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ISABELLY SILVEIRA FREITAS

**PRODUÇÃO DE HIDROGÊNIO A PARTIR DE BOROHIDRETO DE SÓDIO
UTILIZANDO RESÍDUO DE CASCA DE COCO VERDE COMO CATALISADOR**

REDENÇÃO – CE

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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.

Orientador: Prof. Dr. José Cleiton Sousa dos Santos

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ABSTRACT

The growing demand for renewable energy sources has driven research on the sustainable use of residual biomass. In this context, green coconut husk (GCF) has emerged as a promising raw material for the production of high-value-added inputs and energy generation. To understand the progress of this field, a bibliometric analysis of scientific publications indexed in the Web of Science database was carried out, covering the period from 2000 to 2024. A total of 1,211 publications were identified, highlighting the significant increase in interest in the valorization of this biomass since 2017. Countries such as India, China and Brazil lead research in the area, focusing on chemical conversion, production of activated carbon and application in contaminant adsorption. The analysis revealed that the main strategic themes include the development of biomass conversion technologies and their application in sustainable energy generation. Based on these advances, this study aimed to develop a sustainable catalyst from green coconut shell for hydrogen production via hydrolysis of sodium borohydride (NaBH_4). The study specialization was optimized using the Taguchi method, varying conditions such as temperature, reagent concentration and catalyst amount. The material was characterized by Thermogravimetric Analysis (TGA), X-ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM), confirming its porous structure and the presence of active catalytic sites. The best reaction condition was obtained with 300 mg of GCF-C, 750 mg of NaBH_4 , temperature of 50 °C and stirring of 120 rpm. A kinetic analysis demonstrated that the trends were significantly oriented towards the energy barrier, presenting an activation energy of 28.95 $\text{kJ}\cdot\text{mol}^{-1}$ and a linear and stable hydrogen production rate. The most influential factor in the process efficiency was the NaBH_4 concentration, accompanied by the catalyst quantity and the occurrence temperature. Compared to non-catalyzed systems, GCF-C showed superior performance, evidencing its potential as a viable and low-cost alternative for hydrogen production. The results confirm that green coconut shell can be used as an efficient catalyst, promoting the valorization of agro-industrial waste and contributing to a circular economy. In addition, the use of this biocatalyst represents a clean approach for hydrogen generation, aligning with the demands of the energy transition. Future studies should focus on the stability of the development in successive reaction cycles and the scalability of the process for industrial applications.

Keywords: Biomass; sustainable catalyst; borohydride hydrolysis; hydrogen production; circular economy.

RESUMO

A demanda crescente por fontes de energia renováveis tem impulsionado pesquisas sobre o aproveitamento sustentável de biomassas residuais. Nesse contexto, a casca de coco verde (GCF) tem se destacado como matéria-prima promissora para a produção de insumos de alto valor agregado e geração de energia. Para compreender o avanço desse campo, foi realizada uma análise bibliométrica de publicações científicas indexadas na base de dados Web of Science, abrangendo o período de 2000 a 2024. Foram identificadas 1.211 publicações, destacando o aumento significativo no interesse pela valorização dessa biomassa desde 2017. Países como Índia, China e Brasil lideram as pesquisas na área, com foco na conversão química, produção de carvão ativado e aplicação em adsorção de contaminantes. A análise revelou que os principais temas estratégicos incluem o desenvolvimento de tecnologias de conversão de biomassa e sua aplicação na geração de energia sustentável. Com base nesses avanços, este estudo objetivou o desenvolvimento de um catalisador sustentável a partir da casca do coco verde para a produção de hidrogênio via hidrólise do Borohidreto de sódio (NaBH_4). A especialização do estudo foi otimizada por meio do método de Taguchi, variando condições como temperatura, concentração de reagentes e quantidade de catalisador. O material foi caracterizado por Análise Termogravimétrica (TGA), Difração de Raios-X (XRD), Espectroscopia no Infravermelho por Transformada de Fourier (FTIR) e Microscopia Eletrônica de Varredura (MEV), confirmando sua estrutura porosa e a presença de sítios catalíticos ativos. A melhor condição reacional foi obtida com 300 mg de GCF-C, 750 mg de NaBH_4 , temperatura de 50 °C e agitação de 120 rpm. Uma análise cinética demonstrou que as tendências foram significativamente voltadas para a barreira energética, apresentando uma energia de ativação de 28,95 kJ.mol⁻¹ e uma taxa de produção de hidrogênio linear e estável. O fator mais influente na eficiência do processo foi a concentração de NaBH_4 , acompanhada pela quantidade catalisador e pela temperatura da ocorrência. Em comparação com sistemas não catalisados, o GCF-C mostrou um desempenho superior, evidenciando seu potencial como alternativa viável e de baixo custo para a produção de hidrogênio. Os resultados confirmam que a casca de coco verde pode ser utilizada como um catalisador eficiente, promovendo a valorização de resíduos agroindustriais e contribuindo para uma economia circular. Além disso, a utilização desse biocatalisador representa uma abordagem limpa para a geração de hidrogênio, alinhando-se às demandas da transição energética. Estudos futuros devem focar na estabilidade do desenvolvimento em ciclos reacionais sucessivos e na escalabilidade do processo para aplicações industriais.

Palavras-chave: Biomassa; catalisador sustentável; hidrólise de borohidreto; produção de hidrogênio; economia circular.

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1 INTRODUCTION

In recent years, the search for alternative energy sources that are sustainable and environmentally friendly has become one of the main focuses of research in several scientific areas (Iian, 2024; Mahmudul *et al.*, 2022; Requena-Leal *et al.*, 2024; Saba *et al.*, 2023; Sikiru; Abioye; *et al.*, 2024; Wang, Haicui *et al.*, 2024). Hydrogen, due to its unique characteristics, such as high energy content and the absence of pollutant emissions during its combustion, has been widely considered as one of the most promising fuels to replace fossil fuels (Hui *et al.*, 2024; Liu, Guoqing *et al.*, 2024; Sadeq *et al.*, 2024). Among the hydrogen production methods, the hydrolysis of sodium borohydride (NaBH_4) stands out as a promising route for hydrogen generation (Abdelhamid, 2021a), being a catalyzed process that can achieve high hydrogen generation capacity, with conversion rates of more than 90% achieved when the molar ratio of water to sodium borohydride is not less than 4:1 (Liu, Bin Hong; Li; Suda, 2009).

NaBH_4 , a stable compound, is capable of releasing hydrogen when in contact with water, and the presence of a suitable catalyst is essential to increase the efficiency of this process (Chen, W. *et al.*, 2017; Fang *et al.*, 2020; Jia *et al.*, 2022). Recent research has explored different materials as catalysts, among which those based on metals stand out (Li, Jinghua *et al.*, 2020; Luo *et al.*, 2019; Paksoy *et al.*, 2021; Patil *et al.*, 2021; Walter *et al.*, 2008). However, the search for more economical and environmentally friendly alternatives has led to the development of new biomass-based catalytic supports (Lionetti *et al.*, 2024; Turkyilmaz *et al.*, 2024), an abundant and low-cost agricultural residue.

Green coconut husk has promising characteristics, such as high specific surface areas, micropore volumes, and high contents of acidic functional groups (Higai *et al.*, 2021), which can be exploited to support catalytic materials in hydrolysis reactions. Furthermore, the use of biomass residues is aligned with the principles of circular economy and sustainability, offering a potential route to reduce waste and valorize agricultural by-products.

This work aims to investigate the production of hydrogen via hydrolysis of sodium borohydride using catalysts supported on green coconut biomass. In 2 OVERVIEW OF HYDROGEN PRODUCTION AND STORAGE, a theoretical framework on hydrogen production will be presented, focusing on the use of coconut shells and catalysts. In 3 EXPLORING THE POTENTIAL OF COCONUT BIOMASS: BIBLIOMETRIC STUDY, a bibliometric assessment will be presented to analyze renewable energy production from coconut residues, highlighting recent advances and global trends.

In 4 CATALYST SUPPORTED ON COCONUT SHELL BIOMASS FOR HYDROGEN PRODUCTION: SYNTHESIS AND CHARACTERIZATION, the experimental investigation will be conducted, in which catalysts supported on green coconut biomass will be synthesized, characterized, and tested to promote the hydrolysis of sodium borohydride, evaluating their efficiency in hydrogen generation.

1.2 GENERAL OBJECTIVE

- Synthesize and determine the efficiency of a sustainable catalyst, produced from green coconut waste, in the H_2 generation reaction through the hydrolysis of sodium borohydride.

• *Specific Objectives*

- Map scientific production on technologies for obtaining energy from green coconut shells;
- Synthesize and characterize the sustainable catalyst from green coconut residue;
- Determine the efficiency of the sodium borohydride hydrolysis reaction with the catalyst;
- Determine the optimal reaction conditions for maximum H_2 production using the green coconut residue catalyst.

2 OVERVIEW OF HYDROGEN PRODUCTION AND STORAGE

2.1 INTRODUCTION

The growing need for cleaner and renewable energy sources has driven research and development of external technologies for hydrogen production and storage (Byeon *et al.*, 2024). Considered a strategic energy vector, hydrogen has a high energy content and can be used as a fuel in different applications without generating carbon emissions (Kumar, Lee e Park, 2024). However, its production still faces challenges, both in terms of efficiency and economic forecasts.

Several hydrogen production routes have been studied, including hydrocarbon reforming, water electrolysis and chemical processes, such as metal hydride hydrolysis (Sharifishourabi; Dincer; Mohany, 2024). Among these options, sodium borohydride (NaBH_4) stands out for its high hydrogen density and stability, allowing the controlled generation of H_2 through its hydrolysis (Karakaya; Pehlivan; Ceyhan, 2024a). This process, however, depends on the use of practices that increase its efficiency and reduce the need for energy for the event.

The occurrences used in this context can be classified according to their composition and structure, ranging from noble metals, metal alloys and supports structured in different matrices (Huang, Jijiang *et al.*, 2022). The search for sustainable raw materials has led to the development of new alternatives, such as the use of agro-industrial waste, which together have the potential to reduce environmental impact. In addition to hydrogen production and its role in the economy, another fundamental challenge is the storage of H_2 . Different approaches have been explored, including storage in gaseous and liquid states and in the form of chemical compounds, such as metal hydrides. NaBH_4 presents itself as a viable alternative in this context, allowing the release of hydrogen on demand and offering advantages in terms of safety and control of occurrence (Karakaya; Pehlivan; Ceyhan, 2024a).

In view of this scenario, this chapter addresses the importance of hydrogen as an energy vector, its physical-chemical characteristics and the main production methods, with an emphasis on the hydrolysis of sodium borohydride. The different types of studies employed in this process and the possibilities of hydrogen storage are also considered, highlighting the relevance of using agro-industrial waste as a sustainable catalytic support. These concepts provide a theoretical basis for the experiments presented in the following chapters.

2.2 HYDROGEN PRODUCTION

2.2.1 *Importance of Hydrogen*

Energy is fundamental to the execution of nearly all human activities, and the necessary changes for a global low-emission economy, partly motivated by the pursuit of carbon neutrality targets, have been accelerating (Cheekatamarla et al., 2024). However, despite these efforts and the worsening energy crisis, the development of robust energy management solutions has often been underestimated, especially when considering the intermittent nature of some renewable energy sources, such as solar and wind (Şenol; Selimefendigil; Öztop, 2024). Therefore, energy storage systems are essential for addressing the intermittency of renewable energy sources, ensuring a stable and reliable energy supply (Saadat; Farazmand; Sameti, 2024).

Due to its clean and versatile properties, hydrogen has emerged as a promising solution to the challenges of climate change and Sustainability (Alamiery, 2023; Byeon *et al.*, 2024; Yan *et al.*, 2020). Its high energy density and zero-emission characteristics during energy generation processes highlight its potential as a key component of a future clean energy matrix (Kopac, 2024; Kumar; Lee; Park, 2024). A hydrogen-based energy system is an effective choice for both stationary and mobile infrastructure applications, providing high-quality energy in a clean and sustainable manner. This versatility facilitates the integration of hydrogen into existing infrastructures, making it possible to reduce fossil fuel dependence and mitigate climate change in a more cost-effective way (Raj *et al.*, 2024).

Energy generated from hydrogen can also be employed in various industrial applications, ranging from large-scale operations to smaller processes, such as chemical production, which also aids in environmental protection (Obiora *et al.*, 2024). This versatility allows for high-potential end uses, ranging from its application as a raw material to its use in industrial thermal energy and its reconversion into electrical energy (Mehr *et al.*, 2024).

Currently, hydrogen represents about 2% of the global energy market, with an annual consumption of 50 million tons (Eikeng; Makhsoos; Pollet, 2024). The International Hydrogen Council predicts that by 2050, the hydrogen industry will reduce CO₂ emissions by 6 billion tons, create a \$2.5 trillion market, and account for 18% of global energy consumption (Gu *et al.*, 2024).

The growing demand for the transition to a global low-emission economy makes the search for clean energy sources increasingly necessary. In this context, hydrogen's ability to supply high-density energy without emissions, as well as its versatility, reinforces its potential

as a crucial component of the future energy matrix, significantly contributing to the fight against global warming and pollution (Xu, Yaohui *et al.*, 2024).

2.2.2 Characteristics of Hydrogen

Hydrogen, composed of one proton and one electron, is a colorless and odorless gas characterized by its non-toxicity, low density, and high flammability, even at low temperatures. Some of its chemical properties are shown in Table 2.1 (Mohd Amin; Mohd Zaid, 2024).

As the lightest and most abundant element in the universe, hydrogen constitutes approximately 75% of the elemental mass and can be found in various forms, such as diatomic molecules (H₂), atomic hydrogen, and hydrides (Armaroli; Balzani, 2011). The high reactivity of hydrogen enables a wide range of chemical reactions, making it a versatile element ideal for various processes, including fuel cells, rocket propulsion, and industrial applications (Algayyim *et al.*, 2024).

Table 2.1 - Properties of Hydrogen.

Characteristics	Description
Gas	Odorless, colorless, tasteless.
Toxicity	Non-toxic.
Density	1/14th the density of air.
Flammability	Highly flammable.
Abundance	75% of the elemental mass of the universe.
Forms	H ₂ , atomic hydrogen, hydrides.
Reactivity	High chemical reactivity.
Applications	Fuel cells, rocket propulsion, industrial processes.
Clean Combustion	Produces only water vapor when burned with oxygen.
Environmental Impact	Renewable and sustainable energy source.

Source: (Algayyim *et al.*, 2024).

Among the properties shown in Table 2.1, another characteristic of interest for the use of hydrogen-derived energy is its clean combustion, due to the production of H₂O molecules during its combustion, making it a greenhouse gas-free energy source (Ansell, 2023). Thus, hydrogen possesses several properties that make it an alternative energy source for a more sustainable future. However, the low density of hydrogen in its gaseous state compared to air necessitates the use of high pressures and low temperatures (Zhang, Yuemeng; Wang; Yao, 2023).

2.2.3 Production Routes

Various methods are used in hydrogen production, including electrochemical, thermochemical, thermal, chemical, biological, photonic, and sonic processes (Sharifishourabi; Dincer; Mohany, 2024). Renewable energy sources such as solar and wind, as well as biomass, can be utilized for the sustainable production of hydrogen, aiming to achieve an even cleaner energy source (Sikiru; Oladosu; *et al.*, 2024). Currently, hydrogen is primarily obtained from fossil fuels, with more than 90% of global production coming from processes such as steam methane reforming, partial oxidation of methane, and coal gasification (Zhao; Wang, 2024). Despite their promise, carbon-free hydrogen production technologies are still expensive and relatively new, making scalability challenging in the short term (Zheng; Zhao; Wang, 2024).

Water electrolysis, using renewable energy, is an example of a carbon-free hydrogen production pathway that offers a promising solution to reduce dependence on fossil fuels and significantly cut greenhouse gas emissions (Nnabuife *et al.*, 2024). This process splits water into hydrogen and oxygen using an electric current (Xia *et al.*, 2024), occurring through two half-reactions: at the cathode, water is reduced to form hydrogen gas, and at the anode, water is oxidized to generate oxygen gas (Li, Zhao *et al.*, 2024).

Although this method is a promising technique for hydrogen production, one of the main challenges is the high energy consumption associated with the process, due to the slow kinetics of the electrochemical reactions, which require an overpotential significantly higher than the theoretical 1.23 V, typically ranging from 1.8 to 2.4 V (Lotfi; Barati Darband, 2024). This high potential makes electrolysis less efficient and more costly, necessitating advances in catalyst technology and reductions in energy costs to make it a viable alternative for hydrogen production. Currently, producing hydrogen through renewable electrolysis costs around 2.94 USD per kg, but this value could drop to an impressive 0.70 USD per kg by 2050 as new technological innovations transform the market (Hamlehdar; Beardsmore; Narsilio, 2024).

The Steam Methane Reforming (SMR) process stands out as the most established method for industrial hydrogen production, accounting for more than 40% of global production (Faheem *et al.*, 2021). Despite being the most common hydrogen production pathway, SMR generates significant greenhouse gas emissions, producing approximately 9 to 11 kg of CO₂ per kg of H₂ (Saeidi *et al.*, 2023). Its intense energy use and low efficiency in energy resource management present challenges in aligning with current sustainability goals and efforts to combat climate change (Di Giuliano; Gallucci, 2018).

In the conventional SMR process, methane is compressed and heated before being introduced into the reformer, where endothermic methane reforming occurs to form synthesis gas (He *et al.*, 2023). The synthesis gas then passes through the water-gas shift converter, where it is converted into hydrogen and carbon dioxide with the help of nickel-based catalysts. After these reactions, the hydrogen is purified to remove impurities (Masoudi Soltani *et al.*, 2021). While other methods have conversion efficiencies similar to SMR, SMR is far more economical for hydrogen production, with costs of 0.66 USD/kg in the Middle East, 0.73 USD/kg in the USA, and 0.70 USD/kg in Canada (Ayub *et al.*, 2024).

Gasification is a mature technology that breaks down carbonaceous materials, such as coal and waste, using insufficient oxygen to produce gaseous fuels with energy value (Aich *et al.*, 2024; Giwa; Taziwa, 2024). Four types of coal are commonly used in gasification: lignite, sub-bituminous coal, bituminous coal, and anthracite. The process occurs at temperatures ranging from 850–1050°C (Lv *et al.*, 2023; Midilli *et al.*, 2021) and can be carried out by two main methods: surface gasification and underground gasification (Chen, Fangxuan *et al.*, 2024). The cost of hydrogen production from coal gasification falls within the cost range of natural gas conversion, varying according to the depth and thickness of the coal seam (Pei *et al.*, 2016). Without Carbon Capture and Storage, costs range from \$1.17 to \$8.59 per kg of H₂ for surface gasification and from \$1.78 to \$8.47 per kg of H₂ for underground gasification (Chen, Fangxuan *et al.*, 2024). With Carbon Capture, costs increase to \$1.63 to \$9.24 per kg of H₂ for surface gasification and to \$2.11 to \$8.95 per kg of H₂ for underground gasification (Chen, Fangxuan *et al.*, 2024).

In summary, the pathways for hydrogen production vary in terms of development level, efficiency, and environmental impact. Despite the dominance of global hydrogen production by methods such as steam methane reforming and coal gasification, there is a growing pursuit of more sustainable methods, such as water electrolysis using renewable energy. These efforts face challenges such as high costs and technical limitations for implementation. Therefore, the continuous advancement of research to develop techniques that reduce costs and improve efficiency is crucial for cleaner and more sustainable hydrogen production.

2.2.4 Hydrogen Storage

Hydrogen storage is more challenging and expensive than fossil fuel storage, primarily due to its low volumetric energy density (Smith *et al.*, 2024). Although hydrogen has a high calorific value per mass, its calorific value per volume is significantly lower, complicating its

storage and transport, which requires low-temperature and high-pressure conditions to store reasonable quantities (Afanasev *et al.*, 2024).

Hydrogen can be stored in various ways, including as compressed gas, liquid hydrogen (Mishra; Gadore; Ahmaruzzaman, 2024), ammonia, liquid organic hydrogen carriers (Drawer; Lange; Kaltschmitt, 2024) or in metal hydrides (absorption and desorption by metal) (Nivedhitha *et al.*, 2024), providing flexibility in its applications. For large-scale implementations, underground geological formations can be utilized for storage, allowing hydrogen to be stored for extended periods and withdrawn when necessary (Raza *et al.*, 2024).

