

Use of aldol condensation between furfural and acetone for biofuel production:

A review

Uso da condensação aldólica entre furfural e acetona para produção de biocombustível: Uma revisão

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Abstract

The continued reliance on fossil fuels and the associated environmental challenges have spurred the development of renewable energy sources, with biorefineries emerging as a prominent solution for converting biomass into valuable fuels and platform chemicals. Among these, the production of furfural, derived from C5 sugars in lignocellulosic materials, holds particular significance. Extended-chain hydrocarbon fuels and intermediates, such as 4-(2-furyl)-3-buten-2-one (FAc, C8) and 1,4-pentadiene-3-one-1,5-di-2-furanyl (F2Ac, C13), are synthesized via aldol condensation between furfural and acetone, followed by hydrogenation and hydrodeoxygenation processes. This study emphasizes the critical role of catalysts, particularly heterogeneous catalysts, in increasing the efficiency and selectivity of these reactions. Solid catalysts, compared to their homogeneous counterparts, offer substantial advantages, including ease of recovery, reusability, and increased sustainability. Advances in analytical techniques, such as gas chromatography-mass spectrometry (GC-MS) and other state-of-the-art methods, have been instrumental in refining the characterization of heterogeneous catalysts, ensuring improved product quality and process optimization. Additionally, the study explores cutting-edge methodologies for catalyst characterization, utilizing tools such as field emission scanning electron microscopy (FESEM), X-ray photoelectron spectroscopy (XPS), Fourier-transform infrared spectroscopy (FTIR), and inductively coupled plasma optical emission spectroscopy (ICP-OES) to obtain precise quantitative and qualitative insights. This review provides a detailed analysis of the integration of these technologies into biofuel production, highlighting the critical role of innovative catalysts—particularly bifunctional systems

under controlled conditions—and the development of optimized conversion routes. These strategies are essential for advancing industrial efficiency, improving process selectivity, and contributing to the sustainability of the energy sector.

Keywords: Aviation biofuels. Heterogeneous catalysis. Aldol condensation.

Resumo

A contínua dependência de combustíveis fósseis e os desafios ambientais associados têm impulsionado o desenvolvimento de fontes de energia renováveis, com as biorrefinarias emergindo como uma solução proeminente para converter biomassa em combustíveis valiosos e produtos químicos de plataforma. Entre estas, a produção de furfural, derivada de açúcares C5 em materiais lignocelulósicos, possui particular importância. Combustíveis e intermediários de hidrocarbonetos de cadeia estendida, como 4-(2-furil)-3-buten-2-ona (FAc, C8) e 1,4-pentadieno-3-ona-1,5-di-2 furanil (F2Ac, C13), são sintetizados via condensação aldólica entre furfural e acetona, seguida por processos de hidrogenação e hidrodesoxigenação. Este estudo enfatiza o papel crítico dos catalisadores, particularmente os heterogêneos, no aumento da eficiência e seletividade dessas reações. Catalisadores sólidos, em comparação com seus equivalentes homogêneos, oferecem vantagens substanciais, incluindo facilidade de recuperação, reutilização e maior sustentabilidade. Avanços em técnicas analíticas, como cromatografia gasosa-espectrometria de massa (GC-MS) e outros métodos de última geração, têm sido fundamentais para refinar a caracterização de catalisadores heterogêneos, garantindo melhor qualidade do produto e otimização do processo. Além disso, o estudo explora metodologias de ponta para caracterização de catalisadores, utilizando ferramentas como microscopia eletrônica de varredura por emissão de campo (FESEM), espectroscopia de fotoelétrons de raios X (XPS), espectroscopia no infravermelho com transformada de Fourier (FTIR) e espectroscopia de emissão óptica com plasma acoplado indutivamente (ICP-OES) para obter insights quantitativos e qualitativos precisos. Esta revisão fornece uma análise detalhada da integração dessas tecnologias na produção de biocombustíveis, destacando o papel crítico de catalisadores inovadores — particularmente sistemas bifuncionais sob condições controladas — e o desenvolvimento de rotas de conversão otimizadas. Essas estratégias são essenciais para avançar a eficiência industrial, melhorar a seletividade do processo e contribuir para a sustentabilidade do setor de energia.

