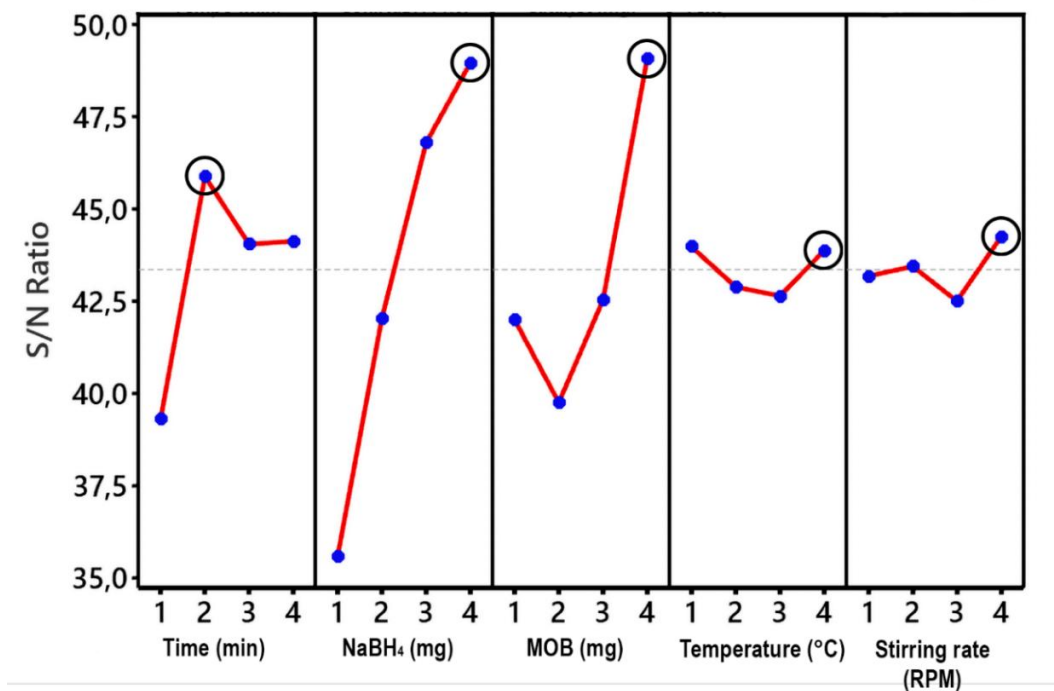
Factor levels	t	NaBH <sub>4</sub>	MOB	T	Stirring rate
1	39.3	35.6	42.0	43.9	43.1
2	45.8	42.0	39.7	42.8	43.4
3	44.0	46.8	42.5	42.6	42.5
4	44.1	48.9	49.0	43.8	44.2
<b>Delta</b>	6.5	13.3	9.3	1.3	1.7
<b>Rank</b>	<b>3</b>	<b>1</b>	<b>2</b>	<b>5</b>	<b>4</b>

**Source:** Author (2024).

Figure 3.4 shows the effect plot of parameters influencing H<sub>2</sub> generation, created with ©Minitab software (version 19). It highlights the best experimental values for factors like time, NaBH<sub>4</sub> mass, MOB, temperature, and rotation rate. The horizontal line indicates no impact, while an inclined line shows the influence of varying factor levels (Karaoglu; Yolcular, 2022b). Comparing the slopes, the increasing line has a greater impact on H<sub>2</sub> generation, while the decreasing line has a lesser effect (Karaoglu; Yolcular, 2022a). Figure 3.4 shows that the amount of sodium borohydride (level 4) has the greatest impact on hydrogen production, as indicated by the ascending slope. The descending slope at level 3.4 suggests a reduced effect

with lower substrate quantities. Hydrogen production is primarily influenced by reaction time (2), NaBH<sub>4</sub> amount (4), and catalyst amount (4), while temperature (4) and stirring rate (4) have minimal influence, as shown by the shallow slope.

**Figure 3.4** S/N ratio and variable ordering (Taguchi L16 using MOB catalyst), the circle on the parameters indicates that the decreasing slope does not have much influence and the increasing line has significant impact on the parameters.



**Source:** Author (2024).

Table 3.5 presents the S/N ratio and variable ordering (Taguchi L16 using MOB catalyst). The circles on the parameter levels in the figure indicate regions where a decreasing slope exerts little influence on the response, whereas an increasing slope denotes a significant positive impact of the parameter on the process performance. The Taguchi analysis identifies the factor levels as Time (35 min), Temperature (60 °C), NaBH<sub>4</sub> (750 mg), MOB (300 mg), and rotation rate (130 rpm). Among these, NaBH<sub>4</sub>, MOB, and time are the most influential variables, directly affecting H<sub>2</sub> production. Adjusting these factors to their most effective levels maximizes hydrogen generation and reduces variability in the experiments.

**Table 3.5** Optimal conditions for hydrogen generation based on the signal-to-noise (S/N) analysis.

Factors	Levels	Great point
Temperature °C	4	60
Time (min)	3	35
NaBH <sub>4</sub> (mg)	4	750.0
MOB (mg)	4	300.0
Stirring rate (rpm)	2	130

**Source:** Author (2024).

Values from the model (ANOVA) are illustrated in Table 4. The *F-test* is used to identify factors that impact the response model (Karaoglu; Yolcular, 2022a). High values in the *F-test* and in the Sum of Squares (SS) indicate a greater influence of each parameter on the characteristics and final performance of the process (Karmakar *et al.*, 2020; Siegel *et al.*, 2016). Additionally, the *F* value determines the *p-value* in ANOVA, measuring the probability of providing evidence against the null hypothesis (Karaoglu; Yolcular, 2022a). This means that *p-values* less than 0.05 suggest that the associated variable significantly affects response, with a 95% confidence interval (Rajmohan; Palanikumar, 2013; Simão Neto *et al.*, 2024). On the other hand, variables with *p-values* greater than 0.05 do not significantly impact response and, therefore, can be excluded from the model, contributing to the simplification of the analysis (Türkcan; Elçiçek; Özdemir, 2021).

The results in Table 3.6 indicate that the ANOVA analysis highlighted three key factors: time (0.0215), NaBH<sub>4</sub> (0.0024), and MOB (0.0077) as statistically significant within the model's confidence range. Additionally, temperature and RPM had *p-values* above 0.05, indicating no statistical significance and failing to meet the reliability test criteria. In contrast, the three significant factors—time, NaBH<sub>4</sub>, and MOB—showed substantial contributions to hydrogen production, with NaBH<sub>4</sub> contributing 58.87% and the MOB catalyst contributing 27.03%. The higher the percentage contribution of a factor, the more impactful it is on the model's response (Ur Rahman *et al.*, 2021). Conversely, RPM had the least influence,

contributing 0.87%. The "Temperature" factor was removed from Table 3.6 to ensure sufficient degrees of freedom for the analysis and identify significant parameters.