Table 2.2 presents a comparative analysis of the main hydrogen storage methods, highlighting their characteristics in terms of volumetric density, pressure/temperature, cost, and safety. Although more efficient and compact, the high-pressure method involves high costs and stringent safety requirements due to the elevated pressure involved in this method (Wang, Hongyuan *et al.*, 2024). On the other hand, liquid hydrogen offers the highest volumetric energy density, but the extremely low temperature required (-253°C) makes the process expensive and of moderate safety (Zhang, Tongtong *et al.*, 2023).

Table 2.2 - Characteristics of Hydrogen Storage Methods.

Storage Method	Volumetric Density	Pressure/T°	Characteristics	Cost	Safety
High Pressure	26-42 kg/m ³	350-700 bar	Efficient and compact	High	Stringent
Liquid State	70,9 kg/m ³	-253°C	High density, expensive	High	Moderate
Solid State	>100 kg/m ³	-	High density, expensive	Variable	Low

Source: (Bishnoi; Pati; Sharma, 2024)

Finally, solid-state hydrogen storage with metal hydrides offers advantages over gaseous and liquid forms, including simpler environmental conditions, higher volumetric density, and greater safety (Dong *et al.*, 2024; Liu, Liu *et al.*, 2024; Minko *et al.*, 2024), and allows for long-term storage (Drawer; Lange; Kaltschmitt, 2024). However, it has disadvantages: it requires high temperatures, consuming a lot of energy, and the absorption and release of hydrogen can be slow, especially at low temperatures, affecting efficiency (Klopčič *et al.*, 2023; Nivedhitha *et al.*, 2024).

Another important characteristic of hydrogen is its storage lifespan, which is considerably longer than that of current batteries, helping to mitigate the intermittency of renewable energy sources (Bishnoi; Pati; Sharma, 2024). This capability enables the provision of stable and reliable energy, as well as the conversion of excess renewable energy into stored hydrogen,

which can be consumed during periods of low renewable energy production in continuous cycles (Jeje *et al.*, 2024).

Thus, compared to fossil fuel storage, hydrogen storage is more challenging and expensive, largely due to its low volumetric energy density, necessitating complex methods involving high pressures, low temperatures, and highly reactive metal hydrides. However, despite the challenges, hydrogen's long storage lifespan and its ability to mitigate renewable energy intermittency highlight its potential as a sustainable energy vector.

2.2.5 Metal Hydrides

Hydrides are combinations of hydrogen with metals or metalloids, which are semimetals that react as metals with nonmetals and as nonmetals with metals (Şenol; Selimefendigil; Öztop, 2024). The formation of metal hydrides involves physisorption of H₂, dissociation into H atoms, diffusion to interstitial sites and precipitation of a concentrated hydride phase, with a significant negative entropy change due to the high crystallographic order of the solid (Zohra *et al.*, 2024). Metal hydrides are classified into three types: elementary (hydrogen and a metal), intermetallic (two metals with storage capacities below 2% and temperatures from -50 to 200 °C), and complex (high storage capacities up to 18.5% by weight, but requiring high temperatures) (Drawer; Lange; Kaltschmitt, 2024).

2.2.6 NaBH₄

Hydrolysis of sodium borohydride produces 4 mol of H₂ per mol of NaBH₄ and has a high hydrogen storage capacity (10.6% by weight) (Kirk *et al.*, 2023), in addition to providing high-purity hydrogen (Ekinci; Şahin; Baytar, 2024). NaBH₄ decomposes slowly above 673 K, making thermal activation impractical, but it can release hydrogen efficiently when reacted with water (Graś; Lota, 2021).

Metal hydride hydrolysis is a technique that stands out for generating hydrogen in a safe way for the ecosystem, being accessible, highly reliable, capable of hydrolyzing at low temperatures, low cost and easy to recycle. In addition to providing efficiency in hydrogen production, it minimizes environmental impacts, making it a viable and sustainable option for modern energy applications (Karakaya; Pehlivan; Ceyhan, 2024a). One of the main positive aspects of this process is that 50% of the H₂ generated comes from H₂O molecules, in addition to the secondary product, sodium metaborate (NaBO₂), being harmless to the environment and can be recycled to regenerate NaBH₄, characteristics that combined contribute to the sustainability of the process (Bozkurt; Özer; Yurtcan, 2018).

However, the use of a catalyst is important, as it is essential for the efficient hydrolysis of NaBH_4 at room temperature and pressure, as the reaction does not occur spontaneously under these conditions (Ekinici; Şahin; Baytar, 2024). Thus, the choice of catalyst is essential for efficient hydrogen production from sodium borohydride, as it accelerates the reaction and improves the efficiency and purity of the process (Avcı Hansu, 2023; Liu, Yongjing *et al.*, 2024a). Transition metals, such as Co and Ni, are preferred for their effectiveness and low cost, especially when supported on materials such as TiO_2 and Al_2O_3 to avoid agglomeration and increase reusability (Bozkurt; Özer; Yurtcan, 2018).

2.3 CATALYSTS IN HYDROGEN PRODUCTION

2.3.1 Definition and Function of Catalysts

The first time the term ‘catalysis’ was mentioned was in 1836 when Berzelius identified a new class of substances that could accelerate chemical reactions through a mechanism called ‘catalytic contact’ (Di Monte; Kašpar; Farnetti, 2009). However, it was only in 1894 that Ostwald provided a rigorous definition, describing it as the process of accelerating chemical reactions through the addition of a different material (Roduner, 2014).

Catalysts provide alternative pathways for reactions, requiring less energy to initiate (Isahak; Al-Amiery, 2024a). Simply put, a catalyst increases the speed of a chemical reaction. It works by binding to the reactant molecules, facilitating the formation of the product, and then detaching intact to catalyze new reactions (Kakaei; Esrafil; Ehsani, 2019). The activation energy represents the energy threshold that reactants must overcome for the chemical transformation to begin (Yu *et al.*, 2023). Lowering this energy barrier allows more molecules to have enough energy to react, increasing the speed at which chemical equilibrium is reached. Thus, catalysts accelerate chemical reactions and direct the formation of specific products, but challenges such as the complexity of residues, the value of recovered materials, and the need for efficient and economical catalysts still limit large-scale industrial applications (Huang, Jijiang *et al.*, 2022).

2.3.2 Types of Catalysts

The catalysts used in industry can be classified into three main groups: heterogeneous catalysts, such as porous solids and nanoparticles, which do not mix with the reagents; homogeneous catalysts, which are in the same phase as the reagents, meaning they are dissolved in the mixture; and biological catalysts, such as enzymes, which are proteins with high specificity and efficiency (Zhang, Weijie; Zhang, 2024).

Heterogeneous catalysts, high-tech products, play a fundamental role in economic and social development (Toulhoat et al., 2016). The role of heterogeneous catalysts in the global economy is crucial. It is estimated that around 30% of world production depends directly or indirectly on these materials, with more than 90% of chemicals and fuels being produced by heterogeneous catalytic processes (Toulhoat et al., 2010). Despite their industrial importance, understanding the active sites in heterogeneous catalysts, often dispersed in amorphous materials like silica or confined in microporous structures such as zeolites, remains a challenge due to the complexity of these systems (Ong; Verel; Copéret, 2017).

Homogeneous catalysts, where the metal acts in ionic form, offer high catalytic activity. However, the narrow operational pH range and the tendency to form metal precipitates limit their application, especially in processes aimed at contaminant reduction (Wang, Zhaobo *et al.*, 2024). Even though they are high-performance catalysts, capable of promoting reactions quickly and selectively, even under relatively mild experimental conditions, their economic and environmental viability would be significantly improved if it were possible to efficiently recover the catalysts, preventing the loss of these high-value materials (Román-Martínez; Lecea, 2013).

The different types of catalysts have their benefits and disadvantages. Hydrogen production catalyzed by hydrogenase enzymes (a biological catalyst) is more efficient than that catalyzed by synthetic materials. However, the sensitivity of these enzymes to oxygen, the high production cost, and the low density of active sites limits their application in industrial processes (Wang, Mei *et al.*, 2015).

2.3.3 Catalytic Efficiency

The solubility of homogeneous catalysts in the reaction medium facilitates the formation of intermediate complexes, significantly accelerating the reaction kinetics (Isahak; Al-Amiery, 2024b). The miscibility between catalyst and reagents creates a continuous medium where the formation of intermediate transition states is facilitated, enabling reactions that, under non-catalyzed conditions, would be kinetically hindered (Orege *et al.*, 2022)(Yuan *et al.*, 2020). Homogeneous catalysis offers powerful tools for the investigation and development of new chemical processes, allowing precise control over the selectivity and efficiency of reactions (Anaspure et al., 2022).

Commonly found in the solid phase, heterogeneous catalysts have interfaces that promote the adsorption of reagents, facilitating the occurrence of chemical reactions through complex

mechanisms influenced by the nature of the active sites present on their surface (Isahak; Al-Amiery, 2024b)(Vogt; Weckhuysen, 2022)(Daiyan *et al.*, 2020). Heterogeneous catalysis typically involves the adsorption of reagents on the catalyst surface, where chemical reactions occur and the products are subsequently desorbed (Bañares; Daturi, 2023). The application of heterogeneous catalysts is vast, ranging from large-scale industrial processes to solutions for environmental problems. Through their differentiated mechanisms, these catalysts exemplify the ingenuity harnessed to leverage thermal energy for efficient chemical transformations (Li, Hongliang *et al.*, 2019).

The unique properties of enzymes, such as high efficiency, selectivity, and environmental compatibility, are making biocatalysis an area of great interest for the chemical industry (Chen, Kai; Arnold, 2020). Biocatalysts using immobilized enzymes have attracted considerable attention due to their significant applications in the synthesis of high-value chemicals, pharmaceuticals, and drug intermediates, offering high catalytic efficiency and high yields of target molecules. The main advantages of these biocatalysts include enhanced stability over a wide pH and temperature range (Sulman; Matveeva; Bronstein, 2019). However, the high enzymatic activity common in natural substrates does not always replicate in compounds of industrial interest, restricting the large-scale use of enzymes (Rueda *et al.*, 2016).

2.3.4 Durability and Reusability

Heterogeneous catalysis offers advantages such as high durability of catalysts, high efficiency in reactions, and the possibility of reuse in multiple catalytic cycles (Mukhtar *et al.*, 2022). Heterogeneous catalysts, whether acidic or alkaline, can be easily separated by simple methods such as simple filtration, and are easily reusable (Bohlouli; Mahdavian, 2021). On the other hand, when it comes to homogeneous catalysts, one inevitably encounters the challenge of separating the catalyst (Molnár; Papp, 2017). Recycling homogeneous catalysts without altering their homogeneous nature is one of the main challenges for the industrial feasibility of many reactions. Although there are numerous strategies to develop more efficient and reusable homogeneous catalysts, the literature lacks a comprehensive analysis of the different modifications made to these catalysts, making it difficult to select the best approach for each case (Diekamp; Seidensticker, 2023).

The recycling process of biological catalysts can be achieved through immobilization, which has several unique advantages from a synthetic perspective, such as greater enzyme stability, ease of handling, significantly improved potential, and accessibility for non-specialists

(Foley; Maguire, 2019). The immobilization of biocatalysts, whether isolated enzymes or whole cells, provides greater enzymatic stability, protecting them from product inhibition, temperature, and pH variations. Additionally, immobilization facilitates the recovery and reuse of biocatalysts, adding economic value to biocatalytic processes (Eş; Vieira; Amaral, 2015).

2.4 SUSTAINABILITY AND AGRO-INDUSTRIAL WASTE

2.4.1 *Agro-industrial Waste*

Due to the increase in environmental impacts caused by the large amount of agro-industrial waste generated by the agricultural sector, studies on recycling these residues have been analyzed, as if they are not treated immediately, they contribute to social, economic, environmental and public health problems (Schiebel *et al.*, 2024). These residues are produced through the industrialization of natural products and this causes a great impact, for example, it is estimated that from the processing stage to delivery to the consumer, 1.6 tons of residues are lost or wasted, such as fruit peels, for example (Astudillo *et al.*, 2023; Leyva-López *et al.*, 2020).

In view of this problem, it is necessary to find application methodologies for reusing these residues, which to date have only been studied with the production of biogas, biofuels and animal feed (Ahmed; Gaafar; Nishida, 2024; Saravanan; Senthil Kumar; *et al.*, 2023; Saravanan; Yaashikaa; *et al.*, 2023; Vickram *et al.*, 2023). This type of residue also contains molecules that have positive activities for humans, such as proteins, lipids and polysaccharides, thus showing a positive response to the recycling and application of these materials (Bala *et al.*, 2023; Morales, 2023; Sarker *et al.*, 2023). Examples in the literature show the application of recycling agricultural waste, such as olive waste, which contributes to the production of olive oil and other products used in agriculture, in addition to having antioxidant activity and being able to slow down the process of vascular diseases, cancer and neurological diseases (Lo Giudice *et al.*, 2021), one can also find works where polysaccharides from fruit waste were found that showed antioxidant and anti-inflammatory activities (Mulinari Turin de Oliveira *et al.*, 2022). Given the above, it can be seen that there are alternatives for the application of recycling agro-industrial waste, showing that there are applications, mainly, for treating diseases.

2.4.2 *Green Coconut Shell and its Composition*

Coconut is known for its versatility, as it is a seed, fruit and nut, and therefore has several applications. For example, coconut shells are considered a good lignocellulosic material, that

is, rich in lignin and cellulose, but they are dumped in landfills without proper processing (Sharma *et al.*, 2023). The increase in coconut consumption leads to a considerable increase in waste production, resulting in negative environmental and economic impacts, with the inedible part of the fruit constituting approximately 62-65% of the entire fruit (Obeng *et al.*, 2020). In the literature, we can find applications of waste derived from coconut shells, for example with the study of nanoparticles, biosorbents, thermal insulation, agglomerates and reinforcing agents, but their study is still very limited (Singh *et al.*, 2024a).

The green coconut shell is rich in chemical compounds with antimicrobial and antioxidant properties, so techniques are needed to extract these materials, such as flavonoids, carbohydrates, saponins, reducing sugars, tannins, steroids, glycosides, alkaloids, anthraquinones, phenols and phytosterols, in addition to studies that have already found antiacetylcholinase activities in components of the green coconut shell (Kibria; nessa; Rahman, 2018; Li, Nana *et al.*, 2021).

2.4.3 Processing of Green Coconut Shell

The green coconut shell is the main part of the waste when it comes to processing, so most of the fruit and studies report that around 3 million tons of green coconut shells are discarded into the environment per year (Singh *et al.*, 2024a). When not processed properly, the green coconut shell occupies green areas, such as beaches and land, causing environmental degradation of the place, so it takes around 10 years to degrade in the environment, which can lead to the spread of diseases (Banerjee; Aditya; Saha, 2013). It is estimated that around 10% of the waste that is processed is used by industries, while most of it remains on the beaches, in addition to when it is in landfills under anaerobic conditions, it contributes to the formation of metado, one of the gases that contributes to the greenhouse effect (Barros Dias *et al.*, 2019).

No studies were found in the literature that use the green coconut shell as a catalyst in the production of hydrogen, once again showing the need to apply this work, which will serve as a basis for future research that will be carried out.

2.5 CHALLENGES AND FUTURE TRENDS

2.5.1 Current Challenges

Although there are a number of potential applications for energy generation from coconut biomass, there are also certain challenges that must be overcome (Ighalo *et al.*, 2023b). The major technical challenges faced in the conversion of biomass into biofuels are all interrelated to its ash content (Alabi; Sambo, 2023). The high ash content may reduce heating efficiency

and heat transfer, and increases agglomeration and fouling in combustion chambers, affecting the biomass decomposition rate and promoting ignition problems (Alabi; Sambo, 2023; Simões *et al.*, 2024). Some of the challenges are focused on modeling biomass pyrolysis due to the influence of inorganic species and the interaction among the distinct components (Hameed *et al.*, 2019; Simões *et al.*, 2024; Vuppaladadiyam *et al.*, 2023). The inorganic species present in biomass act as catalysts in pyrolysis reactions, altering pyrolysis temperatures and reaction rates (Simões *et al.*, 2024; Vikram; Rosha; Kumar, 2021; Vuppaladadiyam *et al.*, 2023).

Many innovative technologies in coconut waste processing are still in the early stages (Vieira *et al.*, 2024). Biomass valorization through existing infrastructure requires economically/technically robust routes (Kwon *et al.*, 2024). The maximizing the yield and efficiency of coconut cultivation, harvesting and processing processes requires the adoption and development of innovative agricultural technologies (de Araújo *et al.*, 2024a). Another important point is to expand investments, from coconut production to residue recovery and processing, by redirecting residue to the consumer market through a sustainable circular economy (Fertahi *et al.*, 2023; Vieira *et al.*, 2024).

2.5.2 Future Perspectives

Remarkable advances have been achieved in the use of coconut biomass (Vieira *et al.*, 2024). The effective conversion of biomass into biomass-derived fuel is restricted by the characteristics of the biomass to be used. Therefore, it is essential to understand the biomass physicochemical behavior for better designing and optimizing the conversion equipment[8,9]. Economic/financial feasibility studies on the coconut biomass use as a raw material for pyrolysis represent a potential area for future research as they receive little attention (Azeta *et al.*, 2021b). Coconut biomass is a renewable material with promising prospects in future energy generation, that will reduce reliance on other sources and safeguard national energy security (Agrizzi *et al.*, 2024).

3 EXPLORING THE POTENTIAL OF COCONUT BIOMASS: BIBLIOMETRIC STUDY

Abstract

This study explores the growing interest in the sustainable use of green coconut biomass, particularly coconut husk, for energy production and high-value-added materials. Through an extensive bibliometric analysis of scientific publications from 2000 to 2024, a significant increase in research output since 2017 was identified, reflecting the global need for alternative energy sources and efficient waste management strategies. Coconut husk, rich in lignocellulosic compounds, has shown great potential for thermochemical conversion processes such as pyrolysis, gasification, and biochar production, as well as for the development of activated carbon and adsorbents for wastewater treatment. The analysis highlights key contributors, including India, China, and Brazil, as leading countries in the development of technologies for biomass conversion. The integration of advanced bibliometric tools, including VOSviewer and CiteSpace, revealed emerging research trends and critical gaps, offering insights into future technological advancements. This study underscores the importance of promoting international collaboration and public policies to encourage the sustainable utilization of green coconut biomass.

Keywords: Green Coconut, Biomass, Lignocellulosic Conversion, Sustainable Energy, Waste Valorization

3.1 INTRODUCTION

Every day, a substantial amount of biomass waste, which includes agricultural residues, by-products, lignocellulosic and woody biomass, is generated in large volumes (Sekhon; Kaur; Park, 2021). Thus, every year, a significant amount of residual biomass, in the order of millions of tons, is discarded in landfills or even in open-air dumps, especially in developing nations (Andrade *et al.*, 2024b).

A notable case in Brazil is the fibers and shells of the green coconut (*Cocos nucifera L.*), due to the significant consumption of coconut water both in its natural and processed form in the country's coastal cities (Andrade *et al.*, 2024b). With a global production of 62.5 million tons per year, coconut waste is one of the most abundant biomasses in the world, a condition that highlights the urgent need to develop sustainable and innovative solutions for the use of this waste on a global scale (Azeta *et al.*, 2021a).

Coconut, like other plant biomass, is composed of lignocellulosic materials, consisting of cellulose, hemicellulose and lignin (Khan *et al.*, 2023; Uddin Monir *et al.*, 2024). It is composed of several parts with different applications, from the trunk and leaves of the palm tree, which are used in construction and crafts, to the hard inner shell, which is converted into activated carbon (Ignacio; Miguel, 2021). Coconut waste can also be processed to produce charcoal, which is used as fuel in rural areas (Inseemeeesak; Areeprasert, 2020).

The improper disposal of coconut biomass waste represents a significant environmental problem in many producing countries. When this waste is not disposed of correctly, it often takes up large spaces, where its anaerobic decomposition will release greenhouse gases (Ighalo *et al.*, 2023a). As an alternative to correct disposal, these shells are incinerated, which ends up contributing to the emission of greenhouse gases, such as carbon dioxide (CO₂) (Olatunji; Adedeji; Madushele, 2023).

In this context, in recent years, global interest in the production of fuels and chemical products from the reuse of residual lignocellulosic biomass has grown (Costa *et al.*, 2022). Furthermore, currently, research considered innovative is focused on achieving natural and renewable sources of fuel, which can be converted in a safe and non-polluting manner (Chinnamma *et al.*, 2015). Therefore, Coconut Shells have great potential for exploration, since even if they are partially effectively used as briquettes for energy production, most of them are

discarded into the environment as waste (Trisunaryanti *et al.*, 2022). In addition, they are considered a carbon-neutral raw material, which contributes to the reduction of carbon dioxide emissions into the atmosphere (Costa *et al.*, 2022).