Palavras-chave: Biocombustíveis de aviação. Catálise heterogênea. Condensação aldólica

1. Introduction

Consumption of non-renewable resources, such as oil, natural gas and coal accompanied the development achieved in the last century. Petroleum is being used at a rate that exceeds the natural replenishment rate, highlighting its characteristics as an exhaustible and non-renewable resource. Recognizing the challenges associated with fossil fuels, as well as society's persistent dependence on their use, the urgency of developing sustainable energy resources that can effectively replace fossil fuels is highlighted in global efforts. Renewable alternatives such as biomass and wind energy are identified as meeting these criteria. In contrast to non-renewable fuels, these resources are abundant and support the development of technologies to reduce carbon emissions, helping to mitigate the effects of global warming (Serrano-Ruiz *et al*., 2011).

Biomass is a plentiful renewable material, an abundant renewable resource, capable of generating energy and providing biomaterials to meet society's needs. Continuous advances in biotechnology and chemical processes are contributing to the development of biorefineries, a new concept that aims to use biomass to produce platform chemicals, valuable for the development of alternative fuels. The integration of agroenergy crops is seen as an opportunity for sustainable development in bioenergy and biomaterials, signaling a paradigm shift in production. Highlighting a crucial stage in the construction of a sustainable industrial and social system, promoting the reduction of greenhouse gas emissions (Ragauskas *et al*., 2006).

The current goal is to find alternatives to oil, as two-thirds of global greenhouse gas emissions result from the combustion of petroleum-based transportation fuels. Biomass-derived transportation fuels are already being produced, with projections indicating a potential increase to 6 million barrels per day by 2030, 6% of total fuels used. A viable possibility is identified to reduce the excessive use of petroleum-derived fuels (Shylesh *et al.,* 2017).

In practice, the complete conversion of biomass energy into liquid fuels faces challenges due to its low overall thermal conversion efficiency, thus requiring technological improvements aimed at maximizing the energy and economic efficiency of the processes, mainly through process integration and optimization studies. The manufacturing of liquid fuels derived from biomass involves the removal of oxygen, which results in an increase in the density and calorific value of the fuel. However, it is considered desirable to leave some oxygen in the fuel to improve the combustion characteristics (Huber *et al*., 2006). Liquid biofuels, in contrast to petroleum-based fuels, are seen as carbon neutral because the $CO₂$ generated in their use is balanced by the subsequent growth of biomass (Chu *et al.,* 2012).

The success of biorefineries lies in understanding effective processes to convert diverse chemical components into biomass-derived constituents despite substantial efficiency advances over the past 50 years, biorefining is still in its early stages of development. Establishing a bio-based industry involves evaluating various conversion technologies, including enzymatic, catalytic and thermochemical processes (Chheda *et al.,* 2007). To be viable for the Transport sector, fuels require specific physical attributes that facilitate efficient distribution, storage and combustion, therefore, they will be efficient substitutes for current petroleum-derived fuels (Román-Leshkov *et al.,* 2007).

It is therefore essential to ensure an abundant supply and to efficiently use underutilized resources, such as residues from agriculture, forests, and non-edible crops. These organic materials (cellulose, lignin, and hemicellulose) are converted by acid hydrolysis into C-5 and C-6 monomers. The sugar fractions undergo acid dehydration to produce furanic compounds, such as furfural (C5) and 5-hydroxymethylfurfural (HMF or C6). Therefore, they are fundamental for the manufacture of biofuels and biorefineries in the future, as they can be transformed into larger organic molecules through aldol condensation with other carbonyl molecules, forming C-C bonds (Desai *et al.,* 2019).

Well-applied examples of this technique include the condensation reaction between furfural and acetone, generating 4-(2-furyl)-3-buten-2-one (FAc) as the main product. This is followed by the hydrogenation and hydrodeoxygenation of this compound in the next step, resulting in the formation of C8 diols, effective in creating different types of biofuels. Catalysts play an essential role in accelerating the process, simplifying the conversion and reducing the number of distinct catalytic steps (Ramos *et al.,* 2018).

This review aims to present a detailed overview of the use of aldol condensation between furfural and acetone in the production of biofuels. Through the topics covered in this review, it is expected to understand the stages of the biofuel production process and the reactions involved (aldol condensation, hydrogenation, hydrodeoxygenation), leading to new approaches in the process. Likewise, demonstrate the importance of developing new catalysts and precursors to achieve higher levels of conversion and precise selectivity results, obtaining the desired final product.

The review was conducted in Scopus, Web of Science, and ScienceDirect, focusing on peerreviewed articles published in English or Portuguese. Inclusion criteria prioritized high citation counts, journal impact, and alignment with the themes of "furfural," "aldol condensation reaction," "biofuels," "aviation," and "solid catalysts." Filters were applied to emphasize recent studies and contributions to catalytic processes and biofuel advancements.