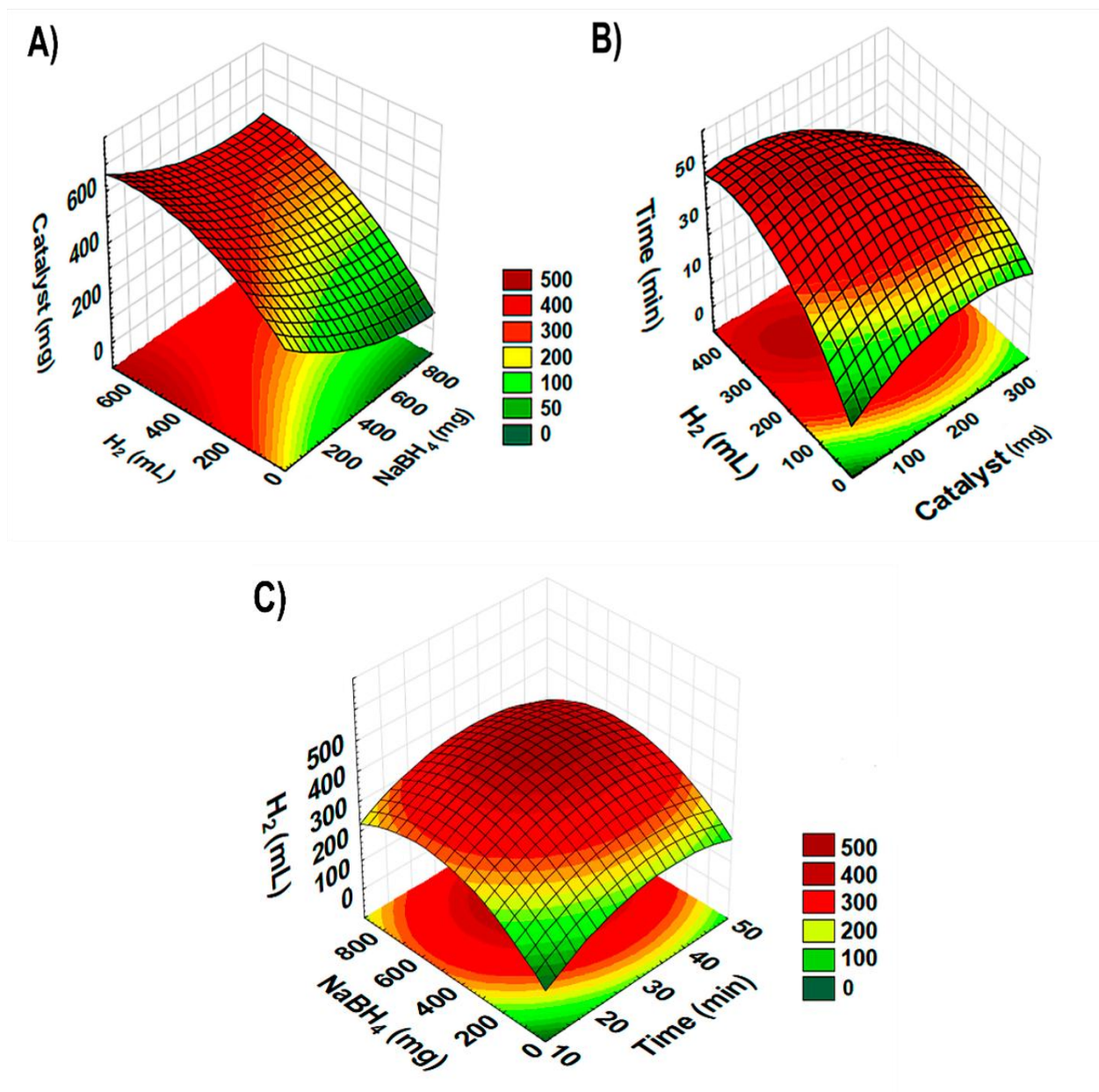
Figure 3.5 illustrates the optimal conditions for H<sub>2</sub> production. In (figure 3.5A), the red area highlights the highest H<sub>2</sub> volume generated, achieved with 500 mg of NaBH<sub>4</sub> and 300 mg of catalyst. (Figure 3.5B) relates H<sub>2</sub> volume, catalyst amount, and reaction time, showing that H<sub>2</sub> production increases with higher catalyst levels and longer reaction times. In (figure 3.5C), which correlates NaBH<sub>4</sub>, reaction time, and H<sub>2</sub> volume, it is evident that more significant amounts of NaBH<sub>4</sub> and extended times maximize hydrogen production. The variation of the NaBH<sub>4</sub> concentration from 250 mg to 750 mg increased the H<sub>2</sub> production by approximately 58.87%, confirming its predominant influence. The increase in the amount of MOB from 100 mg to 300 mg resulted in a 27.03% increase in the H<sub>2</sub> generation, reinforcing its relevance. The extension of the reaction time from 15 min to 35 min led to a 13.23% increase in the H<sub>2</sub> production, although with a smaller impact. Temperature and RPM were identified as less influential factors by the Taguchi method (contributions below 5%) and, therefore, excluded from the sensitivity analysis.

**Table 3.6** Linear model (ANOVA) of the factors influencing the catalytic activity of the MOB catalyst, including degrees of freedom (DF), sum of squares (SS), mean of squares (MS), F value, p-value and percentage contribution of each factor. The factors analyzed include time, NaBH<sub>4</sub> concentration, MOB, Temperature and Stirring rate RPM.

Factors	DF	SS	MS	F value	p-value	Contribution (%)
Time	3	94.518	31.506	17.16	0.0215	13.23%
NaBH <sub>4</sub>	3	420.413	140.137	76.33	0.0024	58.87%
MOB	3	193.029	64.343	35.04	0.0077	27.03%
Temperature	(3)	-	-	-	-	-
RPM	3	6.233	2.077	1.13	0.4606	0.87%
Total	12	714.195	-	-	-	100.00

**Source:** Author (2024).

**Figure 3.5** 3D contour plots. A) Volume of H<sub>2</sub> generated by the reaction of NaBH<sub>4</sub> with catalyst in aqueous medium; B) Influence of reaction time and amount of catalyst on H<sub>2</sub> production; C) Response surface showing the volume of H<sub>2</sub> generated as a function of reaction time and amount of NaBH<sub>4</sub>.



Source: Author (2024).