With the help of bibliometric analysis, it is possible to map the main topics, reveal knowledge gaps and guide strategic decisions in research and development. In addition, the following research questions (RQs) are sought to be answered:

- RQ1: How has scientific production progressed in the development of technologies for obtaining energy from Green Coconut Husk?
- RQ2: Which institutions have developed significantly in the publication of articles?
- RQ3: What are the main keywords mentioned in the search?
- RQ4: What are the emerging research areas?

This study aims to explore sustainable approaches to energy generation using green coconut husk, performing an advanced bibliometric analysis to map emerging trends and recurring themes in the scientific literature. The focus is on identifying the main contributors, including authors, countries, and institutions, in addition to highlighting the most relevant research areas and keywords.

The analysis also seeks to reveal new trends and significant advances, while identifying critical challenges that need to be addressed. Thus, this work provides a panoramic and in-depth view of the current state of research, offering valuable insights to guide future investigations and support the formulation of public policies that encourage the sustainable use of green coconut husks in energy production.

3.2 METHODOLOGY

3.2.1 Data Collection

The bibliometric search was conducted using the Web of Science database, following a rigorous methodological approach (Cavalcante *et al.*, 2024; da Silva Aires *et al.*, 2024; Dari *et al.*, 2024b; de Castro Bizerra; Sales; *et al.*, 2024a; Melo *et al.*, 2023; Simão Neto *et al.*, 2024). Initially, the keyword "coconut" was used as the main search term in all fields, resulting in the identification of 23,358 articles. To refine the search and focus on relevant studies, the additional keywords "Biomass," "Energy," and "Waste" were applied in the topic field, reducing the corpus to 263 articles. The search was limited to the period from 2000 to 2024,

covering significant and current publications from the last 24 years.

The selection was restricted to the document types "Article" and "Review article," excluding categories such as "Proceedings article," "Early access," and "Book chapters" to ensure the quality and relevance of the data. The search was conducted exclusively in English, due to the predominance of this language in scientific publications with global reach.

The resulting data were exported in Tab-delimited text (for VOSviewer), Plain text (for CiteSpace) and. BIB (for Bibliometrix) formats, allowing detailed analysis and visualization of patterns and advances in research on the use of green coconut husk as a sustainable energy source.

3.2.2 Data Visualization

Three main software tools were used to analyze the collected data: VOSviewer (<http://vosviewer.com>), CiteSpace, and Bibliometrix. VOSviewer was used to create and visualize bibliometric network maps, focusing on co-authorship, co-citation, and term networks. This tool provided a comprehensive view of the interconnections between various elements in the scientific literature.

CiteSpace was selected to detect emerging patterns and trends, as well as to identify research gaps and visualize the temporal evolution of the topics studied. This tool facilitated the analysis of changes over time and the emergence of new themes in the field.

Bibliometrix, an R package specialized in bibliometric analysis, was used to perform quantitative analyses and produce detailed visualizations. This tool allowed the execution of co-citation, co-authorship, and bibliographic coupling analyses, offering metrics and graphs that complement the analyses of the other tools.

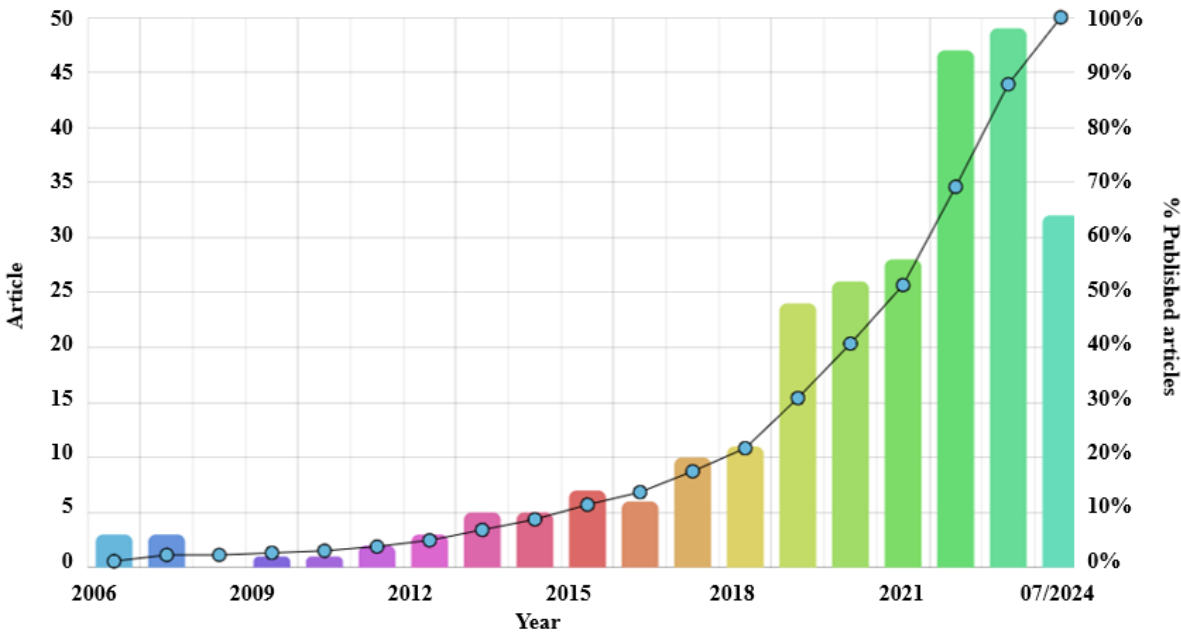
The integration of these tools enabled a robust and accurate quantitative analysis of bibliometric data, clearly revealing research trends and patterns in the field of energy production from Coconut. The combination of these methodological approaches provided a holistic and detailed view of the field, contributing to an in-depth understanding of the research dynamics and technological advancement in this area.

3.3 RESULTS AND DISCUSSION

3.3.1 Annual frequency of Publications and Citations on potential of coconut biomass (2000 – 2024)

The collected data provides a comprehensive and detailed overview of research related to the use of coconut as biomass (Machado *et al.*, 2020), energy source (de Araújo *et al.*, 2024b) and in waste management (Singh *et al.*, 2024b). The analysis covers 2006 to 2024, although the initial search was limited to 2000 to July 2024. This variation suggests that although the search was designed to cover 24 years, relevant publications emerged from 2006 onwards. Significant publications before this year may indicate less interest or development in the early 21st century.

Figure 3.1 - Annual Evolution of Production of Articles with Pareto Analysis (2006-2024).



Source: Author (2024).

With a total of 125 varied sources, the research benefits from a large database (WoS), which strengthens the robustness and credibility of the findings. The final corpus of 263 selected articles, visualized by year in Figure 3.1, reflects a rigorous filtering process, ensuring that only relevant and high-quality studies were included. This methodological rigor is essential to ensure the research results are well-founded and relevant. The annual growth rate of studies, 14.05%, is particularly significant. This rate not only demonstrates growing interest but also suggests a continued and substantial expansion of research in the area. This growth can be

attributed to factors such as increased awareness about sustainability (Bhutto *et al.*, 2019), the search for alternative energy sources (Uddin *et al.*, 2019) and the urgent need for effective solutions for waste management (Kataya *et al.*, 2023).

In the first years of the period analyzed (2006-2010), the number of publications was low, with only 8 works, reflecting an emerging interest in the topic. This preliminary period corresponds to the first initiatives and discoveries about coconut's potential in biomass and energy applications, suggesting that the field was still being explored. The absence of publications before 2006 may indicate that the field was in its preliminary stages or that there was a gap in the documentation and indexing of previous research. Since 2006, there has been a significant increase in research output, which suggests an important milestone in awareness or discoveries that have boosted academic and scientific interest. Between 2011 and 2016, there was a gradual increase in publications, of 28 journals, respectively, indicating a period of moderate growth. During this period, research began to gain greater attention, due to initial advances and the growing search for alternative energy sources and sustainability. This gradual growth reflects a more structured development of the field. As of 2017, scientific production has accelerated significantly, culminating in a peak around 2022, showing a total of 146 publications and 47 in 2022 alone. This sharp increase can be attributed to several factors, including increased funding for sustainable research, technological advances, and a growing global concern about waste management and renewable energy sources and the rate of activity and innovation.

Interestingly, the graph shows a decline in scientific production in the most recent years (2023-2024), with a reduction of 34.69% from the growth rate of previous years. This decline may indicate a temporary saturation in publications, suggesting that major discoveries have already been documented, or it may be the result of recent challenges, such as changes in funding priorities or the reorientation of research efforts toward new, emerging areas. This recent decline suggests that the field may be going through a phase of adjustment and reevaluation, which is important for identifying new research and application opportunities. It is important to highlight that, by the end of the second half of 2024, the number of publications may exceed 2023, reinforcing the idea of global advances in the area. The exponential growth of this area over the last 18 years reflects the growing global interest in the sustainable reuse of green coconut biomass. This field of study is rapidly evolving, quickly meeting global needs and incorporating technological advances efficiently.

3.3.2 Co-authorship Analysis between Institutions and Authors

Table 3.1 presents the ranking of the top 10 affiliations, research areas, countries, journals, and authors based on the number of publications on the potential of coconut biomass. The first section shows the ranking of the most prominent areas of the entire database analyzed. First, the area of "Energy Fuels " stands out, with 112 publications, corresponding to 42% of the total. This data highlights the significant impact of coconut biomass on the production of energy fuels, influencing sectors such as industry, public services, and, especially, the transportation sector, which over the years has consolidated itself as the largest consumer of fuels globally (Andrade *et al.*, 2024a; Kabir Ahmad *et al.*, 2022a; Liu, Zhengang; Han, 2015).

In second place is the area of " Engineering ", with 89 publications, representing 33.5% of the total. This number highlights the importance of engineering processes and techniques in the conversion of coconut biomass into high-value-added products (Natalia, 2021; Regista; Fuad; Dewi, 2021). Also in this section, other areas stand out, such as "Environmental Sciences Ecology " (59 publications), "Science Technology Other Topics " (58 publications), " Chemistry " and " Materials Science" (51 and 30 publications, respectively).

The second section of Table 3.1 describes the 10 most relevant journals on the topic, with the journal " Renewable Energy" leading with 14 publications, representing 5.2% of the entire database. This journal covers a wide range of topics related to clean energy sources, such as solar, wind, and bioenergy, among others. It is known for its in-depth analyses, market reports, and news on the latest innovations and developments in the renewable energy industry (Abdullah *et al.*, 2023; Energy, 2021; Griffin; Batten; Beer, 2013). Coconut biomass has demonstrated significant potential for the production of clean energy, which makes this topic relevant to " Renewable Energy". In second place is the journal " Energies ", with 11 publications, representing 4.1% of the total. It is worth noting that most of the 10 journals with the highest number of articles focus their publications on topics such as clean energy and energy transition, highlighting the importance of coconut biomass as a high-value product.

India, with 48 publications (18.1%), ranks first among the countries with the highest number of publications on coconut biomass. Studies indicate that the country has great potential to increase its energy production through biomass, which has led to increasing exploitation of coconut husks, seen as a significant byproduct of agroindustry. This trend points to an increase in energy generation capacity to 35,994.52 MWe by 2030-31 (Negi *et al.*, 2023). However,

progress has been slow, which explains the large number of publications focused on this area. China ranks second, with 45 publications (16.9%). Recognized as one of the largest producers of science, China has focused on transforming coconut husks into high-value-added products, aiming to reduce the environmental impacts caused by improper disposal (Duan *et al.*, 2023; Hu *et al.*, 2024; Liu, Zhengang; Balasubramanian, 2012; Zhang, Yamian *et al.*, 2021). Next, Brazil, Malaysia, and Indonesia stand out, with 36 (13.5%), 34 (12.8%) and 16 (6.0%) publications, respectively. It is also important to highlight the appearance of Nigeria in eighth position, with 13 publications, representing 4.9%, which demonstrates the growing concern of African countries with the use of this biomass, especially in regions with large coconut plantations.

The top three affiliations with the highest engagement in coconut biomass research were the Indian Institute of Technology System (IIT System), the National Institute of Technology (NIT System), and University Sains Malaysia, with 12 (4.5%), 7 (2.6%) and 7 (2.6%) publications, respectively. In the ranking of authors, although Mendoza-Castillo DI and Reynel -Ávila HE occupies the first two positions, with 4 publications each (representing 1.5% of the total), the difference for the other authors is minimal, as the following authors in the ranking have 3 publications each.

Table 3.1 - Ranking of the top 10 publication affiliations, Research areas, countries, journals, and authors based on the number of publications.

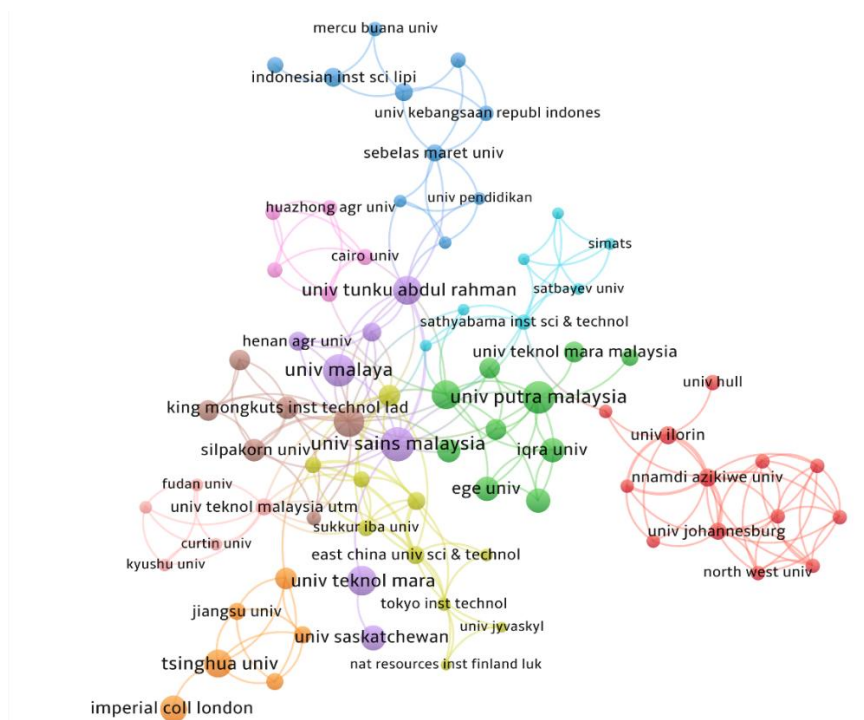
Ranking	Parameters	Number	% ^a
<i>Research areas</i>			
1st	Energy Fuels	112	42.2%
2nd	Engineering	89	33.5%
3rd	Environmental Sciences Ecology	59	22.2%
4th	Science Technology Other Topics	58	21.8%
5th	Chemistry	51	19.2%
6th	Materials Science	30	11.3%
7th	Physics	17	6.4%
8th	Agriculture	13	4.9%
9th	Thermodynamics	13	4.9%
10th	Biotechnology Applied Microbiology	9	3.3%
<i>Journals</i>			
1st	Renewable Energy	14	5.2%
2nd	Energies	11	4.1%

3rd	Journal Of Cleaner Production	10	3.7%
4th	Biomass Conversion and Biorefinery	10	3.7%
5th	Waste And Biomass Valorization	9	3.39%
6th	Fuel	8	3.0%
7th	Industrial Crops and Products	6	2.2%
8th	Renewable Sustainable Energy Reviews	6	2.2%
9th	Energy	5	1.8%
10th	Energy Fuels	5	1.8%
<i>Countries</i>			
1st	India	48	18.1%
2nd	Peoples R China	45	16.9%
3rd	Brazil	36	13.5%
4th	Malaysia	34	12.8%
5th	Indonesia	16	6.0%
6th	Thailand	15	5.6%
7th	England	14	5.2%
8th	Nigeria	13	4.9%
9th	USA	13	4.9%
10th	Mexico	10	3.7%
<i>Affiliations</i>			
1st	Indian Institute of Technology System Iit System	12	4.5%
2nd	National Institute of Technology Nit System	7	2.6%
3rd	Universiti Sains Malaysia	7	2.6%
4th	Universiti Teknologi Malaysia	7	2.6%
5th	Universiti Teknologi Petronas	7	2.6%
6th	Universidade Federal Do Espirito Santo	6	2.2%
7th	Anna University	5	1.8%
8th	Banaras Hindu University Bhu	5	1.8%
9th	Centre National De La Recherche Scientifique Cnrs	5	1.8%
10th	Council Of Scientific Industrial Research Csir India	5	1.8%
<i>Authors</i>			
1st	Mendoza-castillo DI	4	1,5%
2nd	Reynel-Ávila HE	4	1,5%
3rd	Adeniyi AG	3	1,1%
4th	Axaopoulos PJ	3	1,1%
5th	Bonilla-petriciolet A	3	1,1%
6th	Bot BV	3	1,1%
7th	Destyorini F	3	1,1%
8th	Ding L	3	1,1%
9th	Emenike CE	3	1,1%

Source: Author (2024).

Figure 3.2 shows the consortium of institutions generated by VOSviewer ©. From this figure, it is possible to identify five main networks, highlighted in purple, green, red, brown, and blue, in addition to secondary networks represented by orange, light orange, light blue, pink, and light green. It is important to highlight that, in both the purple and green networks, the two universities with the largest number of connections are from Malaysia: University Sains Malaysia and the University Putra Malaysia.

Figure 3.2 - Network analysis of the most prominent institutions.



Source: Author (2024).

In addition, we note that universities such as Mercu University Buana, the Indonesian Institute of Sciences (LIPI), and Sebelas Maret University form a significant cluster, suggesting a strong regional collaboration network. The presence of institutions from different continents, such as Cairo University in Egypt, Huazhong University in China, and Curtin University in Australia, also highlights the global nature of these collaborations. Analyzing these networks can provide valuable insights into the dynamics of international research, allowing the identification of centers of excellence and potential gaps in scientific collaboration.

Table 3.2 - Ranking of the 10 most prominent institutions in coconut biomass research.

Ranking	Institution	Country	Number of Publications
1st	Univ Teknol Petronas	Malaysia	22
2nd	Universidade Khon Kaen	Thailand	15
3rd	Univ Putra Malásia	Malaysia	14
4th	Univ Fed Espírito Santo	Brazil	13
5th	Univ Ilorin	Nigeria	12
6th	Univ Malaya	Malaysia	12
7th	Univ Teknol Mara	Malaysia	12
8th	Univ Sains Malásia	Malaysia	11
9th	Univ Fed Rio Grande Do Norte	Brazil	10
10th	University of Sergipe	Brazil	10

Source: Author (2024).

The Table 3. presents the ranking of the 10 most prominent institutions in coconut biomass research. In first place is the "University Teknologi Petronas", from Malaysia, with 22 publications, highlighting the country's significant influence in the development of studies on this biomass. Research carried out in this region indicates that coconut biomass can be converted into charcoal and synthesis gas (syngas) through the processes of pyrolysis and gasification, using different temperatures. In second place is the " (Ahmad *et al.*, 2023) Khon University Kaen", from Thailand, with 15 publications. It is important to highlight that half of the most prominent institutions listed in the ranking are from Malaysia, totaling 71 publications (more than 50% of the total). Brazil also stands out in the ranking, with the presence of the universities "The Federal University of Espírito Santo", "The Federal University of Rio Grande do Norte" and "The Federal University of Sergipe", which together total 33 publications.