### 3.3.3 Materials characterization

#### 3.3.3.1 Scanning Electron Microscopy (SEM)

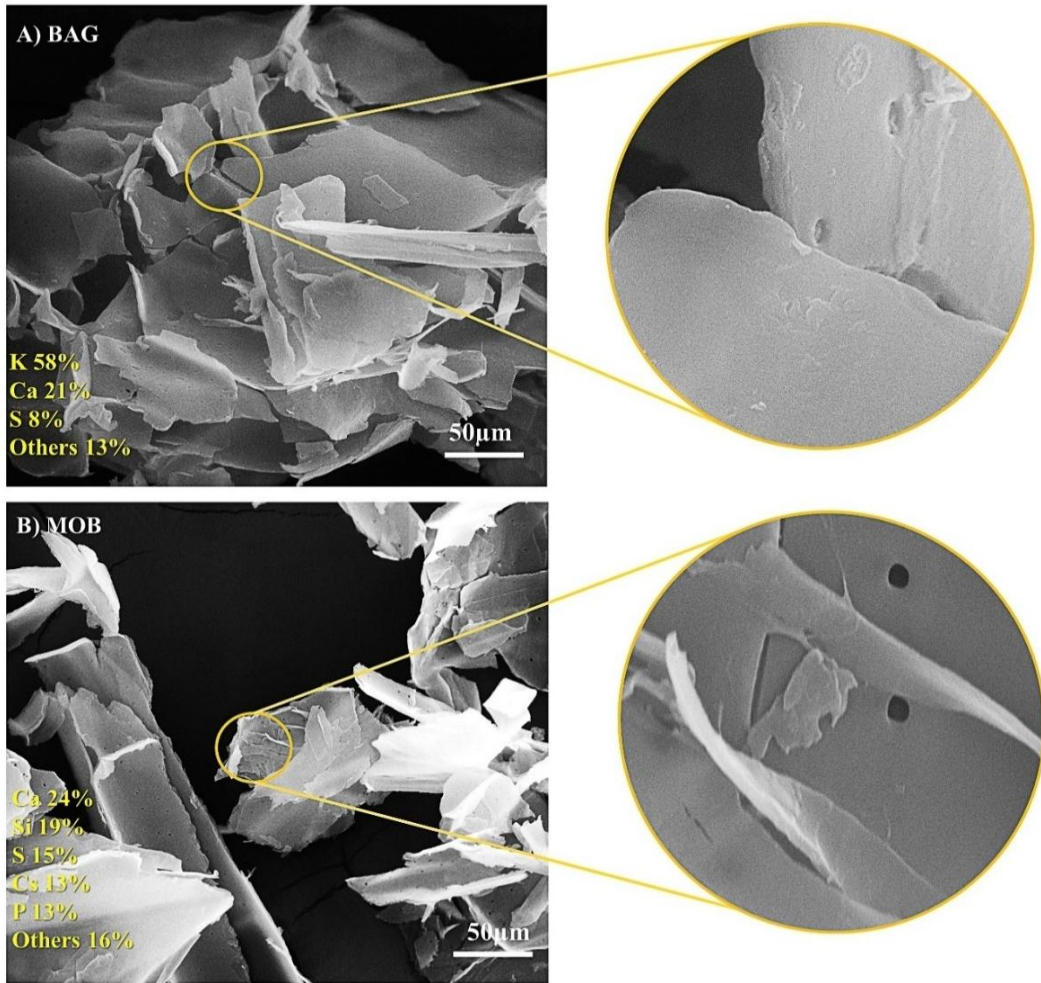
SEM analysis of the BAG samples (A) without chemical treatment and (B) MOB impregnated with 15%  $\text{H}_3\text{PO}_4$  is shown in Figure 3.6. In Figure 3.6A, it can be seen that the structure of the untreated sugarcane bagasse has a smooth and flat surface, although with some visible pores. In Figure 3.6B, after treatment with  $\text{H}_3\text{PO}_4$ , a significantly more porous structure can be seen, with well-defined pores and the presence of acid on the surface of the catalyst.

The increase in porosity is directly related to the greater number of active sites, which are regions on the surface of the catalyst where chemical reactions occur. The more active sites available, the greater the efficiency of the catalyst, as it can accelerate reactions more effectively and on a larger scale (Isahak; Al-Amiery, 2024). In addition, greater accessibility to the pores facilitates the transport and storage of molecules within the structure, optimizing their ability to capture and interact with the reagents (Wang, Aixia; Ma; Zhao, 2024).

Previous studies have shown that biomass-derived catalysts modified with acids or metals exhibit significant variations in surface area, directly impacting their catalytic efficiency. According to Bekirogullari *et al.* (2020), coffee powder impregnated with Zn showed good catalytic activity when adding  $\text{ZnCl}_2$  to the material, an increase in surface area and pore volume was observed, however, there was a reduction in pore size. This means that the Zn particles did not only remain on the surface, but also filled the micropores of the material (Bekirogullari, 2020).

While for Fangaj *et al.* (2020), a comparison was made between before and after catalyst treatment. Initially, the surface was smooth and flat. With the addition of  $\text{H}_3\text{PO}_4$ , a porous structure was formed. In addition, the burning process resulted in a more homogeneous surface, indicating that the increase and formation of pores increase the surface area of the material, allowing greater exposure of the active sites (Fangaj; Ceyhan, 2020).

**Figure 3.6** SEM image and XRF elemental analysis of sugarcane bagasse. A) Pure BAG without treatment and B) MOB catalyst impregnated with 15% phosphoric acid.



**Source:** Author (2024).

### 3.3.3.2 X-ray Fluorescence (XRF)

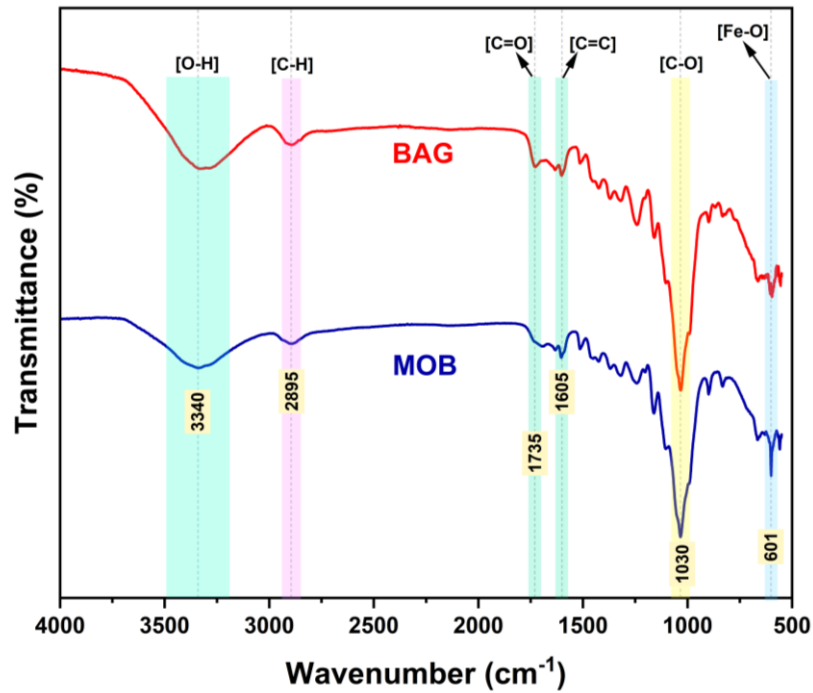
Elemental analysis by XRF was conducted on the BAG and MOB samples to identify their elemental composition. Figure 3A shows that the BAG sample consists of Potassium (K) 58%, Calcium (Ca) 21%, Sulfur (S) 8%, and other elements like Silicon (Si) 7%, Manganese (Mn) 2%, Iron (Fe) 2%, Copper (Cu) 1%, and Zinc (Zn) 1%, together making up 13% of the total composition. In comparison, the MOB sample (Figure 3B) demonstrates significant changes, with Potassium no longer detected, Calcium increasing to 23% (up by 3%), Silicon rising to 19% (an increase of 11%), and Sulfur increasing to 15%. Additionally, a new element, Phosphorus (P), appears at 12%, directly attributable to the phosphoric acid treatment, confirming the success of the process.