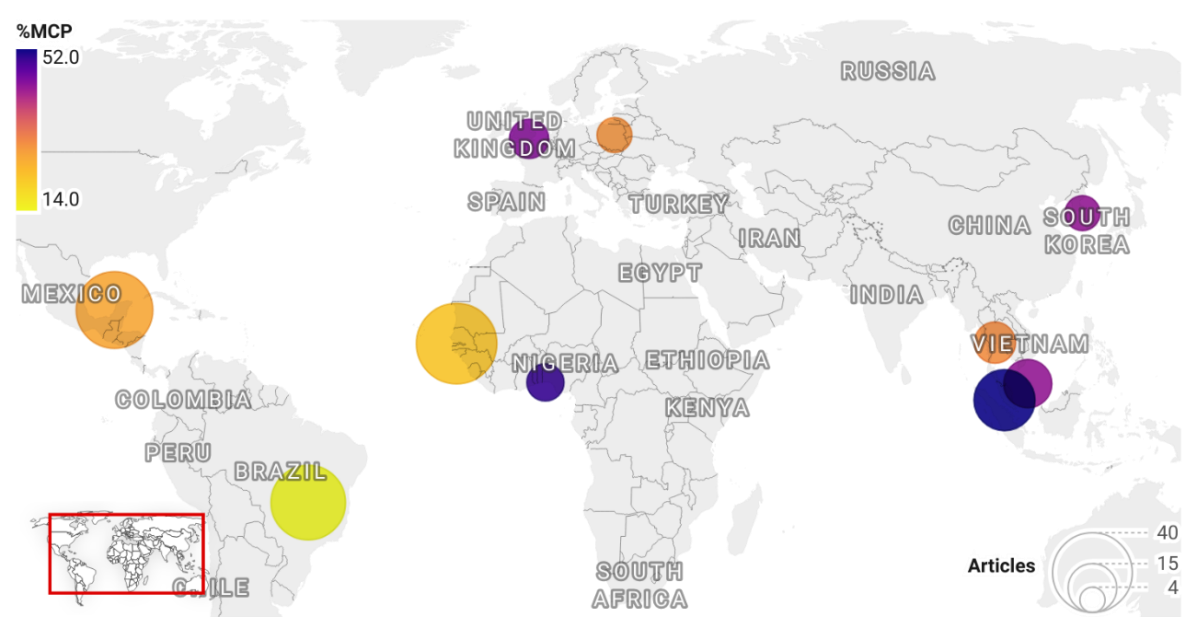
3.3.3 Cooperation Network between Countries/Regions

The Figure 3.3 illustrates the geocoding of the 10 most relevant countries based on the corresponding author. In this figure, each country is represented by a colored circle, the size of which indicates the number of articles published by authors from that country.

The color of the circle ranges from yellow to blue, reflecting the "Multi-Country Publications" (MCP), which, in turn, indicates that the scientific articles were co-authored by researchers from different countries (Moshobane; Khoza; Niassy, 2022). It can be seen that

countries such as Brazil, Mexico, and Egypt have larger circles, which represent a high number of publications. On the other hand, countries such as Malaysia, China, England, and Nigeria have circles of relatively medium size, but in colors ranging from purple to blue, standing out for having a higher MCP index. This type of publication is indicative of robust international collaboration, evidencing the integration and cooperation between scientific communities around the world.

Figure 3.3 - Map of the 10 most relevant countries by corresponding author.

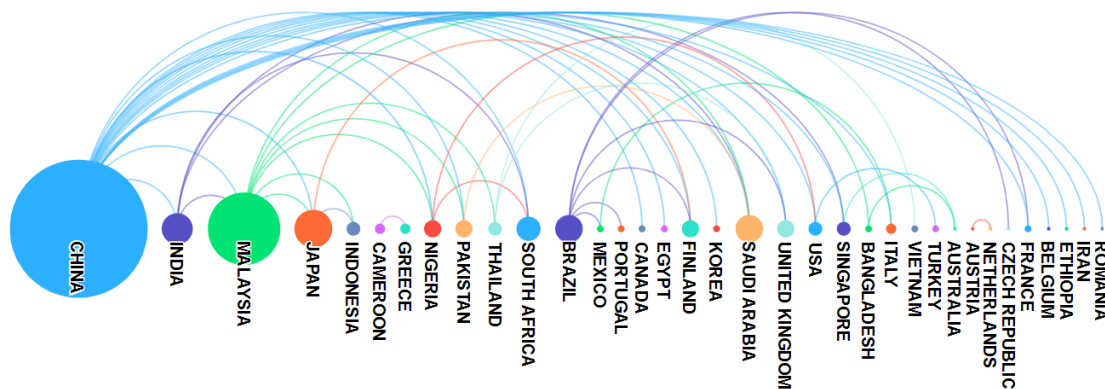


MCP = Multi Country publications.

Source: Author (2024).

Figure 3.4 presents a visual representation of the collaborations between the countries analyzed in the database. China stands out as the country with the largest number of collaborations, exerting significant influence over other regions, followed by Malaysia and India. It is worth noting that the three countries with the largest number of collaborations belong to the same continent, which facilitates cooperation between them due to geographical proximity.

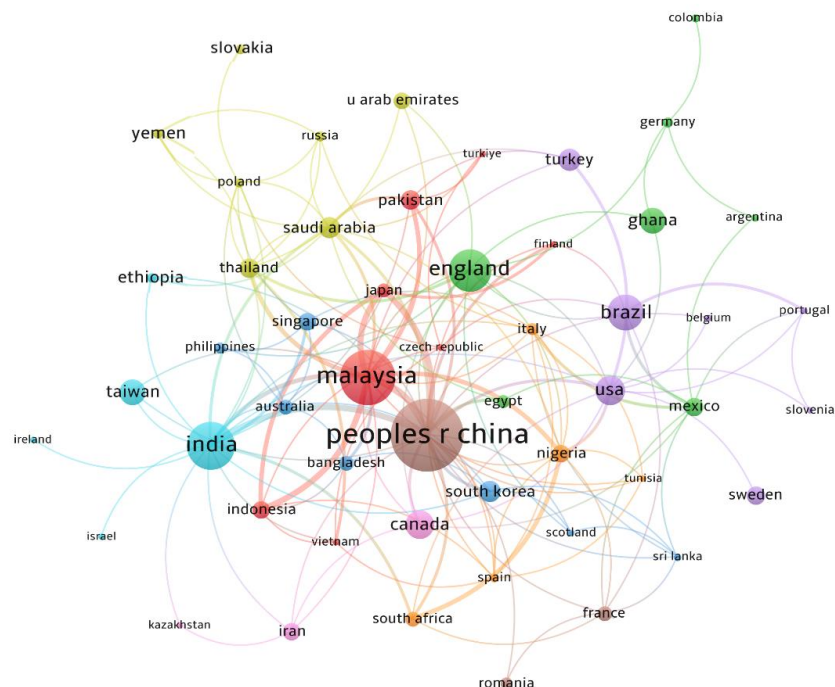
Figure 3.4 - Network of countries in collaboration.



Source: Author (2024).

This phenomenon is also observed in Figure 3.5, which highlights other collaboration networks, such as the green network, dominated by England, and the purple network, with emphasis on Brazil. The presence of countries such as Ghana, Nigeria, and Ethiopia in Figure 3.6 demonstrates that it is not only Europe and Asia that are focused on the high energy value of coconut biomass. The African continent has also made significant advances in its collaborations, influencing the global research scenario.

Figure 3.5 - Analysis of collaboration networks between influential countries.



Source: Author (2024).

3.3.4 Keywords with the highest search index

Keyword analysis is of great importance in research, as it helps us identify emerging information, understand the stages of research, highlight new methodologies under development, and identify prominent terms in the global scientific community. (Dari *et al.*, 2024a; de Castro Bizerra; Leandro Fernandes Melo; *et al.*, 2024; de Castro Bizerra; Sales; *et al.*, 2024b). In this study, a database of keywords was analyzed, from which those with the greatest relevance were extracted. Table 3.3 presents the 20 most relevant keywords related to the topic in question. These keywords are grouped according to their frequency of occurrence, with emphasis on terms such as Biomass (64 occurrences), Waste (43 occurrences), Energy (34 occurrences), Pyrolysis (31 occurrences), and Performance (30 occurrences), which together represent approximately 44.3% of the total occurrences of the 20 keywords with the highest search index.

Sankey diagram (Figure 3.6) that illustrates the connection and flow of information between different groups. The left column lists the institutions, which include universities and research centers from various countries around the world that have conducted studies on the topics represented by the keywords. The central column displays the keywords that are the focus of the research and reflect the main topics of scientific interest. The right column lists the countries associated with the research, indicating where these studies are most active or relevant.

The connections in the diagram link institutions to keywords, and in turn, link these keywords to countries, revealing which institutions are researching certain topics and in which regions this research is taking place or is most relevant. The figure also highlights how different institutions collaborate or focus their research on specific topics and how these keywords are distributed across different countries, reflecting the geographic focus of scientific research.

This type of visualization is useful for understanding the global distribution of research and identifying which themes are prominent in different regions. For example, the Federal University of Ceará and the Federal University of Sergipe stand out in themes such as “Pyrolysis” and “Biochar”, showing a strong Brazilian contribution to studies on pyrolysis and biochar. Similarly, the University of Malaya and the University of Sains Malaysia are linked to these same themes, consolidating Malaysia as an important hub for these technologies.

The keyword “Biomass” is strongly connected to institutions in Thailand and other Asian universities, evidencing a great interest in these regions for renewable energy sources.

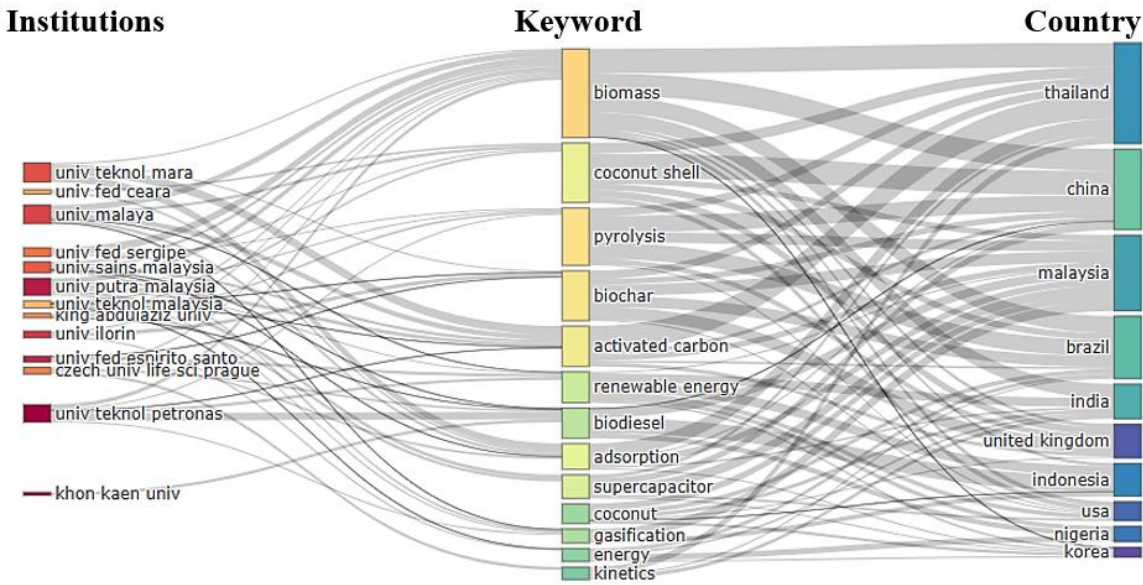
“Activated Carbon” stands out in Brazilian research, especially at the Federal University of Esp rito Santo and other institutions, underlining the relevance of this technology in Brazil.

Table 3.3 - 20 most relevant keywords.

Rank	Keywords	Occurrences	Rank	Keyword	Occurrences
1st	Biomass	64	11st	Removal	19
2nd	Waste	43	12nd	Optimization	18
3rd	Energy	34	13rd	Carbon	16
4th	Pyrolysis	31	14th	Temperature	16
5th	Performance	30	15th	Biochar	15
6th	Coconut shell	24	16th	Conversion	15
7th	Activated carbon	21	17th	Kinetics	14
8th	Adsorption	20	18th	Coconut-shell	13
9th	Cellulose	19	19th	Gasification	13
10th	Lignocellulosic biomass	19	20th	Combustion	12

Source: Author (2024).

Figure 3.6 - Sankey Diagram: Connections between Institutions, Keywords and Countries in Biomass and Renewable Energy Research.



Source: Author (2024).

the *Journal of Analytical and Applied Pyrolysis*. The fourth and fifth articles were published in the same journal, *Renewable & Sustainable Energy Reviews*, with a total of 293 and 190 citations in 2018 and 2011, respectively.

Table 3.4 - The 10 most cited articles in the research area of hydrogen generated via biogas reforming.

Ranking	Title	Journal	Publication year	Total citations	Average citation per year	Ref.
1st	From coconut shell to porous graphene-like nanosheets for high-power supercapacitors.	Journal of Materials Chemistry A	2013	758	63,17	(Sun <i>et al.</i> , 2013)
2nd	Promising biomass-based activated carbons derived from willow catkins for high performance supercapacitors.	Electrochimica Acta	2015	367	36,7	(Wang, Kai <i>et al.</i> , 2015)
3rd	Fast pyrolysis of rice straw, sugarcane bagasse and coconut shell in an induction-heating reactor.	Journal of Analytical and Applied Pyrolysis	2006	327	17,21	(Tsai; Lee; Chang, 2006)
4th	Microwave assisted preparation of activated carbon from biomass: A review.	Renewable & Sustainable Energy Reviews	2018	293	41,86	(Ao <i>et al.</i> , 2018)
5th	A comprehensive review of biomass resources and biofuels potential in Ghana. Equilibrium, kinetic, and thermodynamic studies of lead ion and zinc ion adsorption from aqueous solution onto activated carbon prepared from palm oil mill effluent.	Renewable & Sustainable Energy Reviews	2011	190	13,57	(Duku; Gu; Hagan, 2011)
6th	Pyrolysis characteristics and global kinetics of coconut and cashew nut shells.	Journal of Cleaner Production	2017	187	23,38	(Adebisi; Chowdhury; Alaba, 2017)
7th	Synthesis of biomass as heterogeneous catalyst for application in biodiesel production: State of the art and fundamental review.	Fuel Processing Technology	2006	184	9,68	(Tsamba; Yang; Blasiak, 2006)
8th	The past, present and future of carbon black as a rubber reinforcing filler - A review.	Renewable & Sustainable Energy Reviews	2018	182	26	(Tang <i>et al.</i> , 2018)
9th		Journal of Cleaner Production	2020	181	36,2	(Fan; Fowler; Zhao, 2020)

10th	Life Cycle Assessment of activated carbon production from coconut shells.	Journal of Cleaner Production	2016	166	18,44	(Arena; Lee; Clift, 2016)
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Source: Author (2024).

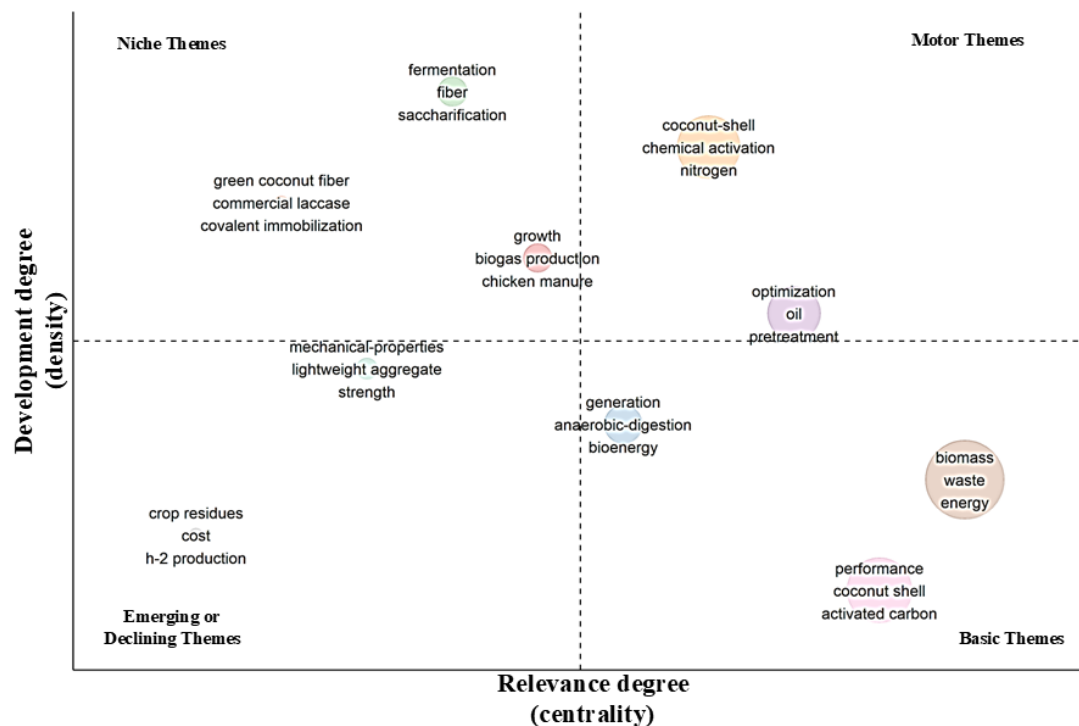
It is interesting to note that although the fourth article has a lower total number of citations (293), it has an average of 41.86 citations per year, the second highest on the list, indicating that it has had a significant impact in a short period. This value is surpassed only by the first article, which has an average of 63.17 citations per year since its publication in 2013. This suggests that the first article, despite having a higher total number of citations, also stands out for having a high number of citations concentrated in a relatively short period of years, while the third article, published in 2006, has an average of only 17.21 citations per year, reflecting an impact distributed over a longer period.

3.3.6 Main Areas

Green coconut offers great potential for the development of sustainable solutions. Given that it is a fruit widely consumed worldwide and generates much waste, reusing these materials opens up a range of ways for reuse (Kabir Ahmad *et al.*, 2022d). Among them is the use of green coconut fibers for various purposes, especially for academic and industrial purposes, including support materials for the synthesis of catalysts (Anil *et al.*, 2024; Yousefian *et al.*, 2023), and the manufacture of biochar (Ighalo *et al.*, 2023c; Suman; Gautam, 2017) as adsorbents for the removal of toxic contaminants from wastewater (Tyagi; Anand, 2024). This approach emerges as a way to reduce the carbon footprint, as well as to reuse a material that would otherwise be discarded in the environment and transform it into something new, adding new value to it (Belluati *et al.*, 2024b).

Figure 3.8 presents the relationship between centrality and density of the most recurrent keywords in studies on the potential of coconut biomass. The keywords are visualized as spheres, whose size indicates the relative importance of each term in the set of documents analyzed. This diagram facilitates the visualization of the distribution of keywords, highlighting both centrality and density and helping to identify the areas of greatest interest and relevance in the field. The upper left quadrant contains "Niche themes", such as fermentation saccharification of fibers, covalent immobilization of commercial laccase from green coconut fiber, and growth of biogas production from chicken manure.

Figure 3.8 - Map of the main clusters correlating impact and centrality of research topics on potential uses of coconut biomass.



Source: Author (2024).

The first two topics are not very central and have relatively low centrality, indicating specific importance within their areas, but not essential for the field as a whole, while the growth of biogas production from chicken manure is close to centrality. The lower left quadrant has "Emerging or declining themes", covering topics such as mechanical properties slightly aggregated strength, and crop residues cost H₂ production. With low centrality and density, they suggest potential emergence or decline, gaining or losing relevance within the main theme of green coconut biomass. In the lower right quadrant are the "Basic Themes", such as biomass residual energy, coconut shell activated carbon performance, and anaerobic digestion bioenergy generation, the latter a little more central to the main field.

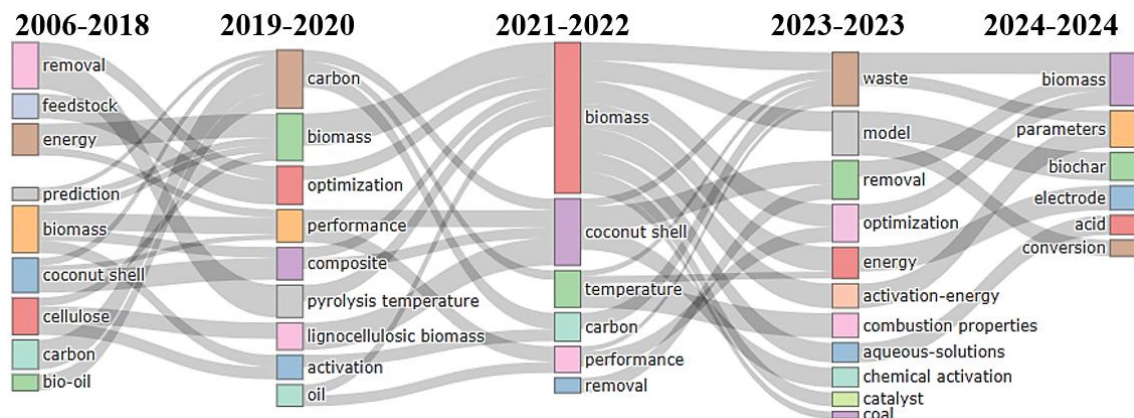
The other two topics are important but not central and highly relevant themes. In the upper right quadrant are the "Driving Themes", such as chemical activation nitrogen from coconut shells, which has high relevance but is not a central theme, while the optimization oil pretreatment, which is one of the most centralized topics and with much work in the area, is a theme with slightly less relevance than the previous one, but a robust theme.