The absence of Potassium in the MOB sample can be explained by the chemical reactivity of potassium compounds during the acid treatment, leading to their dissolution and subsequent removal during washing steps. This modification in elemental composition aligns with the treatment goal of enhancing the material's catalytic potential. Furthermore, the SEM and XRF analyses highlight the effectiveness of the  $\text{H}_3\text{PO}_4$  treatment in altering the material's structure and composition, as evidenced by the increased porosity shown in Figure 6B.

### 3.3.3.3 Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR spectra of the BAG and MOB samples, shown in Figure 3.7, reveal distinct functional groups at bands 3340, 2895, 1735, 1605, 1030, and 601  $\text{cm}^{-1}$ , helping to understand how the samples interact with substances during the process. The 3340  $\text{cm}^{-1}$  band shows strong absorbance due to hydroxyl (O-H) stretching in adsorbed water, carboxylic acids and phenolic compounds suggesting a more hydrophilic surface, improving the dispersion of the reaction phase and maximizing the contact between the substrate and the catalytic sites (Sharma, Surbhi *et al.*, 2021). The 2895  $\text{cm}^{-1}$  band indicates aliphatic (C-H) stretching, revealing hydrocarbon chains (Mehta; Saboo, 2024). The 1735  $\text{cm}^{-1}$  band corresponds to (C=O) stretching in carbonyls of ketones and aldehydes (Hassan, Md. Mahafujul *et al.*, 2024). while the 1605  $\text{cm}^{-1}$  band represents aliphatic (C=C) groups found in alkenes and aromatics (Yahya; Elshaarawy, 2024). The 1030  $\text{cm}^{-1}$  band is attributed to phosphate groups ( $\text{PO}_3$ )<sup>2-</sup> (Ghauri *et al.*, 2009). The 601  $\text{cm}^{-1}$  band, associated with (Fe-O) vibrations, confirms the presence of metals in smaller quantities (de Menezes *et al.*, 2024). In addition, FTIR analysis demonstrated the presence of phosphate groups (P–O–P, band 935  $\text{cm}^{-1}$ ) after treatment with  $\text{H}_3\text{PO}_4$ , which act as active acid sites, favoring the activation of  $\text{NaBH}_4$  and facilitating the release of  $\text{H}_2$  (Ghauri *et al.*, 2009).

**Figure 3.7** FTIR spectra of sugar cane bags. Pure form (BAG) and modified catalyst (MOB).

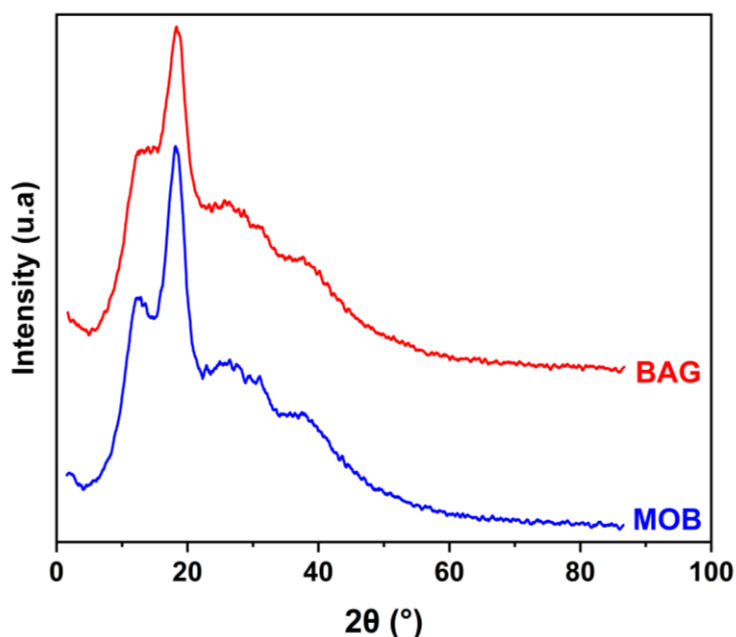


Source: Author (2024).

#### 3.3.3.4 X-ray Diffraction (XRD)

The XRD patterns were used to estimate the catalyst's crystallinity (MOB). Two sharp diffraction peaks observed at  $2\theta = 12.65^\circ$  and  $18.17^\circ$  indicate a high crystallinity index. Peaks within the  $15^\circ$  to  $24^\circ$  range are associated with crystalline cellulosic materials in its composition, such as sugarcane bagasse (Johar; Ahmad; Dufresne, 2012). The crystalline structure of cellulose results from interactions between adjacent molecules, which bond through hydrogen bridges and Van der Waals forces (Zhang, Yi-Heng Percival; Lynd, 2004). These peaks are associated with the crystalline structures of the sugarcane bagasse (BAG) sample. However, the catalyst (MOB), after undergoing alkaline and phosphoric acid treatments, showed an increase in crystallinity, as depicted in Figure 3.8. This increase is attributed to the progressive removal of non-cellulosic amorphous components during the chemical treatments, which promotes the reorganization and enhancement of the crystalline structure (Johar; Ahmad; Dufresne, 2012).

**Figure 3.8** XRD diagram for the determination of the crystallinity and amorphous phase of the BAG and MOB samples.



**Source:** Author (2024).

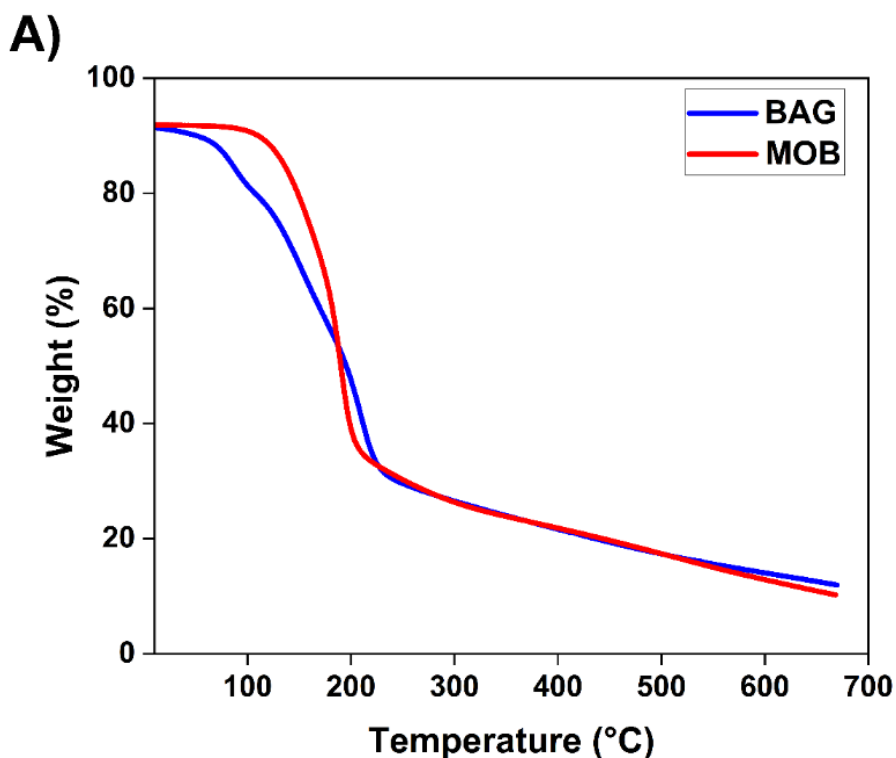
### 3.3.3.5 Thermogravimetric Analysis (TGA)

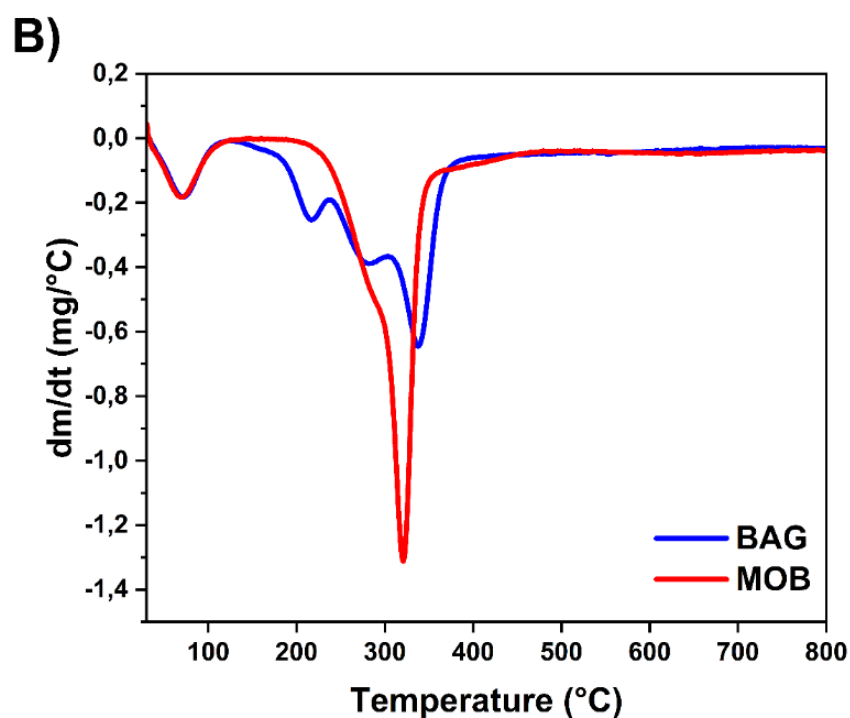
Figure 3.9 illustrates the (TGA) and (DTG) of the samples heated from 30 to 800 °C under an inert nitrogen (N<sub>2</sub>) atmosphere. It is worth mentioning that the materials were not impregnated with metal, reducing possible reactions between the biomass constituents and N<sub>2</sub>. Thus, the choice of the nitrogen atmosphere proved to be suitable for the study, as it is a low-cost and widely available gas than Argon.

The BAG sample (Figure 3.9A) exhibited four stages of degradation: the first stage (70–110 °C) involved a 12.36% weight loss due to moisture and light volatile compound removal 89. The second stage, occurring at 215 °C, recorded an additional weight loss of 25.43%, mainly attributed to the decomposition of hemicellulose. In the third stage, a weight loss of 36% was observed at 262 °C, indicating the decomposition of cellulose, one of the main polysaccharide components of biomass 90. Finally, at 376 °C, the fourth stage exhibit a weight loss of 22.16%, associated with the degradation of lignin, the most thermally resistant fraction of biomass 91. In contrast, the MOB sample (Figure 3.9A) exhibited two stages: 8.18% weight loss at 110 °C (moisture removal) and 60.89% at 323 °C, indicating combined decomposition of cellulose and

hemicellulose, suggesting a simpler structure. The (DTG) provides critical insight into the thermal behavior of the BAG and MOB samples by identifying the temperatures at which mass loss occurs most rapidly. As shown in Figure 3.9B, the DTG graph highlights the key temperatures of significant thermal transformations. Both samples exhibit pronounced peaks around 300 °C, indicating rapid decomposition processes or significant reactions occurring within this range. This suggests that 300 °C is a critical temperature for the thermal degradation of both materials, likely reflecting the breakdown of major biomass components.

**Figure 3.9** TGA and DTG analysis of sugarcane bagasse under N<sub>2</sub> atmosphere. A) TGA curves of pure sugarcane bagasse and sugarcane bagasse modified with phosphoric acid. B) DTG curves show the weight loss rates as a function of temperature.





Source: Author (2024).