The evolution diagram of the main keywords (figure 3.9) provides a chronological and segmented view of the evolution of research topics in the area over four periods: 2006-2018, 2019-2020, 2021-2022, 2023-2023, and 2024-2024. In the first period, the predominant keywords include "removal", "feedstock", "energy", "prediction", "biomass", "coconut shell", "cellulose", "carbon" and "bio-oil". This initial period focuses on the fundamentals of the production of studies on the properties and applications in which green coconut can be used. Such studies accompanied the development of new biomass conversion techniques. Currently, several techniques are used in biomass conversion such as pyrolysis (Kumar Mishra; Singh; Acharya, 2024), gasification (Verma *et al.*, 2023), torrefaction (Mamvura; Danha, 2020), hydrothermal carbonization (Cheng *et al.*, 2022), and microwave heating (Begum *et al.*, 2024).

Between 2019 and 2020, there was a shift in research focus, with a greater focus on “carbon” and “biomass” involving green coconut. In addition, there is a focus on optimizing the processes for converting coconut and its by-products into bioenergy, as shown by the citation for “pyrolysis temperature”. In the period 2021 to 2022, the main keywords predominantly include “biomass” and “coconut shell”, as well as “temperature”, “performance”, “removal” and “carbon”. This reflects an interest in improving the processes of this biomass, with an additional focus on research techniques and sustainability.

In 2023-2023, there was a greater diversification and specialization of keywords, with terms such as "model", "waste" and "energy" showing how the focus on sustainability remained strong. "Activation energy", "combustion properties", "chemical activation" and "catalyst" show how chemical processes have become important within research on how to transform coconut waste into by-products through various processes. "charcoal" exemplifies that there have been studies on the transformation potential that coconut can have in being transformed into biochar.

Figure 3.9 - Evolution diagram of the main keywords on potential uses of green coconut biomass organized chronologically to illustrate the evolution of research topics in the area over time (2006 to 2014).



Source: Author (2024).

In the following period, 2024-2024, it is observed that the keywords, again with the broad term of "biomass" appear with a good focus on the reuse of green coconut biomass. "Parameters", "electrode" and "acid" show how there is still a focus on the best way to convert coconut into renewable energy. Just like the following word "conversion", exemplifies the same. "Biochar" shows that there were fruits of research involving the green production of biochar using coconut.

The dynamic evolution and expansion of research in this area over the years show a significant increase in the occurrence of keywords due to the diversification and specialization of the topics of new methods and research to develop processes using coconut. Highlighting the evolution of mainly chemical processes in this area. This shows the complexity and importance of this area of study, so that it can drive the global use of sustainable energy products and a focus on evolution towards a low-carbon economy (Mujtaba *et al.*, 2023).

3.4 CONCLUSION

This study demonstrated the growing importance of using green coconut biomass, especially its husk, as a sustainable alternative for the production of energy and value-added materials. The bibliometric analysis revealed a significant increase in the number of publications related to the topic since 2017, reflecting an intensification of research focused on the development of innovative technologies for the use of lignocellulosic biomass. This growth is driven by the global need for energy transition and the search for more efficient solutions for the management of agro-industrial waste.

Green coconut husk stands out for its composition rich in lignin, cellulose and hemicellulose, which makes it an attractive raw material for thermochemical processes, such as pyrolysis and gasification, in addition to the production of biochar and activated carbon. Recent research also points to its application as an adsorbent in wastewater treatment and in the synthesis of heterogeneous catalysts, expanding the possibilities for reusing this waste in high value-added industrial processes.

The rapid growth in publications indicates that the exploration of green coconut biomass is consolidating itself as a key area of research, with significant contributions coming from countries such as India, China and Brazil. These countries have led technological development initiatives, especially in the conversion of biomass into clean fuels and functional materials, taking advantage of the potential of one of the most abundant and underutilized raw materials globally.

In view of this expansion scenario, it was possible to observe that it is essential to investigate new technological approaches, such as the optimization of thermochemical processes and the integration of biomass conversion techniques with carbon capture and storage (CCS) systems. In addition, the strengthening of international collaborations will be essential to consolidate the role of green coconut husk in the global energy matrix, contributing significantly to the mitigation of greenhouse gas emissions and to the circular economy.

4 CATALYST SUPPORTED ON COCONUT SHELL BIOMASS FOR HYDROGEN PRODUCTION: SYNTHESIS AND CHARACTERIZATION

Abstract

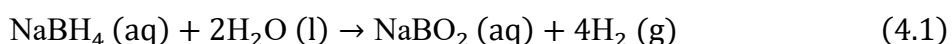
Green coconut fiber (GCF)-supported catalysts were synthesized and evaluated for hydrogen production via sodium borohydride (NaBH_4) hydrolysis. The phosphoric acid-treated (GCF-C) study showed significant improvements in catalytic efficiency due to its increased porosity and surface area. Characterization techniques, including thermogravimetric analysis (TGA), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM), confirmed the structural modifications. Using the Taguchi L16 method, the optimum evidence conditions were identified as 300 mg GCF-C, 750 mg NaBH_4 , at 50 °C and 120 rpm. A kinetic analysis revealed a linear hydrogen production rate with an activation energy (E_a) of 28.95 $\text{kJ}\cdot\text{mol}^{-1}$, demonstrating the efficiency of GCF-C in reducing occurrence barriers. Despite the promise, challenges in gas quantification due to atmospheric contamination were identified, indicating the need for future experiments with inert gas purging. GCF-C also showed potential for reuse, offering an environmentally sustainable and cost-effective solution for hydrogen production.

Keywords: Hydrogen Production, Sodium Borohydride Hydrolysis, Coconut Fiber Catalyst, Kinetic Study, Green Biomass Catalysts

4.1 INTRODUCTION

The increasing demand for energy and the decline in conventional fossil fuels, together with concerns related to global warming, are driving the research community to seek green, renewable and sustainable energy sources (Balbay; Selvitepe; Saka, 2021). Among the various energy resources, hydrogen (H₂) is considered an ideal and sustainable energy vector due to its high energy density (Ekinici *et al.*, 2024; Liu, Yongjing *et al.*, 2024b). In addition, it has the potential to reduce the carbon footprint in the energy, transportation and industrial sectors (Bhandari; Adhikari, 2024).

Hydrogen can be produced through several methods, such as methane reforming, water electrolysis, biomass gasification, and methanolysis or hydrolysis of chemical hydrides (Kaya, 2020b). Metal hydrides, formed by elements such as Mg, Be and Al, offer an efficient solution for hydrogen storage, allowing its absorption and reversible release (Yang *et al.*, 2024) with high safety and density (Kamran; Turzyński, 2024). Hydride derivatives, such as NaBH₄, stand out for their superior characteristics, including high hydrogen content (10.8% by weight), non-flammability, non-toxicity, stability and the ability to recycle by-products (Ekinici *et al.*, 2024; Liu, Yongjing *et al.*, 2024b; Wang, Hehui *et al.*, 2024). When completely hydrolyzed, 4 moles of Hydrogen can be released for every 1 mole of NaBH₄ (Lin *et al.*, 2021) (Eq. (4.1)):



Furthermore, the theoretical stoichiometry of the reaction predicts that for every 1 gram of fully hydrolyzed NaBH₄, the average volume of hydrogen generated is approximately 2.37 liters of hydrogen, under standard temperature and pressure (STP) conditions (Abraham *et al.*, 2024). However, hydrogen production via hydrolysis of metal hydrides presents significant kinetic challenges under ambient conditions, in the absence of a suitable catalyst (Kadrekari; Patel; Arya, 2020), which plays an essential role in the hydrolysis of NaBH₄.

High-cost noble metals were widely used for this purpose, but due to the high cost and limited availability of these metals, interest in alternative metal catalysts, such as nickel (Ni), cobalt (Co) and iron (Fe), has increased significantly (Ekinici *et al.*, 2024). Recently, HUANG (Huang, Wenkai; Xu; Liu, 2021) in a study designed and synthesized M-Ru/C (Fe-Ru/C, Co-Ru/C, Ni-Ru/C and Cu-Ru/C) bimetallic nanocomposites to improve the production of H₂ from the hydrolysis of NaBH₄. The results showed that the combination Ru/C with Co or Ni performed best, with Co-Ru/C exhibiting the highest catalytic activity.

However, in recent years, there has been growing interest in studies focused on catalysts supported on porous carbon, especially prepared from plant biomass, due to their larger surface area, stability, durability, chemical inertness, and easy preparation (Bu *et al.*, 2021). As a more sustainable option, research is being conducted to evaluate the chemical doping of porous carbon with heteroatoms, including phosphorus (P), sulfur (S), and nitrogen (N) (Saka; Balbay, 2021).

Several agricultural residues, such as walnut shells (Lionetti *et al.*, 2024), horse chestnut (Turkyilmaz *et al.*, 2024), peanuts (Faraji; Saidi, 2023) and green coconut (Gratuito *et al.*, 2008) can be considered suitable raw materials for the production of porous carbon. A substantial amount of biomass waste is generated daily, including agricultural residues, by-products, lignocellulosic and woody biomass (Sekhon; Kaur; Park, 2021). A notable example in Brazil is the fibers and shells of the green coconut (*Cocos nucifera L.*). Annually, millions of tons of this waste are discarded in landfills or open-air dumps (Andrade *et al.*, 2024b). However, waste such as this has stood out as a renewable source for the synthesis of sustainable catalysts, valued for their availability, biodegradability and positive environmental impact (Belluati *et al.*, 2024a).

Based on these considerations, we aimed to synthesize metal-free catalysts using green coconut shell as raw material for the production of biomass-supported catalysts. These metal-free catalysts were used for the first time in the production of H₂ from NaBH₄. The obtained samples were analyzed using XRD, SEM, FTIR and XRF elemental analysis.

4.2 METHODOLOGY

4.2.1 Materials

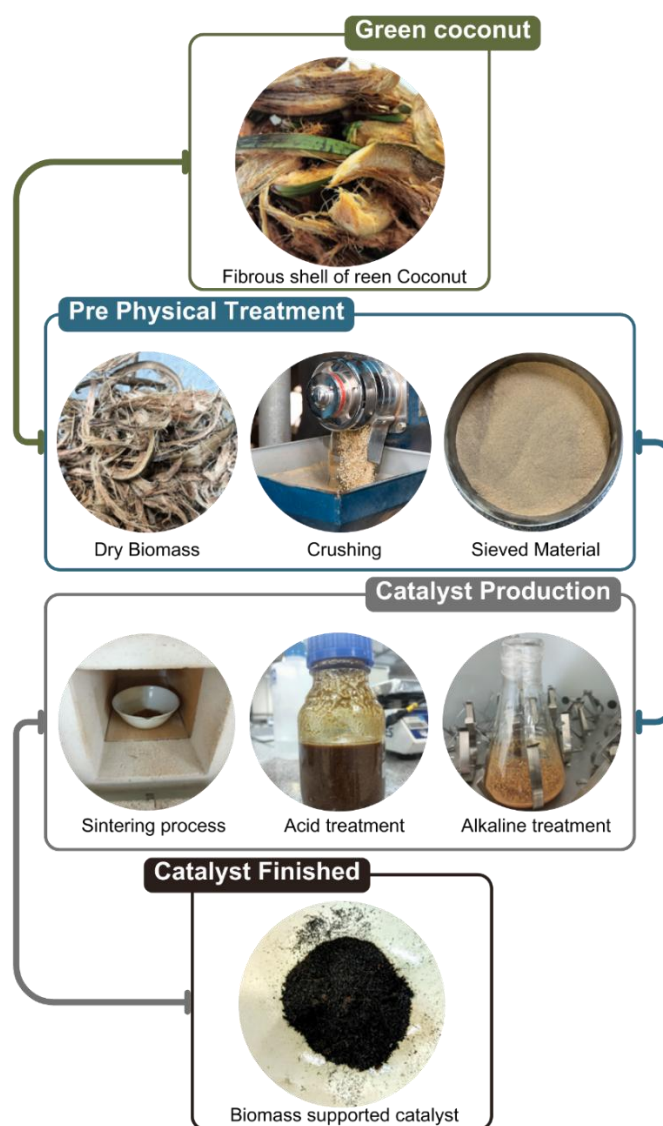
Green Coconut Fiber (GCF) was collected from Sabiaguaba Beach, Fortaleza, Ceará. The reagents used, such as orthophosphoric acid P.A (H₃PO₄) (85%) and sodium borohydride (NaBH₄) (98%), were purchased from Sigma-Aldrich (St. Louis, MO, USA). Sodium hydroxide (NaOH) (98%) was purchased from Dynamical (São Paulo, Brazil). Ultrapure water (Type I) was produced by a Purelab Option Q Helga water purification system. All other chemical reagents used were of analytical grade.

4.2.2 Methods

4.2.3 Preparation of Biocatalyst

After collection, the Green Coconut Fiber sample underwent three washing cycles using ultrapure water, followed by the drying process in an oven for 24 hours at 100°C. It was preserved from air humidity in a glass desiccator for the period prior to the pre-treatment stage, following the method adopted by (Robles *et al.*, 2022), with specific modifications.

Figure 4.1 - Flowchart of the catalyst production process from green coconut.



Source: Author (2024).

The fibrous residue from Green Coconut was subjected to mechanical pre-treatment (Figure 4.1). This process involved grinding the material in a knife mill, followed by passing it through a set of sieves to separate granules, with an average size range of 0.053 - 0.5 mm. The granules then underwent alkaline treatment with a hydrogen peroxide solution (4.3% v/v), prepared and adjusted to a basic medium (pH 11.5), using 6M NaOH. This stage will be carried out on an orbital shaker, for a period of 6 hours and a speed of 200 rpm.

Soon after, the solid material was recovered through simple filtration. It underwent three washing cycles using distilled water, followed by the drying process in an oven, which will last for a period of 24 hours at 60°C.

For the preparation of the catalyst, the methodology was conducted in accordance with the guidelines presented in the literature, with specific modifications (Fangaj; Ceyhan, 2020a; Kaya, 2020c; Saka; Kaya; Bekiroğullari, 2020) In a 125 ml Erlenmeyer flask, 5 g of the pretreated material was added, followed by 50 ml of a 15% H₃PO₄ solution. The solution was then stirred in an orbital shaker for 10 minutes at a speed of 300 rpm. Subsequently, the mixture was allowed to rest for 24 hours to allow impregnation.

Immediately after, the solid material was recovered through simple filtration. It underwent three washing cycles using distilled water, followed by the drying process in an oven, which lasted for a period of 24 hours at 80°C. Once the drying process was completed, the solid material was transferred to a ceramic crucible. It was then sintered at 200°C in a muffle furnace for a period of 45 minutes.

The manufactured catalyst was subjected to three washing cycles using distilled water, followed by the drying process in an oven for a period of 6 hours at 100°C. Finally, the produced catalyst was stored in a closed environment, ensuring its preservation until use.

4.2.4 BIOMASS AND BIOCATALYST CHARACTERIZATION

The characterization of the biomass was carried out with the aim of evaluating the physicochemical properties of the pure and developed coconut. The material was designated as GCF (Green Coconut Fiber) and classified into two forms: GCF(P), referring to the pure coconut, and GCF(C), corresponding to those obtained from the green coconut shell.

4.2.5 Thermogravimetric Analysis (TGA)

To evaluate the thermal manipulation of pure and treated biomass, thermogravimetric analysis was performed. Thermogravimetric analyses (TGA) were conducted using a

MettlerToledo TGA/SDTA851e equipment. Samples with a mass of approximately 10 mg were used and subjected to a continuous flow of nitrogen (N₂) at a rate of 50 mL/min. Heating was performed at a constant rate of 10 °C/min, covering a temperature range from 30 °C to 800 °C (Melo *et al.*, 2024a).

4.2.6 X-ray Diffraction (XRD)

XRD was performed on an X'Pert MPD powder X-ray diffractometer (Panalytical), with a scanning range of $2\theta = 20 - 80^\circ$. A CuK α tube (1.54059 Å) will be used for the samples, operating at 40 kV and 30 mA, and scanning range of $20^\circ - 100^\circ$. Diffraction patterns will be obtained using Brentano Bragg Geometry in continuous mode with a speed of $0.5^\circ/\text{min}$ and a step size of 0.02° (2θ). The Rietveld refinement structure will be used to interpret and analyze the diffraction data using the Maud® program. The crystal size of each sample will be calculated using the Scherrer equation (Jari *et al.*, 2024).

4.2.7 Scanning Electron Microscopy (SEM)

The Scanning Electron Microscopy (SEM) test methodology was performed using the QUANTA 450 FEG microscope. The samples were prepared by fixing them on carbon tape and metallized with silver using the Quorum QT150ES metallization equipment. During the analysis, an electron beam with a voltage of 20 kV was applied, allowing detailed images of the sample surface to be obtained (de Menezes *et al.*, 2024).

4.2.8 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier transform infrared spectroscopy (FTIR) analysis was performed to investigate the activation of the support. The analyses were performed on a Perkin Elmer FT-IR/NIR FRONTIER Fourier transform spectrophotometer. An attenuated total reflectance (ATR) accessory with zinc selenide (ZnSe) crystal was used for the measurements. The analyses were performed with a resolution of 4 cm^{-1} , with the average of 32 scans, covering the range of 4000 to 500 cm^{-1} (Priya; Kennedy; Anand, 2024).

4.2.9 X-ray Fluorescence (XRF)

The methodology employed for X-ray Fluorescence (XRF) analysis involved the use of the SHIMADZU EDX-7000 equipment, operating in an air atmosphere with a 10 mm collimator. The analyzed samples were subjected to two voltage and current settings: 50 kV and 88 μA for the energy range of 0 to 40 keV, and 15 kV and 381 μA for the range of 0 to 20 keV. Data acquisition was performed in 30 seconds for each setting, allowing the qualitative

and quantitative identification of the elements present in the sample, including K, Ca, S, Fe, and others (Melo *et al.*, 2024a).

4.2.10 Characterization of the Generated Gas

The gas analysis was performed qualitatively, with the aim of verifying the presence of hydrogen in the gas generated. Due to the impossibility of adequately purging and cleaning the atmospheric gas present in the headspace of the reaction system, it was not possible to accurately quantify the gas components, focusing only on identifying the presence of hydrogen.

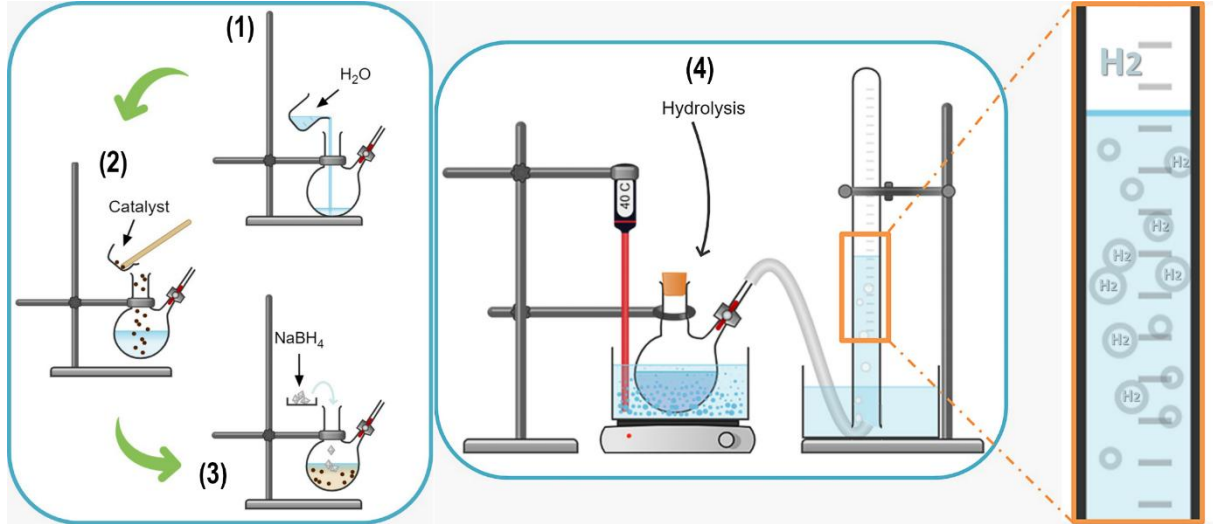
4.2.11 Gas Chromatography (GC)

For qualitative characterization, the samples were analyzed in an advanced Agilent 990 Micro Gas Chromatography (MGC) system, equipped with a high-precision Thermal Conductivity Detector (TCD). The system had two independent column channels, optimized for different separations: a 20-meter Molesieve column and a 10-meter CP-PoraPLOT U. The entire process was conducted using argon as the carrier gas.

4.2.12 Experimental setup

The quantification of the gas generated was performed using a hermetically sealed reaction system, as shown in Figure 4.2. The system consists of a reactor submerged in a heated water bath, coupled to a volumetric water displacement measuring device. The gas released from the reactor is conducted through a tube submerged in a water measuring column, where the displacement of the liquid is used to quantify the volume of gas produced. The displacement of the water in the column is proportional to the volume of gas generated, allowing a direct and accurate measurement (Hussein Hashem *et al.*, 2024).

Figure 4.2 - Scheme of volumetric measurement of gas by water displacement in a reaction system. Procedure: (1) Addition of distilled water, (2) Addition of the catalyst to the solution, (3) Introduction of NaBH₄ after the catalyst, and (4) Hydrolysis reaction for hydrogen production.



Source: Author (2024).

4.2.13 Determination of Hydrogen Generation Rate (HGR)

To evaluate the rate of hydrogen generation through the catalytic hydrolysis of NaBH₄, it was based on the measurement of the volume of hydrogen produced over time (Eq. (4.2)) (Xu, Fengyan *et al.*, 2024). Where the parameters (V), (T) and (M) represent the volume of hydrogen, total reaction time and mass of catalyst used, respectively.

$$HGR = \frac{V}{T \times M} \quad (4.2)$$

Hydrogen generation rate (HGR) is calculated by the formula, resulting in the rate of hydrogen production in milliliters per minute per gram (mLH₂.min⁻¹.g⁻¹)(Xu, Fengyan *et al.*, 2024).

4.2.14 Determination of Activation Energy (E_a)

To investigate the performance of the catalytic system in the hydrolysis of sodium borohydride (NaBH₄), the reaction rate at different temperatures was determined and the corresponding Arrhenius plot was constructed. The hydrogen evolution rate was measured in a temperature range of 20 to 60 °C, applying a heating ramp of 10 °C.min⁻¹. To evaluate the catalytic efficiency in the hydrolysis of the NaBH₄ solution, the apparent activation energy (E_a)

was calculated, which was determined by the Arrhenius equation (Eq. (4.3)) (Xu, Fengyan *et al.*, 2024) based on the kinetic curves of hydrogen generation in a variable range of reaction temperatures.

$$\ln k = \ln A - \frac{E_a}{RT} \quad (4.3)$$

Where A, T and R are respectively the pre-exponential factor, hydrolytic temperature and universal gas constant ($8.314 \text{ J mol}^{-1} \text{ K}^{-1}$) (Xu, Fengyan *et al.*, 2024). A lower value of E_a (activation energy) during hydrolysis indicates that the reaction occurs more easily, that is, it requires less energy to occur. In the context of a biomass-supported catalyst, this means that a lower value of E_a suggests that the catalyst is more active and effective in promoting the reaction, which is desirable to improve the performance of the catalytic process.

4.2.15 Statistical analysis of data

4.2.16 Taguchi experimental design (L16)

An experimental design based on the “Taguchi” statistical method with an orthogonal matrix of the L16 type was used, in which (“L” and “16” mean the Latin square and the number of experiments performed, respectively) to distribute five factors in four levels, aiming to optimize the volume of hydrogen generated. The five independent factors in the design are time (t), amount NaBH_4 (by mass in milligrams), catalyst (C), temperature (T) and agitation (Ag), while the dependent variable is the volume of H_2 generated. The levels of each factor are presented in table 4.1

Table 4.1 - Determination of experimental procedure levels and range of independent parameters.

Level	t (min)	NaBH_4 (mg)	Catalyst (mg)	T (°C)	Agitation (rpm)
Level 1 (L1)	10	100	50	30	120
Level 2 (L2)	20	250	100	40	130
Level 3 (L3)	30	500	150	50	140
Level 4 (L4)	40	750	300	60	150

Source: Author (2024).

Table 4.2 - Experimental design of the Taguchi method.

Exp	t (min)	NaBH ₄ (mg)	Catalyst (mg)	T (°C)	Ag (rpm)
1	10	100	50	30	120
2	10	250	100	40	130
3	10	500	150	50	140
4	10	750	300	60	150
5	20	100	100	50	150
6	20	250	50	60	140
7	20	500	300	30	130
8	20	750	150	40	120
9	30	100	150	60	130
10	30	250	300	50	120
11	30	500	50	40	150
12	30	750	100	30	140
13	40	100	300	40	140
14	40	250	150	30	150
15	40	500	100	60	120
16	40	750	50	50	130

Source: Author (2024).

Statistical analysis was performed using the Statistica software, applying the Taguchi experimental design. Table 4.2 shows the experimental design adopted. Since one of the main objectives of the research was to maximize the activity of the biocatalyst, the "bigger is better" criterion was used to evaluate the variables in each reaction. This approach aims to identify the optimal conditions that maximize catalytic efficiency, allowing the determination of the ideal operating point for the biocatalyst, as shown in Table 4.2.

4.2.17 Determination of Storage Potential

The storage potential test was performed using continuous hydrolysis cycles of sodium borohydride (NaBH₄) with the heterogeneous catalyst GCF (C). The experimental conditions were defined based on the optimum point determined after evaluating the results of the Taguchi method.

At the end of each hydrogen production cycle, the catalyst was recovered by vacuum filtration, followed by washing with distilled water to remove residues. The catalyst was then dried in an oven at 60 °C for 24 hours and stored at 4 °C for a period of 15 days.

After the storage period, the catalyst was reused in a new hydrolysis cycle, and the hydrogen production yield was monitored in each subsequent cycle, with the aim of evaluating the maintenance of catalytic efficiency over time.

4.3 RESULTS AND DISCUSSIONS

4.3.1 Characterization analysis

4.3.2 Thermogravimetric analysis (TGA and DTG)

The thermogravimetric analysis (TGA) and thermogravimetric derivative (DTG) of the GCF (P) and GCF (C) samples are illustrated in Figure 4.3, demonstrating the variations in mass loss when exposed to heating up to 800 °C in an inert nitrogen (N₂) atmosphere. The TGA curve for the GCF (P) sample, representing the pure coconut fiber, revealed four distinct stages of mass loss: 10.24% at 100 °C, 12.87% at 180 °C, 21.97% at 232 °C, 36% at 292 °C and 55.49% at 336 °C. These stages can be attributed to moisture elimination, hemicellulose degradation, cellulose decomposition and lignin degradation.

The first stage of the GCF (P) sample (100°C): It shows a small initial mass loss that can be attributed to the evaporation of adsorbed water and other volatile substances (Basu *et al.*, 2015). Stage 2 (200°C - 300°C): There is a sharp decline in mass, which is related to the decomposition of components such as polysaccharides and different distinct species of oil found in the coconut fiber (Bekirogullari, 2020). Stage 3 (300°C - 500°C): Another significant mass loss is observed, associated with the thermal degradation of cellulose and lignin (Akhyar *et al.*, 2024; Suresh Kumar; Duraibabu; Subramanian, 2014).

Above 500°C: The mass loss continues to decrease but at a slower rate, indicating the decomposition of carbonaceous residues. In the DTG analysis for sample GCF (P), the largest negative peak was around 320°C, corresponding to the maximum decomposition rate of cellulose and hemicellulose (Akhyar *et al.*, 2024). The fact that the peak is sharper suggests a rapid decomposition of these components; a secondary peak at (400°C): is related to the decomposition of lignin.

The analysis of the samples reveals significant differences in the mass loss profiles due to the chemical treatments applied to the coconut fiber. The GCF sample (C), corresponding to the catalyst generated from coconut fiber, presents three main stages of mass loss: 0.13% at 44 °C, 7.54% at 205 °C and 43.79% at 330 °C. The minimum initial loss at 44 °C indicates the

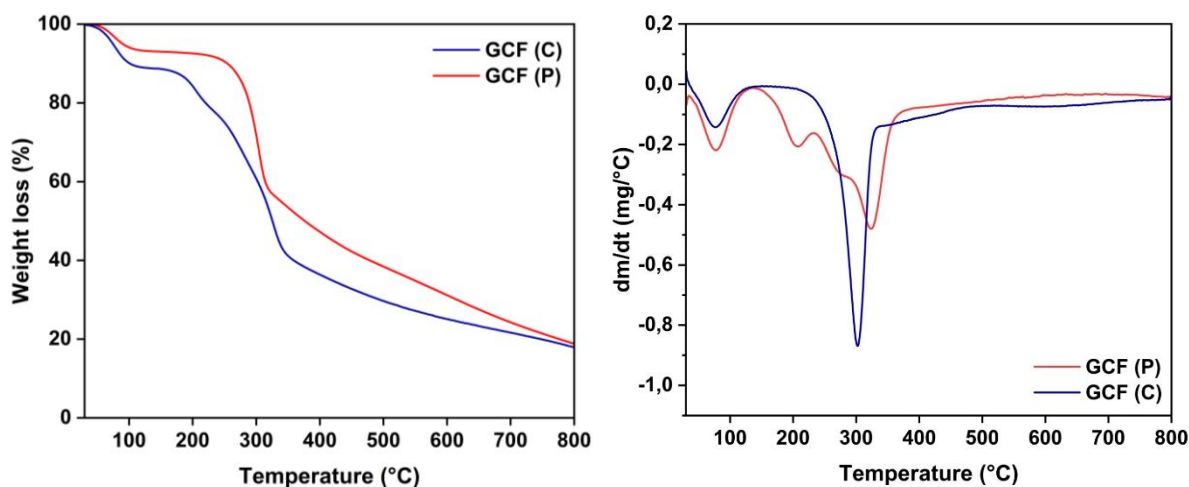
removal of residual moisture or light volatile compounds. The second stage, with 7.54% loss at 205 °C, is associated with the degradation of more stable components, possibly as a result of the chemical treatment that the fiber underwent.

The third and most significant stage, with 43.79% loss at 330 °C, suggests the decomposition of remaining carbonaceous compounds. Comparatively, the chemically treated sample (GCF C) demonstrates greater thermal stability in the first stages of heating, but a more concentrated mass loss in the third stage, around 330 °C, indicating the degradation of organic compounds that remain stable at lower temperatures.

In the initial stages (100 °C), the mass loss, similar to that observed in GCF (P), is related to the evaporation of residual water. In the second stage (250 °C - 350 °C), the TGA curve indicates a more pronounced decomposition compared to GCF (P), suggesting that the treatment with H_2O_2 and H_3PO_4 may have promoted the breaking of ether bonds and the removal of more volatile components and the lignocellulosic structure. In the third stage (above 350 °C), the mass loss becomes slower, indicating greater thermal stability of the remaining residues.

The main peak (300 °C) is shifted to a slightly lower temperature, suggesting that alkaline and acid treatment facilitated cellulose degradation and reduced matrix complexity, leading to faster decomposition. A secondary peak (350 °C - 450 °C), wider and less pronounced, suggests a more gradual decomposition of lignin and other remaining components, possibly due to structural modification induced by chemical treatment (Lomelí-Ramírez *et al.*, 2014).

Figure 4.3 - Thermogravimetric analysis (TGA) and derivative mass loss analysis (DTG) of GCF-C and GCF-P samples.



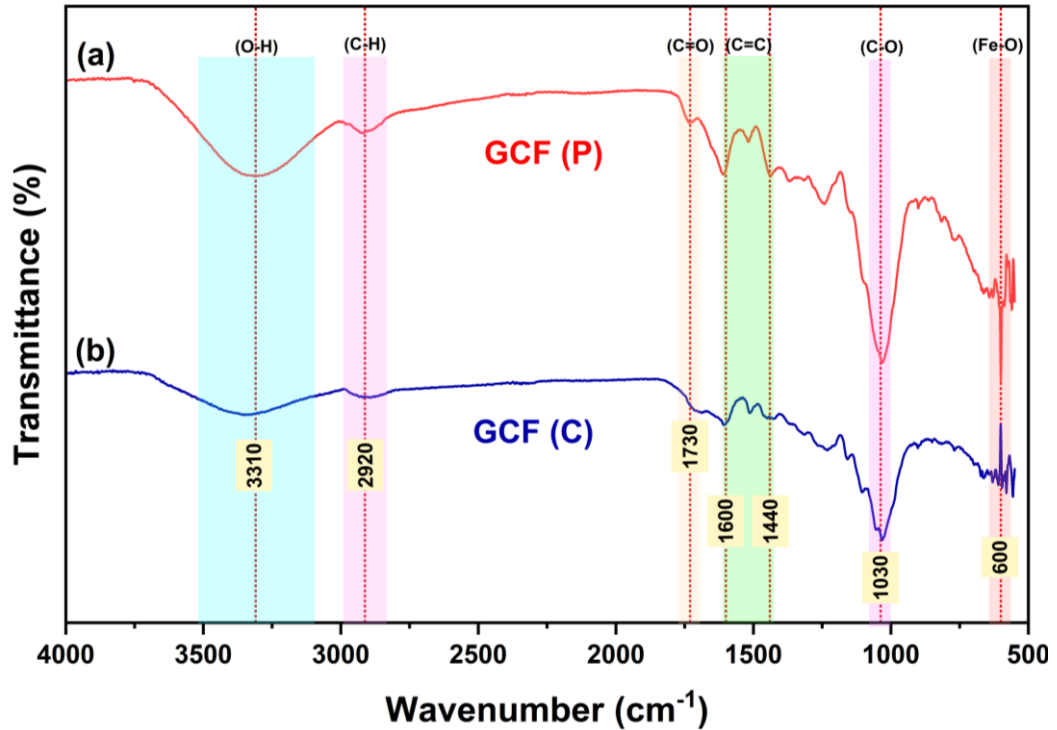
Source: Author (2024).

Fourier transform infrared (FTIR) spectra of the analyzed samples can reveal the compounds generated during thermal degradation. For the GCF (P) sample, the presence of characteristic bands of functional groups such as OH (hydroxyl), C=O (carbonyl), and C-O (esters and alcohols) is expected, indicating the decomposition of hemicellulose, cellulose, and lignin (Lomelí-Ramírez *et al.*, 2014). In the GCF sample (C), chemical treatments can alter these functional groups, introducing new bands in the FTIR, such as those associated with phosphates (PO_4^{3-}) or other structural modifications that result in compounds that are more stable at temperature, always considering that degradation occurs without oxidation.

4.3.3 Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR spectrum presented reveals important absorption bands that indicate different functional groups in the two samples (a) and (b). The analysis was performed on samples (a) GCF (Catalyst) treated with 15% H_3PO_4 and GCF (pure) without any chemical treatment, showing a comparison of the two spectra in Figure 4.4. FTIR spectra presented different bands at 3310, 2920, 1730, 1440, 1030 and 600 cm^{-1} . A broad absorbance was recorded at 3310 cm^{-1} this band is attributed to the stretching of hydroxyl groups (O-H) that contain adsorbed water, alcohol, carboxylic acid and phenolic compounds (Visiamah; Trisunaryanti; Triyono, 2024). The 2920 cm^{-1} band is related to stretching vibrations of aliphatic groups (C-H), indicating hydrocarbon chains (PUZIY *et al.*, 2002; ZHANG *et al.*, 2018). The 1730 cm^{-1} band is characteristic of stretching vibrations (C=O), pointing to groups (carbonyl) in ketone and aldehydes (Demiral; Samdan; Demiral, 2021). Bands found in the 1600 and 1440 cm^{-1} range are attributed to stretching vibrations of aliphatic groups (C=C), common in alkenes, aromatics and deformation (C-H) in groups (CH_2) (Bampi *et al.*, 2024; Jiang *et al.*, 2021; Suprianto *et al.*, 2021). Furthermore, it is possible to observe the 1030 cm^{-1} band that indicates C-O stretching vibrations, present in ethers, alcohols and esters, while the 600 cm^{-1} band presents vibrations characteristic of (Fe-O) tetrahedral sites, confirming the presence of metals even if in small quantities (Melo *et al.*, 2024b; Toderascu *et al.*, 2023).

Figure 4.4 - FTIR spectra of green coconut fibers (GCF) (a) in pure (GCF-P) and catalyst-treated (GCF-C) (b) forms.



Source: Author (2024).

This information allows a detailed understanding of the chemical composition of the two samples. In addition, the stretching observed in the 1030 cm^{-1} band for sample (a) GCF (C) can be explained by the structural changes induced by chemical treatments. The treatment with phosphoric acid and hydrogen peroxide in acidic and basic media probably caused the removal of impurities and the introduction of new functional groups on the surface of the biomass.

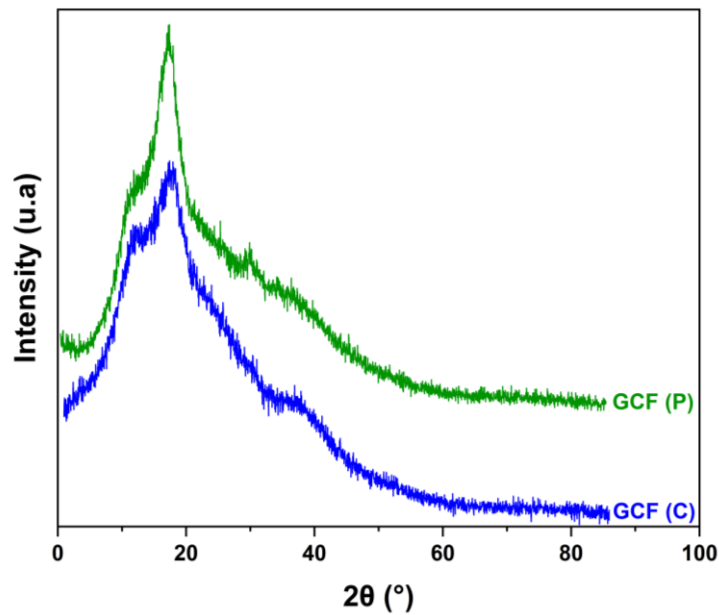
These new functional groups, such as phosphate and peroxide groups, can decrease the energy absorption in the 1030 cm^{-1} region, resulting in a smaller stretching. In contrast, sample (b) GCF(P), which has not undergone any treatment, maintains its original pure biomass structure, with no chemical modifications in the 1030 cm^{-1} band.

4.3.4 X-ray Diffraction (XRD)

Samples (GCF-P) and (GCF-C). were analyzed by means of an X-ray diffractometer in order to determine their purity and crystal structure (Figure 4.5). Diffraction peaks were observed at $2\theta = 11.82^\circ$, 17.39° , 25.33° , 29.88° , and 36.39° , indicating a high crystallinity

index. Peaks in the ranges of 7 to 24° are associated with carbon and sugars that are found in coconut fiber(Kaya, 2020a). However, these peaks, related to the crystal structures present in sample (GCF-P), disappear after treatment with phosphoric acid and subsequent burning of the catalyst (GCF-C).

Figure 4.5 - Comparative Analysis of X-ray Diffraction Intensity for Pure Green Coconut Fiber (GCF-P) versus Green Coconut Fiber Catalyst (GCF-C).



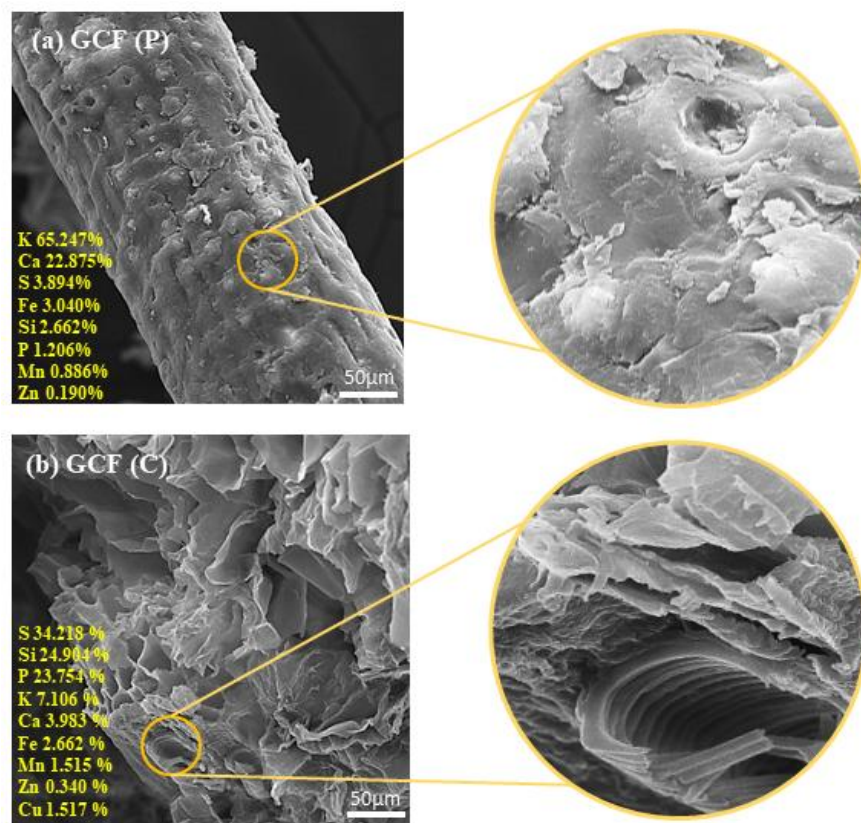
Source: Author (2024).