### 3.3.4 Characterization of the Generated Gas

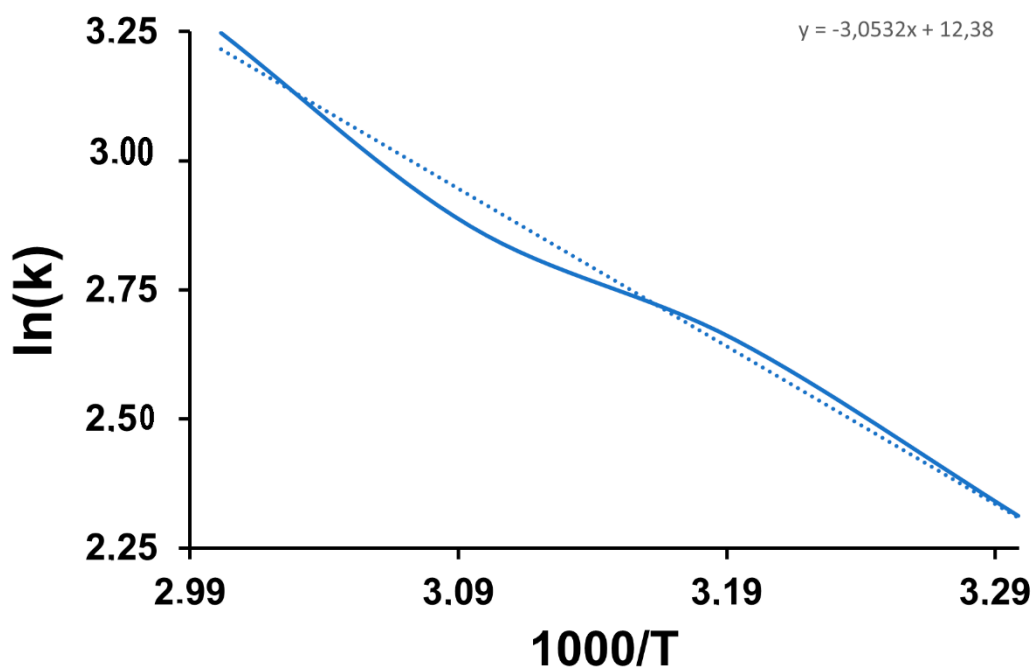
Chromatographic analysis confirmed the presence of hydrogen in the gas generated by the hydrolysis of sodium borohydride, indicating the efficiency of the  $\text{NaBH}_4$  conversion process. However, possible inaccuracies in the isolation of the gas collection bag system allowed atmospheric gases, such as nitrogen (32.7%) and oxygen (13.7%), to dilute the hydrogen (53.6% by volume), compromising accurate quantification and possibly underestimating hydrogen production. Despite these inaccuracies, the MOB system showed potential for hydrogen production. Future studies should include purging the reactor with inert  $\text{N}_2$  gas and improved gas collection and analysis methods for more accurate data.

### 3.3.5 Activation Energy ( $E_a$ ) of materials

The Arrhenius diagram (Figure 3.10) evaluated the efficiency of the MOB catalyst in the hydrolysis of  $\text{NaBH}_4$ , comparing it to other catalysts such as tea leaves (37 kJ/mol) (Srivastava *et al.*, 2024), CoB-supported *Spirulina platensis* microalgae strain (35.25 kJ/mol)

(Saka; Kaya; Bekiroğullari, 2020b) and Co-Ce-B/CSAC (48.22 kJ/mol) (Zhang, Xiuli *et al.*, 2019). Sugarcane bagasse (BAG) presented  $E_a$  33.01 kJ/mol, while the MOB catalyst demonstrated the highest efficiency, with activation energy ( $E_a$ ) approximately 25.38 kJ/mol, indicating a superior reaction rate due to its high stability and phosphorus-enriched composition. The results indicate that the activation energy was significantly reduced (from 33.01 kJ/mol to 25.38 kJ/mol) due to the introduction of phosphoric acid functional groups, which improved the catalytic performance. These results highlight MOB as the most promising option for the hydrolysis of  $\text{NaBH}_4$ .

**Figure 3.10** Arrhenius plot for the catalytic hydrolysis of  $\text{NaBH}_4$ , showing the logarithmic relationship between the rate constant  $\ln k$  and the inverse temperature  $1000/T$ . The plot reveals the activation energy  $E_a$  and the temperature dependence of the process.



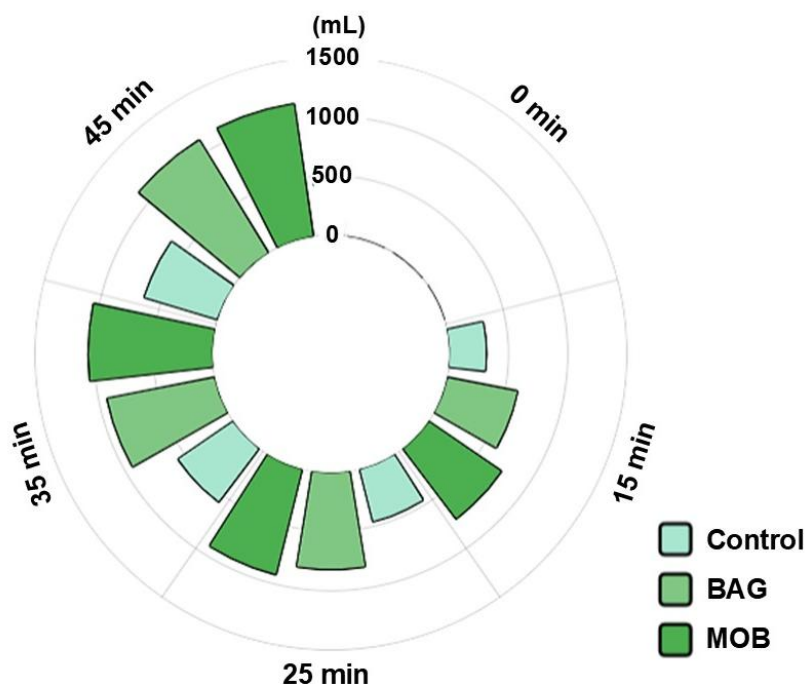
**Source:** Author (2024).

### 3.3.6 Hydrogen Generation Rate (HGR)

The results of the catalytic hydrolysis of  $\text{NaBH}_4$  show that BAG and MOB present close hydrogen generation rates (HGR):  $83.1 \text{ mL H}_2 \text{ min}^{-1} \text{ g}^{-1}$  for BAG and  $84.1 \text{ mL H}_2 \text{ min}^{-1} \text{ g}^{-1}$  for MOB, with a difference of only  $1.04 \text{ mL H}_2 \text{ min}^{-1} \text{ g}^{-1}$ . This proximity suggests similar

catalytic characteristics between the materials, with MOB showing a slight superiority (Dabaro; Kim, 2024). A graphical analysis (Figure 3.11) of the accumulated hydrogen volume over time confirms the superior performance of BAG and MOB compared to the control, which presents significantly lower hydrogen generation (Patel, Dijit M. *et al.*, 2024). The rapid increase in hydrogen volume during the first 25 minutes indicates high initial catalytic activity of both materials. Although the performance difference is small, the advantage of MOB can be decisive for applications that require maximum efficiency and optimization of the occurrence time. This superior efficiency positions MOB as a preferred option in continuous hydrogen generation systems.

**Figure 3.11** Hydrogen production rate via sodium borohydride hydrolysis Volume of hydrogen gas (mL) generated over time (min) during sodium borohydride hydrolysis in the presence of MOB, BAG, and control.



Source: Author (2024).