The decrease in the intensity of the diffraction peaks observed in sample (GCF-C) can be attributed to the chemical treatments applied. The initial treatment with hydrogen peroxide removes lignin and hemicellulose, which are amorphous components of the biomass, exposing more of the crystalline structure of the cellulose. However, this process can also compromise the integrity of the crystalline structure, resulting in a reduction in the intensity of the peaks. In addition, the subsequent treatment with phosphoric acid, followed by burning, aims to increase the porosity of the material. As a result, although these treatments increase the specific surface and porosity of the material, they also cause a reduction in the intensity of the diffraction peaks due to interference in the crystalline structure, in contrast to (CGF – P), which maintains its original structure.

4.3.5 Scanning Electron Microscopy (SEM) and X-ray Fluorescence (XRF)

SEM images of sample (a). CCF (P) without any treatment and (b). GCF (C) treated with 15% H_3PO_4 are observed in Figure 4.6. The (Figure 4.6-a) reveals a relatively smooth surface with some irregularities and small elevations in the green coconut fiber.

Figure 4.6 - Microstructural and Elementary Characterization of GCF-P and GCF-C Using SEM and XRF.



Source: Author (2024).

After the treatment with H_3PO_4 and firing process, sample GCF (C) figure (Figure 4.6-b) presented a textured layered surface exhibiting a more porous structure. This effect is attributed to the treatment with phosphoric acid (H_3PO_4), which is known to increase the surface area and create more active sites for catalysis.

The increase in porosity indicates that the activation process was effective, promoting the removal of volatile materials and the restructuring of the carbonaceous matrix, resulting in a material with greater catalytic potential. The comparison between the two SEM images highlights the substantial transformation in the surface morphology, proving that the catalyst

formation process was successfully carried out. This improvement in the porous structure is essential for catalytic applications, where a larger surface area and the presence of more active sites directly contribute to the efficiency of the catalyst. In addition, an XRF elemental analysis was performed on both samples (Figure 4.6-a) and (Figure 4.6-b) to identify the percentage degree of the composition of the elements present in the samples. When comparing the two samples, a significant increase in the percentage of sulfur (S), silicon (Si) and phosphorus (P) is observed in sample (Figure 4.6-b) compared to sample (Figure 4.6-a). Sulfur increased from 3.394% to 34.218%, silicon from 2.662% to 24.994%, and phosphorus from 1.206% to 23.735%. On the other hand, there is a marked reduction in the potassium (K) and calcium (Ca) contents, with potassium decreasing from 65.247% to 7.106%, and calcium from 22.875% to 2.3938%. There was also a reduction in the levels of iron (Fe), manganese (Mn) and zinc (Zn) in the sample (Figure 4.6-b).

Furthermore, copper (Cu) was detected only in sample (Figure 4.6-b), with a presence of 0.1517%, which was not observed in sample (Figure 4.6-a). These results indicate a significant variation in the elemental composition between the two samples, possibly due to differences in the burning and treatment applied. SEM and XRF analyses were essential to evaluate the effectiveness of the H_3PO_4 treatment in removing impurities and increasing the porosity of the catalyst (Figure 4.6-b), proving the success of the process. The treatment with H_3PO_4 modified the fiber's structure, making it more porous and exposed, and altered its composition by increasing phosphorus and decreasing removable elements such as potassium and calcium. The presence of copper is likely due to contamination or adsorption during the experimental process.

4.3.6 Gas Chromatography (GC)

Chromatographic analysis of the gas generated after the hydrolysis of sodium borohydride revealed the presence of hydrogen, confirming that the decomposition reaction of NaBH_4 to produce H_2 was effective. However, the quantitative analysis of the hydrogen concentration was compromised due to failures in the isolation of the reaction system. The system was not purged with inert gas, resulting in the presence of atmospheric gases such as nitrogen and oxygen in the reactor. This introduced contamination into the generated gas and made it impossible to accurately quantify the hydrogen production efficiency.

Despite these limitations, chromatography indicated that the GCF-C catalytic system generated a significant concentration of hydrogen, reaching 58.8% (vol/vol). This concentration

suggests promising performance of the catalytic system, but the impact of atmospheric contamination, with nitrogen (28.9%) and oxygen (12.2%), demonstrates that the system was not completely purged. The presence of these gases indicates that part of the volume of hydrogen generated may have been diluted, reducing the overall efficiency of the process.

Although the GCF-C system demonstrated potential for hydrogen production, the lack of tight control over the reaction environment prevented an accurate assessment of the catalyst's maximum capacity. To optimize the process, the tests will be repeated by completely purging the reactor with inert gas prior to the reaction and implementing more representative gas collection techniques to eliminate interference from atmospheric gases and ensure more accurate data on hydrogen production efficiency.

4.3.7 Process Optimization by the Taguchi Method

The Taguchi method was used to optimize the catalytic hydrolysis reaction of an aqueous sodium borohydride solution, using a catalyst synthesized from green coconut fiber biomass, aiming at hydrogen generation. The reaction parameters that influence hydrogen generation were identified and optimized with a reduced number of experiments. In this study, the following parameters were analyzed: reaction time, amount of sodium borohydride, amount of catalyst and reaction agitation speed, as previously presented in Table 4.3. The selection of the ranges of the variables for these parameters allowed the evaluation of the interactions between them, enabling the determination of the best level for each of the parameters studied and the achievement of the optimal reaction conditions.

Table 4.3 presents the results of the experimental design, showing the volumes of hydrogen generated for each point analyzed. Observing Table 4.3, it is evident that the amount of sodium borohydride was the parameter that most influenced the process, positively impacting hydrogen generation. This positive effect occurs because sodium borohydride is the substrate that, when decomposed in the presence of the catalyst, releases hydrogen into the system. Therefore, a greater amount of available sodium borohydride increases the amount of hydrogen that can be generated. As demonstrated in experiments 4, 8 and 16, a high amount of sodium borohydride is associated with greater hydrogen generation, since the three experiments performed with 750 mg of the commercial substrate presented the highest volumes generated among all 16 experiments. In contrast, experiments 1, 9 and 13, which used low concentrations of sodium borohydride (100 mg), resulted in a reduced reaction yield. The results also reveal

that the amount of catalyst used in the generation of hydrogen from the hydrolysis of NaBH_4 was a highly effective parameter and influences the rate of hydrogen release.

Table 4.3 - Results of the Experimental Project of the Taguchi L16 Plan, using Pure Green Coconut Fiber (GCF-P) and Green Coconut Fiber Catalyst (GCF-C).

Exp	GCF-C (mL)	S/R (GCF-C)
1	60,67 \pm 5,00	35,66
2	172,00 \pm 5,01	44,71
3	356,67 \pm 5,13	51,05
4	596,67 \pm 2,89	55,51
5	131,67 \pm 12,58	42,39
6	190,00 \pm 5,00	45,58
7	346,33 \pm 12,18	50,79
8	490,00 \pm 8,66	53,80
9	115,00 \pm 5,01	41,21
10	386,67 \pm 11,55	51,75
11	168,33 \pm 7,64	44,52
12	373,67 \pm 10,14	51,45
13	140,00 \pm 5,06	42,92
14	260,00 \pm 5,04	48,30
15	323,33 \pm 7,64	50,19
16	428,33 \pm 2,89	52,64

Exp: Number of experiments, S/N: Signal/Noise, (GCF-C): Green Coconut Fiber Catalyst

Source: Author (2024).

The best catalytic performance was observed in experiment 4, in which 300 mg of catalyst were used in a reaction with 750 mg of NaBH_4 in aqueous medium, releasing 596.67 mL of H_2 in 10 minutes at 60 °C. A similar trend was noted in relation to the reaction temperature. For example, experiments 4 and 16, which presented the largest volumes of hydrogen (596.67 mL and 428.33 mL of H_2 , respectively) (Table 4.2) were conducted at temperatures above 50 °C.

The hydrolysis of sodium borohydride is sensitive to the temperature applied in the reaction (Abdelhamid, 2021b). Studies confirm that hydrogen generation increases with increasing temperature (Karaoglu; Yolcular, 2022). This occurs because the higher temperature increases the kinetic energy of the molecules, facilitating the overcoming of the activation

energy barrier. In the experiments carried out, the catalyst demonstrated the ability to withstand high temperatures without losing its catalytic performance.

4.3.8 Statistical Analysis

The Taguchi method uses the signal-to-noise (S/N) ratio to assess the importance of each parameter studied (Simão Neto *et al.*, 2023). In the present study, the ‘higher is better’ function was applied to identify these ratios based on the volumes of hydrogen generated, aiming to optimize reaction performance.

Table 4.4 - Response to mean S/N ratios and ordering of variables (Taguchi L16 Plan using a GCF-C).

Factor levels	t (min)	Cat (mg)	NaBH ₄ (mg)	T (°C)	Agitation (rpm)
1	46,73236	44,59828	40,54626	46,54951	47,85067
2	48,13960	47,18570	47,58296	46,49011	47,33751
3	47,23344	48,59065	49,13789	49,45428	47,74814
4	48,51267	50,24345	53,35097	48,12417	47,68176
Delta	1,78031	12,80471	5,64517	2,96417	0,51316
Ranking	4°	1°	2°	3°	5°

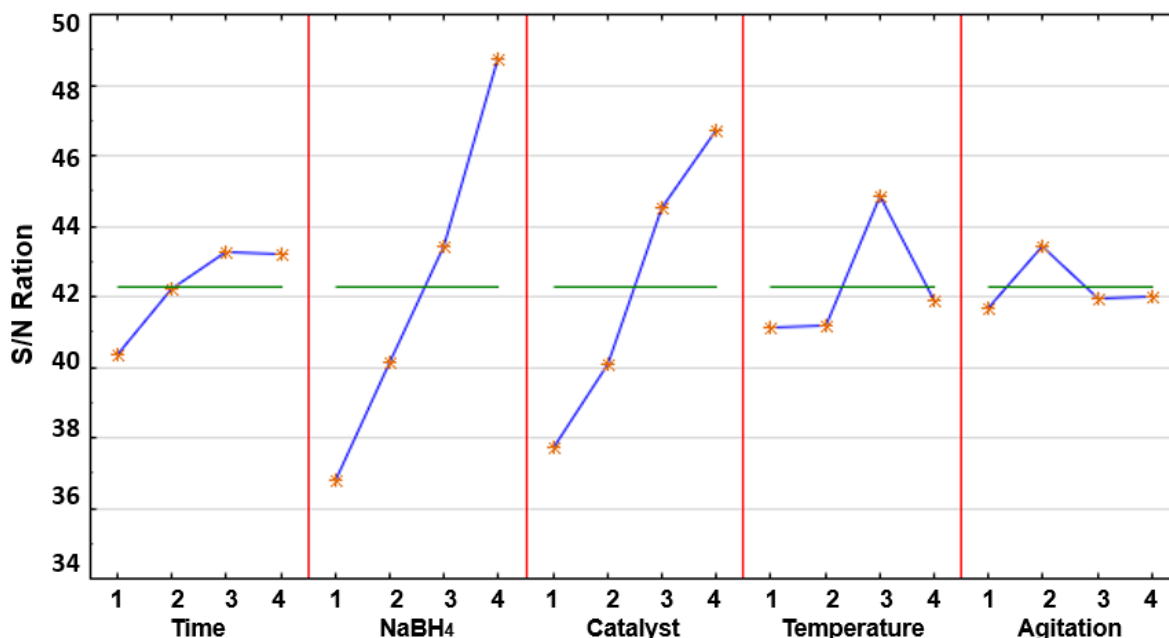
Source: Author (2024).

Table 4.4 and Figure 4.7 provide additional relevant data, including the average S/N for all levels of each factor and the delta values associated with each of these factors. The delta value results are calculated by subtracting the factor with the highest S/N value from the factor with the lowest S/N value, and this analysis uses the delta value to rank the factors and determine their importance to the process. The analysis of Table 4.4 shows that the amount of sodium borohydride, the amount of catalyst, and the reaction temperature were the factors that most influenced the reaction among the other parameters analyzed, with delta values of 12.80, 5.65, and 2.96, respectively. These parameters, especially the first two parameters, showed significantly high delta values, indicating that they have a greater impact on the reaction efficiency compared to the other factors.

In figure 4.7 presents the main effects plot for means, which illustrates how process parameters influence hydrogen generation. This plot, generated by the software ©Minitab (version 19), shows the mean of the characteristic for each level of the analyzed parameters. When the line on the plot is horizontal, this indicates that there is no main effect, meaning that

the levels of the factor have the same impact. In contrast, a sloped line suggests that different levels of the factor affect the characteristic differently (Karaoglu; Yolcular, 2022).

Figure 4.7 - Graphic of the averages of the parameter's variations (GCF-C).



Source: Author (2024).

The magnitude of the effect is reflected in the vertical difference between the points, and the comparison of the slopes of the lines reveals the relative magnitude of the effects of the factors, with variables with steep slopes having a greater impact (Karaoglu; Yolcular, 2022). In Figure 4.7, the concentration of sodium borohydride, with the steepest slope, exerts the greatest effect on the response, while the decreasing slope indicates a reduced effect with decreasing substrate. Furthermore, hydrogen production is significantly influenced by increasing temperature and catalyst amount, while stirring speed has the least influence, as evidenced by the line with the lowest slope.

The analysis of variance confirmed similar results, highlighting the p-value, which assesses the significance of the factors in the studied reaction, as shown in Table 4.5. A p-value below 0.05 indicates significance with up to 95% confidence (Sağır; Elçiçek; Özdemir, 2021). All parameters showed significance within the desired reliability range. The amount of sodium borohydride had the largest contribution, representing 77.19% of the total, as expected. The theoretical optimal conditions for the reaction were 40 minutes, 300 mg of catalyst, 750 g of sodium borohydride, temperature of 50°C, and stirring speed of 120 rpm.

Table 4.5 - Results of analysis of variance (ANOVA) for parameters that affect the hydrogen production (GCF-C).

Factors	SS	DF	IN	F value	p-value	Contribution (%)
t (min)	7,9976	3	2,6659	13,4663	0,030206	1,81
NaBH ₄ (mg)	340,7297	3	113,5766	573,7173	0,000123	77,19
Cat (mg)	68,5573	3	22,8524	115,4361	0,001348	15,53
T (°C)	24,1465	3	8,0488	40,6576	0,006268	5,47
Total	441,4310	12	-	-	-	100

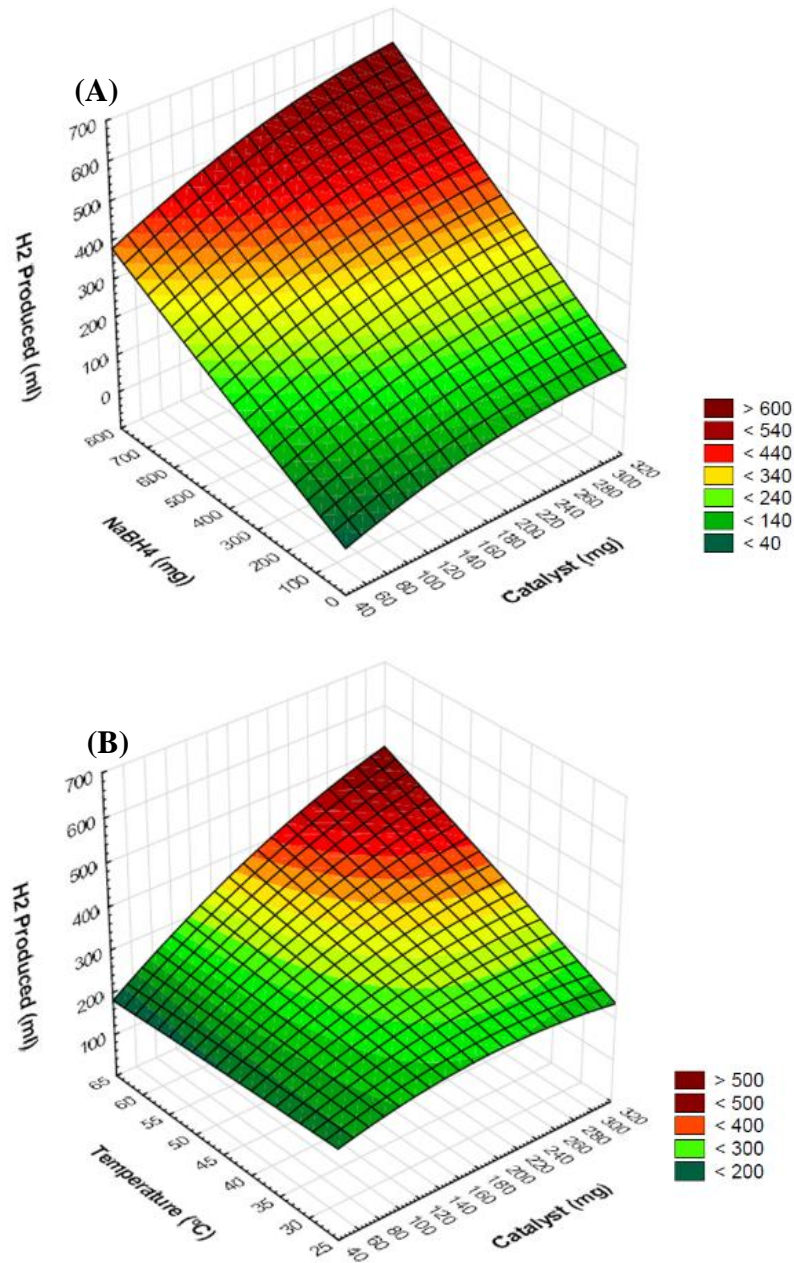
Source: Author (2024).

Contour surface plots, presented in Figure 4.8, were constructed to visualize the interactive effects and illustrate the relative influence of two parameters while keeping the other factors constant.

The Figure 4.8 shows that the largest volume of H₂ (represented by the reddish area in the plot) was obtained with the maximum concentrations of NaBH₄ (750 mg) and catalyst (300 mg). The increase in H₂ generation with the NaBH₄ concentration can be explained by the influence of heat and mass transfer on the hydrogen production rate (Chen, Yuerong; Jin, 2020; Karakaya; Pehlivan; Ceyhan, 2024b).

Heat transfer, which raises the reaction temperature, plays a significant role in the process due to the exothermic nature of NaBH₄ hydrolysis (Demirci, 2023). Fangaj *et al.* (2020) found that a higher concentration of NaBH₄ results in a higher hydrogen density. They used ground apricot kernel shell (GAKS) as an economical and efficient catalyst for the hydrolysis of NaBH₄, and found an activation energy of 30.23 kJ mol⁻¹ for this biomass treated with 15% H₃PO₄(Fangaj; Ceyhan, 2020b). However, high concentrations of NaBH₄ can lead to slower kinetics due to increased solution viscosity and coverage of the catalyst surface by NaBO₂, a byproduct of the reaction (Abdelhamid, 2021b).

Figure 4.8 - Boundary surfaces for hydrogen production using Biocatalyst supported on coconut shell biomass. (A) NaBH_4 (g) versus Catalyst (mg). (B) Temperature ($^{\circ}\text{C}$) versus Catalyst (mg).

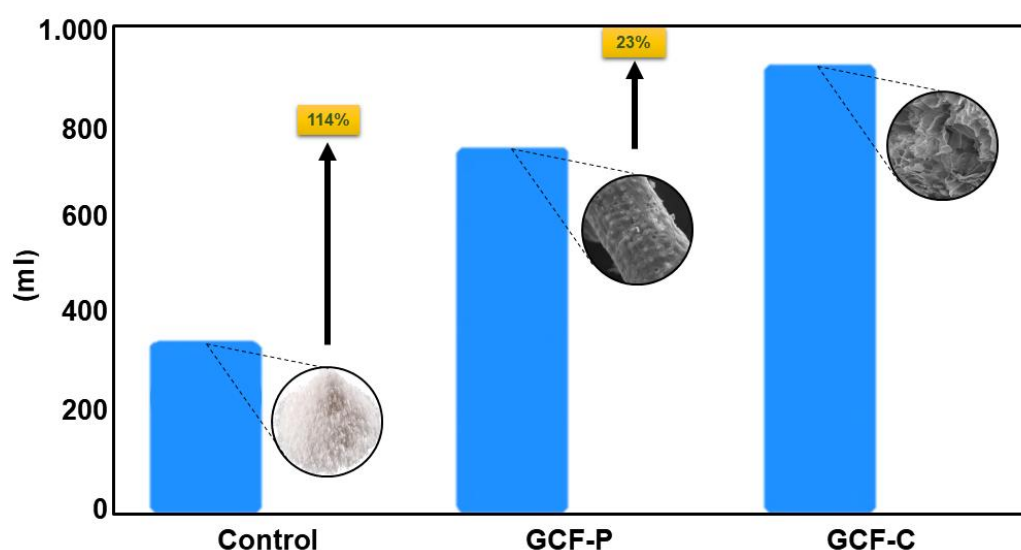


Source: Author (2024).

A faster reaction occurs with a larger catalytic interaction area and low solution viscosity. High concentrations of NaBH_4 can cause NaBO_2 precipitation, blocking the active sites of the catalysts and leading to their deactivation due to the deposition of borate species on their surface (Abdelhamid, 2021b; Demirci, 2023).

The results presented in Figure 4.9 reflect the significant difference in catalytic performance between the three experimental conditions: control (without catalyst), GCF-P, and GCF-C, in the hydrolysis of sodium borohydride (NaBH_4) for hydrogen production, reproducing the optimal conditions identified. The control, which represents the hydrolysis of NaBH_4 without the presence of a catalyst, presented a hydrogen production of approximately 350 mL. This production suggests a spontaneous hydrolysis reaction, but with a very low reaction rate and, consequently, a low hydrogen release efficiency. This result is expected, since sodium borohydride, when not catalyzed, reacts in a limited way due to the activation barriers inherent to the reaction. The GCF-P catalyst provided a production of approximately 850 mL of hydrogen, which corresponds to an increase of 114% in relation to the control. This indicates that GCF-P is effective in accelerating the hydrolysis reaction, making the process much more efficient than without the use of catalysts. This increase in production is explained by the presence of active sites on the surface of GCF-P, which reduce the activation energy of the reaction, allowing NaBH_4 to decompose into hydrogen more quickly and efficiently. However, although GCF-P showed significantly better performance than the control, it still did not reach the maximum possible production, which suggests that its structure or composition may not be ideal for optimizing the hydrolysis reaction. Limitations in porosity or exposure of catalytic sites may be factors that restrict the total hydrogen production.

Figure 4.9 - Comparison of hydrogen production from the hydrolysis of sodium borohydride under optimal conditions determined by the Taguchi method, using the control (without catalyst), GCF-P and GCF-C.



Source: Author (2024).

GCF-C showed the best catalytic performance, with hydrogen production reaching 950 mL, representing a 23% increase compared to GCF-P and 137% compared to the control. This result suggests that GCF-C has superior catalytic properties to GCF-P, which can be attributed to a more efficient porous structure and a larger surface area that allow for greater interaction between NaBH_4 and the active sites of the catalyst. The porous morphology of GCF-C facilitates the adsorption and diffusion of reactant molecules, reducing the mass resistance and allowing for a more efficient decomposition of sodium borohydride. Furthermore, the significant increase in hydrogen production compared to GCF-P and the control suggests that GCF-C offers a higher density of accessible catalytic sites, which results in a higher reaction rate. The superiority of GCF-C highlights the impact of material structure on catalytic efficiency, suggesting that modifications to the catalyst surface and morphology may be effective strategies to further improve hydrogen production.

4.3.9 Kinetic Study of the Catalyst

4.3.10 Production Rate

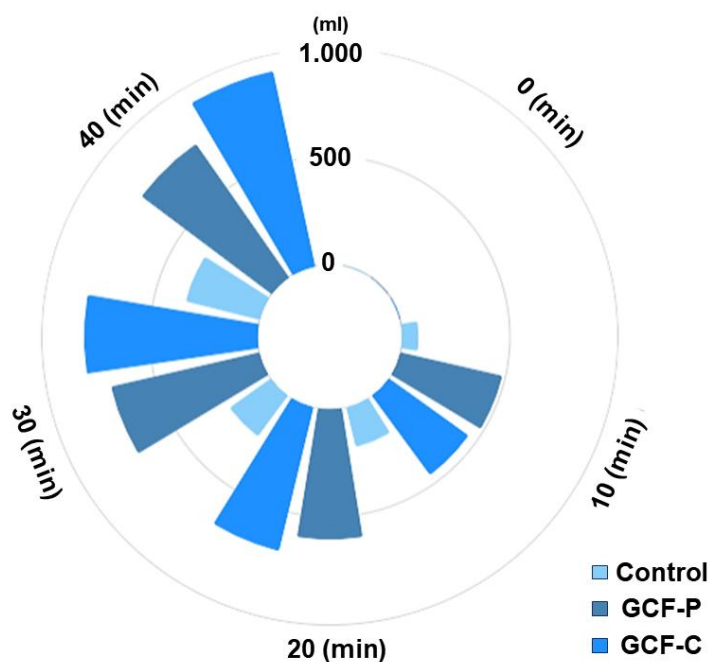
The kinetic study of hydrogen production, illustrated in Figure 4.10, allows a detailed analysis of the behavior of different experimental conditions, including the control reaction, without a catalyst, and the reactions catalyzed by pure biomass (GCF-P) and modified biomass (GCF-C).

In the control condition, where the reaction occurs without the use of a catalyst, slow and nonlinear kinetics were observed. Hydrogen production was marked by periods of low activity interspersed with sporadic increases in the production rate, which highlights the difficulties inherent in the spontaneous hydrolysis of sodium borohydride (NaBH_4). This low efficiency is attributed to the high activation energy required to break the hydrogen bonds in NaBH_4 , resulting in a slow and limited reaction in terms of hydrogen production over time. Similar studies report that the activation energy for the hydrolysis of NaBH_4 without a catalyst can be significantly high in non-catalytic systems (Andrieux *et al.*, 2011).

The graph also reveals that GCF-C presented linear and consistent hydrogen production, indicating uniform reaction kinetics and a stable catalytic mechanism over time. The absence of fluctuations in the production rate suggests that GCF-C maintained its catalytic activity efficiently, with an average production rate of $104.17 \text{ ml H}_2 \cdot \text{min}^{-1} \cdot \text{g}^{-1}$. This high rate indicates that the treatment applied to the modified biomass was effective in improving the catalytic properties of the material. Modifications such as increased porosity (Qin *et al.*, 2019) and the

introduction of new functional groups (Quílez-Bermejo; Morallón; Cazorla-Amorós, 2020) probably directly contributed to the superior catalytic behavior of GCF-C, making it a highly effective catalyst for hydrogen production.

Figure 4.10 - Hydrogen production rate by hydrolysis of sodium borohydride.



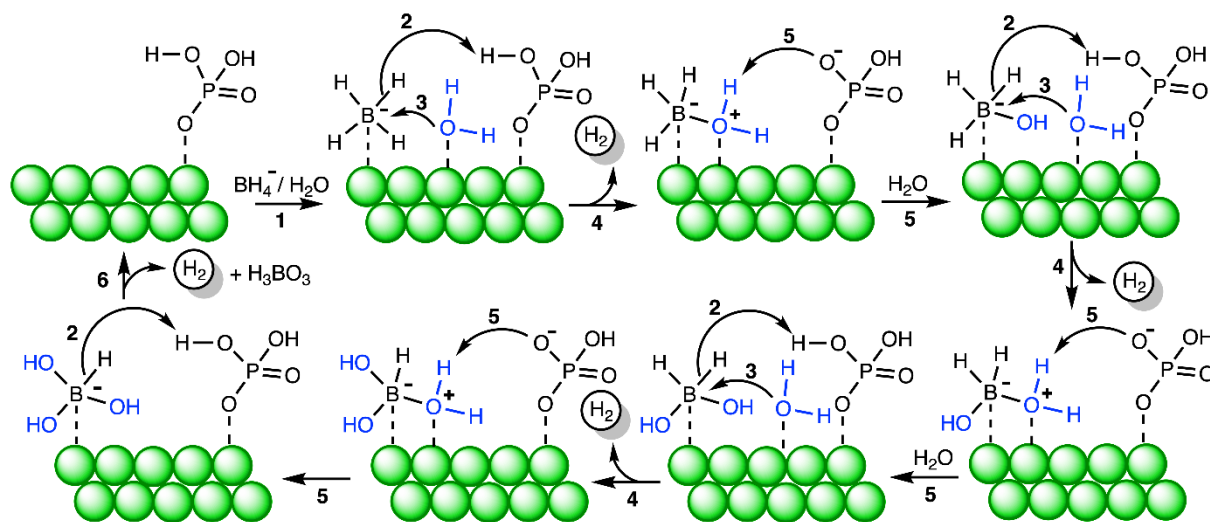
Source: Author (2024).

In contrast, GCF-P exhibited a biphasic behavior, where initially the hydrogen production was comparable to that of GCF-C. However, after 20 minutes, a significant kinetic change occurred, resulting in a significant increase in the production rate. This behavior suggests the activation of an additional mechanism or the overcoming of an energy barrier, leading to an acceleration of the reaction. With an average production rate of $65.83 \text{ mLH}_2 \cdot \text{min}^{-1} \cdot \text{g}^{-1}$, GCF-P demonstrates that biomass, even in its unmodified form, has catalytic potential in the hydrolysis of NaBH_4 . However, the efficiency of GCF-P is still lower than that of GCF-C, possibly due to limitations such as lower porosity and lower presence of catalytic active sites.

The proposed reaction mechanism for hydrogen production via GCF-P elucidates Figure 4.11 the influence of surface-adsorbed phosphoric acid groups in facilitating NaBH_4 hydrolysis through proton transfer and nucleophilic activation. The process starts with the adsorption of NaBH_4 and water molecules on the catalyst surface, where hydride ions interact with acidic

phosphate groups, promoting the dissociation of hydrogen species. This interaction reduces the activation energy by providing a pathway for hydride abstraction and subsequent nucleophilic attack on the boron center by water molecules. The biphasic behavior observed in GCF-P can be attributed to the progressive activation of catalytic sites, where an initial adsorption-limited stage transitions to a reaction-controlled regime as more active sites become available.

Figure 4.11 - Proposed reaction mechanism to produce H_2 by GCF-C.



Source: Author (2024).

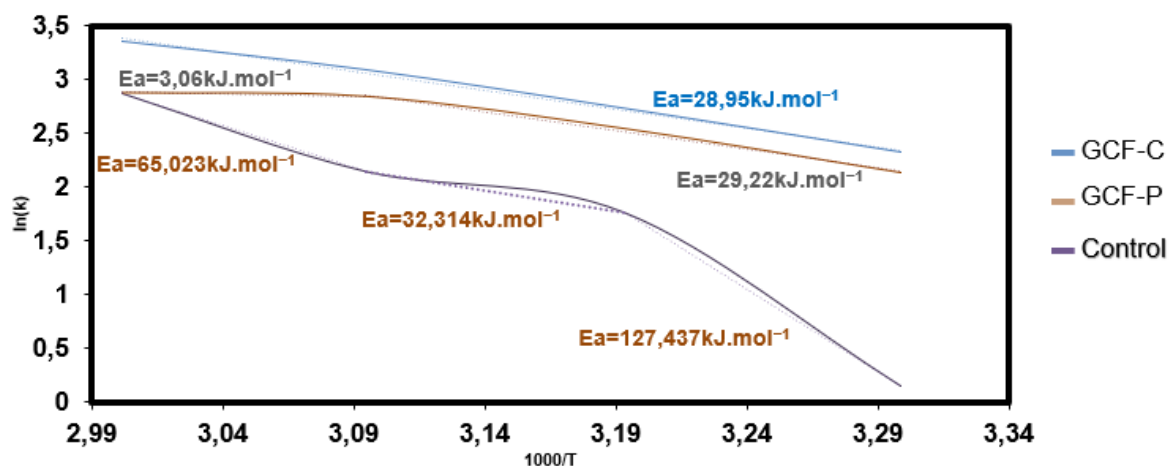
The late kinetic increase suggests that the interaction between borohydride species and phosphate groups undergoes a time-dependent activation process, leading to a higher rate of hydrogen evolution after the initial phase. Furthermore, regeneration of catalytic sites via deprotonation of water molecules ensures sustained activity, distinguishing GCF-P from GCF-C in terms of reaction kinetics and hydrogen yield. This mechanistic interpretation aligns with the observed performance differences, highlighting the role of surface chemistry modifications.

4.3.11 Activation Energy (E_a)

The performance of the GCF-C catalytic system in the hydrolysis of sodium borohydride ($NaBH_4$) is represented by a single linear region in the Arrhenius plot (Figure 4.12), with an apparent activation energy (E_a) of $28.95 \text{ kJ.mol}^{-1}$. This linearity suggested that the reaction mechanism of GCF-C remained constant throughout the studied temperature range, with no abrupt changes in E_a , indicating that the hydrolysis process was homogeneous and stable. Comparatively, nickel (Ni)-based catalysts report E_a values of 73 kJ.mol^{-1} (Cento; Gislon; Prosini, 2009) for the hydrolysis of $NaBH_4$, and 52 kJ.mol^{-1} for stabilized sodium borohydride solutions with supported Ni (Zhang, Qinglin *et al.*, 2007). These values are significantly higher

than the 28.95 kJ.mol⁻¹ observed for GCF-C, suggesting that GCF-C offers a substantially lower energy barrier. This may be advantageous in terms of energy efficiency and cost-effectiveness, since nickel, although more accessible than noble metals, still presents a higher energy demand for reaction activation.

Figure 4.12 - Arrhenius diagram for the catalytic hydrolysis of NaBH₄.



Source: Author (2024).

The GCF-P catalytic system presented two distinct Ea phases. Initially, the Ea was 29.22 kJ.mol⁻¹, similar to that of GCF-C, and the reaction accelerated with increasing temperature. However, in the second phase, the Ea dropped to 3.06 kJ.mol⁻¹, indicating that the reaction rate increased significantly, resulting in a peak in hydrogen production. This behavior suggested that GCF-P could have advantageous applications at higher temperature conditions, where hydrogen release occurred almost immediately after the initial phase. When compared to Ruthenium (Ru) (Zhang, J.S. *et al.*, 2007) on carbon catalysts that have an Ea of 66.9 kJ.mol⁻¹ the GCF-P shows exceptional performance at high temperatures, with a significant cost and availability advantage over platinum.

In contrast, the control reaction, carried out in the absence of a catalyst, demonstrated a substantially higher energy requirement to initiate and sustain the hydrolysis of NaBH₄. The reaction exhibited a nonlinear behavior, with three different Ea throughout the process. Initially, a high Ea of 127.437 kJ.mol⁻¹ reflected a significant energy barrier for the cleavage of the NaBH₄ bonds and the subsequent release of hydrogen, resulting in a very low initial reaction rate. This behavior can be attributed to the fact that, in the absence of catalysts, the hydrolysis reaction occurs in multiple simultaneous steps, producing several hydroxyborates as

intermediates (Andrieux *et al.*, 2011). These short-lived intermediates contribute to the complexity and high energy requirement of the reaction, making the cleavage of the NaBH_4 bonds less efficient.

As the reaction progressed, E_a decreased to $32.314 \text{ kJ.mol}^{-1}$, suggesting an increase in the efficiency of the process due to the formation of reactive intermediates that facilitated the hydrolysis. However, at higher temperatures, E_a increased again to $65.023 \text{ kJ.mol}^{-1}$, possibly due to the formation of by-products, such as sodium metaborate, which when present affects the reaction mechanism, potentially forming a protective layer that limits the further hydrolysis of NaBH_4 (Akkuş *et al.*, 2018; Marreroalfonso *et al.*, 2007). Thus, the increase in E_a reflects the additional difficulty imposed by the by-products and by the change in the conditions of the reaction medium, which affects the efficiency of the hydrolysis without the presence of a catalyst.

4.4 CONCLUSION

The catalysts supported on green coconut shell (GCF) biomass synthesized in this study demonstrated remarkable performance in hydrogen production from the hydrolysis of sodium borohydride (NaBH_4). The functionalization of the biomass with phosphoric acid (GCF-C) was successful in creating a highly porous structure rich in active catalytic sites, which significantly improved the efficiency of the catalytic process. The characterization of the catalyst by thermogravimetric analysis (TGA), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) techniques confirmed the structural modifications in the biomass, evidencing the creation of a stable and highly reactive carbon matrix.

The application of the Taguchi L16 experimental design was essential to optimize the reaction parameters, identifying the ideal conditions for hydrogen generation. The theoretical optimum conditions for the reaction were 40 minutes, 300 mg of catalyst, 750 g of sodium borohydride, temperature of 50°C and stirring speed of 120 rpm. The concentration of NaBH_4 was the most influential factor in hydrogen generation, followed by the amount of catalyst and temperature. These factors were shown to act synergistically, maximizing the performance of the catalytic system.

The kinetic analysis revealed that the GCF-C catalyst presented a linear and stable hydrogen production rate, with an activation energy (E_a) of 28.95 kJ.mol⁻¹, a value significantly lower than that of the control without catalyst, which highlights the effectiveness of GCF-C in reducing the energy barriers of the reaction. In comparison, the GCF-P (pure coconut fiber) catalyst also showed efficiency, although lower than GCF-C, with a hydrogen production rate of 65.83 mLH₂·min⁻¹·g⁻¹.

Morphological analyses confirmed that the applied chemical treatment provided a greater surface area of GCF-C, creating an optimized porous structure that facilitated the diffusion of reagents and the exposure of catalytic active sites. SEM images showed a significant transformation in the morphology of GCF-C, which went from a relatively smooth surface in GCF-P to a highly textured and porous structure after treatment, essential for the observed catalytic performance. X-ray fluorescence (XRF) analysis also revealed an increase in the amount of phosphorus in the treated sample, confirming the effectiveness of chemical doping. Although the study demonstrated the great potential of GCF-C for sustainable hydrogen production, some technical challenges were identified, such as the presence of atmospheric gases that compromised the accurate quantification of the hydrogen generated. To overcome this problem, future experiments will be carried out with the complete purging of the reaction system with inert gases. The reusability and storage stability of the catalyst have not yet been investigated, but based on the initial results, GCF-C is expected to maintain good catalytic stability in repeated reaction cycles. This suggests that GCF-C can be reused without significant loss of efficiency, making it an economically viable and environmentally sustainable alternative for hydrogen production. As a result, catalysts based on green coconut shell biomass (GCF-C) show promising performance in hydrogen production, with high catalytic efficiency. Further work should focus on investigating the potential for reuse of the catalyst, aiming at the potential for large-scale application and its integration on a pilot scale.

5 FINAL CONSIDERATIONS

The bibliometric analysis allowed mapping the scientific production related to energy conversion technologies from green coconut shells, evidencing a significant growth in publications between 2000 and 2024. This increase reflects the growing concern for sustainable waste management and the search for alternative energy sources. The results demonstrated that India, China and Brazil are the main contributors to the development of technologies related to the use of coconut biomass, with applications in thermochemical processes, production of activated carbon and adsorbents. The bibliometrics also revealed gaps and emerging trends, highlighting the need for more applied research to enable the efficient use of this waste in energy generation.

The overview and characterization of sustainable discoveries from green coconut shells (GCF) were successfully carried out, using different analytical techniques. Characterization by TGA, XRD, FTIR and SEM demonstrated the thermal stability of the material, the presence of active groups and a porous structure suitable for catalysis. These findings confirm that green coconut biomass can be a viable support for demonstrations, reinforcing its application in hydrogen production as a sustainable and low-cost solution.

The development efficiency in the hydrolysis of sodium borohydride (NaBH_4) was evaluated through kinetic experiments, which revealed a linear rate of hydrogen production and an activation energy of $28.95 \text{ kJ.mol}^{-1}$. These results indicate that GCF-C significantly reduces the energy barrier of the occurrence, favoring the conversion of NaBH_4 and making the process more efficient compared to non-catalyzed systems.

Finally, the optimal conditions for maximum H_2 production were determined with the Taguchi L16 methodology, establishing that 300 mg of GCF-C, 750 mg of NaBH_4 , temperature of 50°C and 120 rpm are the ideal settings for the occurrence. Under these conditions, the conversion was maximized, demonstrating the potential of the profit derived from green coconut shells for application in sustainable hydrogen generation. The results obtained reinforce the predictions of the reuse of lignocellulosic waste as a solution to energy challenges, contributing to the circular economy and the transition to clean energy. Future research should focus on the application of concrete in continuous systems and on the evaluation of its stability in multiple cycles, evolution, scalability and industrial application.

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