### 3.3.7 Catalyst Reuse and Storage Capacity

The efficiency and reusability of the produced MOB catalyst were evaluated in cycles at room temperature (25 °C). (Figure 3.12) shows the reuse column, which correlates the storage time with the catalytic activity recovered from the MOB. The catalyst was subjected to six reuse

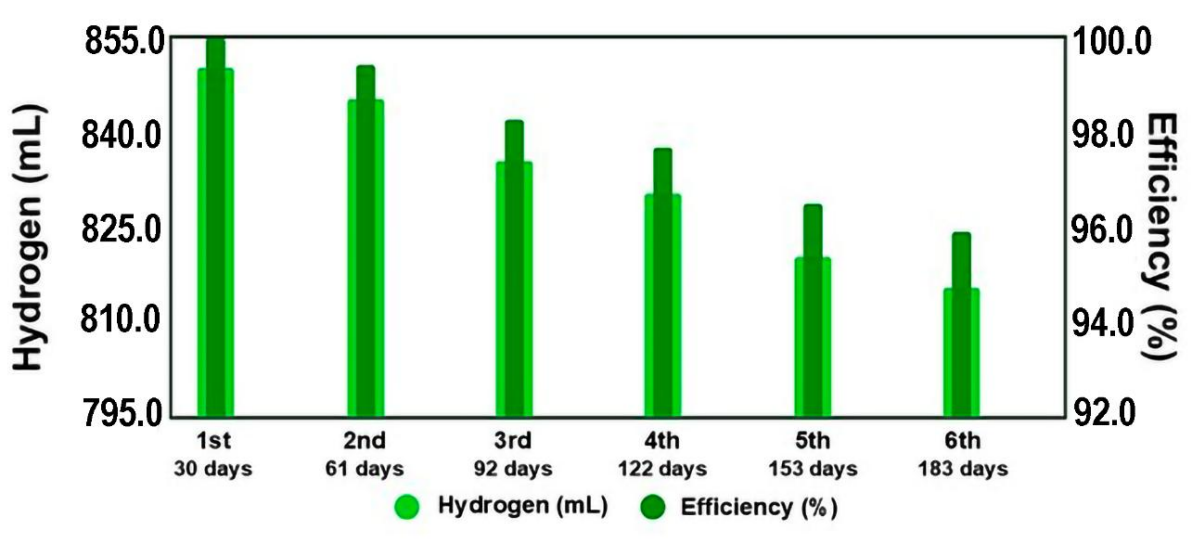
cycles for the hydrolysis of sodium borohydride ( $\text{NaBH}_4$ ) under previously established optimal conditions, including reaction time (35 min), temperature (60 °C),  $\text{NaBH}_4$  concentration (750 mg), catalyst mass (MOB, 300 mg) and rotations per minute (135 rpm). During each experimental cycle, the catalyst was filtered and thoroughly washed with deionized water to remove the accumulation of sodium metaborate present on the surface of the MOB catalyst. The volume of hydrogen ( $\text{H}_2$ ) collected at the end of each cycle was 850, 845, 835, 830, 820, and 815 mL, respectively, as shown in (Figure 3.12).

After the sixth reuse cycle, using the percentage reduction calculation, a 4.11% reduction in the catalytic efficiency of the MOB was observed, attributed to the partial accumulation of sodium metaborate ( $\text{NaBO}_2$ ) and the reduction of acid activity on the catalyst surface (Huang, Yao-Hui *et al.*, 2012). Partial removal of metaborate is crucial to restore catalyst activity. To mitigate this loss, washing with deionized water, a green solvent, was used as a regeneration strategy. However, its effectiveness can be limited by the partial solubility of metaborate and its strong interaction with the catalyst. In view of this, some approaches can improve residue removal. Optimizing the washing protocol, with precise temperature and pH control, can increase efficiency without compromising structural stability. In addition, protective coatings such as phosphoric acid on the catalyst surface can reduce irreversible adsorption of byproducts, extending its useful life and improving regeneration throughout reuse cycles.

In this context, stability over multiple use cycles is a crucial factor for the viability of biomass-derived catalysts. Saka *et al.* (2020) observed that, after successive reuses, the microalgae-based catalyst still maintained 37% of its original efficiency (Saka; Kaya; Bekiroğullari, 2020a). This reduction in catalytic activity was mainly attributed to the loss of active sites during the material washing and recycling processes.

Similarly, (Bekiroğullari *et al.*, 2020) reported that, although they achieved 100% initial conversion, the time required for reaction completion increased progressively, going from 1.4 min to 2.8 min after the fifth reuse cycle (Bekiroğullari, 2020). This decline in efficiency may be related to the structural degradation of the catalyst over the successive washing and recycling processes. On the other hand, the MOB catalyst developed in the present study demonstrated a reduction of only 4.11% in efficiency after six reuse cycles. This performance can be attributed to the greater chemical and structural resistance provided by the treatment with  $\text{H}_3\text{PO}_4$ .

**Figure 3.12** Catalyst reuse and efficiency (MOB) evaluated over 183 days.



**Source:** Author (2024).

### ***3.3.8 Estimation of photovoltaic solar energy consumption***

The photovoltaic mini-generation plant at UNILAB's Auroras Campus produces an average monthly generation of 32642 kWh. Based on this value, energy consumption estimates for the sustainable hydrogen production system can be made based on the plant's energy output. To process and synthesize 20 g of the catalyst, 69 kWh of energy were consumed. Thus, the energy required to produce 1 kg of the catalyst can be estimated at 3450 kWh, corresponding to approximately 10.6% of the plant's average monthly generation. Additionally, given that 300 mg of the catalyst is sufficient to produce 850 mL of H<sub>2</sub>, it can be estimated that producing 1000 L of H<sub>2</sub> requires 353 g of catalyst.

Considering that 300 mg of catalyst is used to generate 850 mL of H<sub>2</sub> and the system must be heated for 35 minutes, the energy required to produce 1000 L of H<sub>2</sub> is approximately 136 kWh. This represents less than 0.5% of the average monthly generation of the photovoltaic plant, demonstrating the system's energy feasibility on a sustainable scale. This low fraction of energy consumption relative to the plant's capacity highlights the process's potential for industrial applications, allowing renewable energy sources to be utilized without significantly compromising the campus's energy autonomy. Compared to existing hydrogen production

methods based on renewable energy, the energy cost of 136 kWh appears to be relatively high, however, the estimates of electricity consumption are for laboratory scale (generic equipment), and additional studies are needed for industrial scale (specific and more efficient equipment).

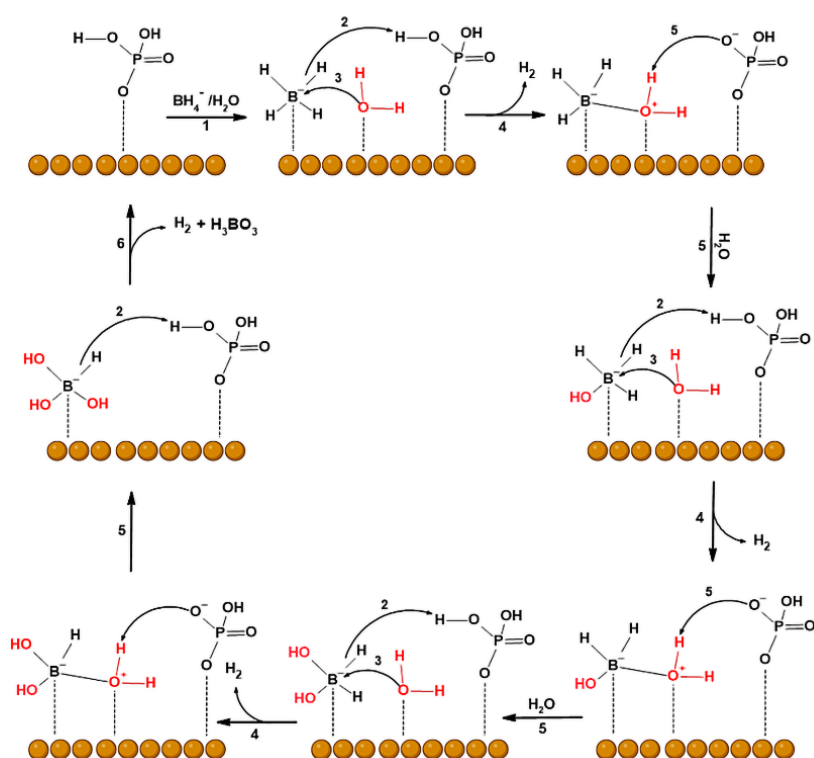
### ***3.3.9 Characterization of reaction mechanism***

To address the influence of  $\text{H}_3\text{PO}_4$  treatment on the catalyst reaction mechanism (MOB), we propose the following mechanism based on characterization data, as depicted in Figure 3.13. The catalyst, consisting of phosphoric acid impregnated on the surface of sugarcane bagasse, exhibits a surface rich in adsorbed phosphoric acid groups. The mechanism is divided into six stages.

In the first stage (1), adsorption occurs, in which borohydride and water molecules are adsorbed on the catalyst surface, interacting with the phosphoric acid groups. In the second stage (2), there is proton abstraction, in which the negatively charged hydride ion dissociates from the boron atom and abstracts a proton from the phosphoric acid group on the catalyst surface. This process is facilitated by the acidity of the adsorbed phosphoric acid groups, which play a crucial role in the activation of the hydride species. Subsequently, in the third step (3), the boron atom undergoes a nucleophilic attack by an activated water molecule.

In step (4), the catalyst is regenerated, in which the water molecule is subsequently deprotonated by the conjugate base (phosphate group) formed during proton abstraction in step (2), thus restoring the active catalytic site for future reactions. In the final steps, (5) and (6), the process continues until  $\text{H}_3\text{BO}_3$  and 4 moles of  $\text{H}_2$  are produced. This mechanism demonstrates how treatment with phosphoric acid enhances the catalytic activity of MOB, as it introduces functional acid groups that facilitate both proton transfer and nucleophilic activation. Furthermore, the great advantage of this process lies in the synergy between the adsorbed phosphoric acid groups and the catalytic steps, making it highly efficient.

**Figure 3.13** Proposed reaction mechanism for  $\text{H}_2$  production using MOB catalyst.



**Source:** Author (2024).

### 3.3.10 Main strengths, limitations and perspectives

Future studies on hydrogen production and the development of catalysts from agro-industrial biomass may benefit from the results obtained in this work. In particular, the use of residual biomass impregnated with 15%  $\text{H}_3\text{PO}_4$  demonstrated great potential for the hydrolysis of  $\text{NaBH}_4$ , with advantages such as low synthesis cost, easy biomass production, 850 mL of  $\text{H}_2$  generated using Taguchi (L16) and reduced number of experiments. In addition, the integration with photovoltaic energy brings benefits as it is a clean energy, making the  $\text{H}_2$  production system sustainable, since the energy for the equipment comes from solar energy. Thus, paving the way for future investigations aimed at optimizing metal-free catalysts impregnated with acids and optimized  $\text{H}_2$  production. However, to make the implementation of this model viable in industrial processes on a large scale, it is essential to explore strategies to mitigate the loss of catalytic activity throughout its use, such as strict washing and drying protocols, use of advanced catalyst synthesis techniques that do not harm the environment, and more studies in the literature using  $\text{H}_3\text{PO}_4$ , requiring complementary studies to ensure its technical and economic viability.

### 3.4 CONCLUSION

In this study, hydrogen production through sodium borohydride ( $\text{NaBH}_4$ ) hydrolysis was significantly improved with the use of a catalyst derived from sugarcane bagasse (MOB). Characterization of the samples by FTIR, XRD, XRF, TGA, SEM, and GC confirmed the effectiveness of the structural modifications in the biomass, resulting in a catalyst with greater reactive potential. In terms of stability, reuse tests indicated a reduction of only 4.11% in the catalytic efficiency of MOB. Furthermore, energy estimates showed that the production of 1 kg of catalyst and 1000 L of  $\text{H}_2$  would require 10.6% and 0.5%, respectively, of the energy generated by a photovoltaic plant, highlighting the viability of the system for industrial applications. Overall, this study explored the optimized conditions for hydrogen production, highlighting the promising performance of MOB, which combines high catalytic efficiency with significant advantages for practical applications in  $\text{NaBH}_4$  hydrolysis.

## 4 FINAL CONSIDERATIONS

In summary, the application of the MOB catalyst in the sodium borohydride hydrolysis reaction for hydrogen production emerges as an innovative and sustainable alternative, offering a practical solution for repurposing lignocellulosic waste, such as sugarcane bagasse. Analyses conducted using techniques like FTIR, XRD, XRF, TGA, SEM, and GC confirmed that the structural modifications made to the biomass significantly enhance its catalytic potential. Moreover, the MOB catalyst maintained high efficiency even after multiple reuse cycles, with only a 4.11% reduction, highlighting its stability and scalability.

The use of the Taguchi method to optimize the catalyst underscores the importance of systematic approaches in developing more efficient and environmentally friendly processes. Hydrogen production was also evaluated through an integrated energy perspective, with a photovoltaic plant supplying just 0.5% of its energy to power the hydrolysis process, ensuring low energy consumption and directly contributing to the system's sustainability. This approach promotes renewable energy use, further reducing the environmental footprint and enhancing its industrial application potential.

Additionally, bibliometric analysis revealed significant growth in research related to sugarcane bagasse since 2016, emphasizing its relevance in the global energy transition. Advances in biomass conversion technologies have enabled the development of high-value products such as biofuels and biofertilizers while promoting sustainability and reducing carbon emissions. However, challenges such as large-scale economic feasibility and the implementation of public policies to foster adoption remain barriers to overcome. Finally, the study results confirmed the high performance of the MOB catalyst, validating its theoretical and practical effectiveness. The use of sugarcane bagasse as a raw material highlights the feasibility of repurposing lignocellulosic waste in technologically significant processes. Integrating lignocellulosic biomass into hydrogen production not only represents an environmentally responsible solution but also marks a significant advance toward clean and sustainable technologies. The combination of biomass and photovoltaic energy in the process reinforces a commitment to sustainability, positioning this method as a substantial contribution to the global energy transition and the development of a low-carbon economy.

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