The topics covered were organized with a focus on the processes relevant to biofuel production, highlighting the reaction route for the synthesis of the precursors 4-(2-furyl)-3-buten-2 one (FAc) and 1,4-pentadien-3-one-1,5-di-2-furanyl (F2Ac), through the aldol condensation reaction, as well as the subsequent steps for obtaining renewable fuels. This systematic approach allowed compiling the most significant advances in the area, presenting a clear and structured view of the state of the art on the subject.

2. Aldolic condensation reaction

2.1 Furfural

Furfural is mainly obtained from C5 sugars, especially through the hydrolysis and dehydration of xylose from lignocellulosic materials (Figure 1). Furfural production benefits from the significant pentosan content in such feedstocks. Lignocellulose, derived from sources such as sugarcane bagasse, serves as an abundant, economical, and sustainable resource for furfural production (Lange *et al.,* 2012).

According to Mariscal *et al.* (2016), furfural $(C_5H_4O_2)$ is a chemical compound identified by having a furan ring with heteroaromatic properties and an aldehyde-type functional group. In the industrial context, there is a continuous effort to promote economically viable and environmentally responsible alternatives for obtaining furfuraldehyde to reduce operational costs for the production of its derivatives. Furfural, in addition to being a versatile compound due to its aromatic and polar properties, is also considered a promising chemical platform, representing a significant opportunity for future advances in the area of fuels and valuable chemicals. However, it has not been fully explored.

Derived from trimers of 2-methyl furfural

Figure 1 - Furfural platform for biofuels.

Furfural is a promising alternative to crude oil-based materials and is used in the production of various everyday products, such as lubricants and plastics, as well as being widely used in the production of resins. It also serves as a precursor to chemical products such as furfuryl alcohol and tetrahydrofurfuryl alcohol, compounds that have high added value (Gürbüz *et al.,* 2013). The application of furans, such as furfural, as precursors in the manufacture of hydrocarbon-based liquid fuels, through the aldol condensation reaction, offers the possibility of generating linear alkanes with molecular weights suitable for diesel or aviation fuel (Alonso *et al.,* 2010).

2.2 Aldol-condensation reaction

According to Alonso *et al*. (2010), aldol condensation is essential to increase the molecular weight of hydrocarbons. To obtain fuels with high yields, it requires hydrogenation, which is realized with high hydrogen pressures and bifunctional catalysts. Condensation products derived from biomass, especially furfural or HMF, contain a high oxygen content, requiring substantial hydrogen input for alternative fuels production. Aldol condensation allows the targeted production of linear alkanes, used for the production of aviation fuel, being favored by the few carbon branches, thus obtaining more selective productions unlike hydrogen-neutral methods, such as oligomerization.

Despite having an aldehyde group, HMF and furfural do not have an α-H atom, which prevents them from self-condensing. However, it is possible for these compounds to condense with other molecules such as acetone, dihydroxyacetone or glyceraldehyde, which are capable of forming carbanion species (Huber *et al.,* 2005).

Normally, this reaction takes place with the aid of basic catalysts, where an α -hydrogen is abstracted from a nucleophile. This step generates an intermediate species of carbanion, which then attacks the carbonyl of HMF or furfural, facilitating the formation of carbon-carbon bonds between two carbonyl-containing compounds. Subsequent steps such as hydrogenation and hydrodeoxygenation generate additional carbonyl compounds, leading to heavier alkanes. This process serves as an intermediate step after aldol condensation in an aqueous medium, using a flow reactor with bifunctional catalysts (Chheda and Dumesic, 2007).

According to He *et al.* (2021) aldol condensation is favored by acidic or basic catalysts under flexible conditions (such as reaction temperatures $\leq 180^{\circ}$ C), lengthens carbon chains and reduces O/C ratios, resulting in carbon precursors. Current research is focused on the development of heterogeneous catalysts for aldol condensation of furanic derivatives. This change is driven by the benefits of its use, including easy reuse and low corrosivity. The resulting condensation products are transformed into hydrocarbon fuels similar to mineral fuels through hydrogenation and hydrodeoxygenation.

2.3 Hydrogenation and hydrodeoxygenation

The standards for aviation fuels require the main components to be alkanes with a chain length ≥C10. This includes branched or cyclic alkanes (C10-C12) and alkanes with several branches (C13- C16), which are considered suitable for aviation engines. As the main constituents of diesel fuel or jet fuel must be alkanes between C10-C20, the process of converting lignocellulose components into diesel fuel or jet fuel requires elongation of the chains and removal of oxygen for conversion into fuels (Nakagawa *et al.,* 2019).

In the context of biomass, its platform molecules, which are predominantly characterized by being highly oxygenated, require oxygen removal reactions to be transformed into liquid hydrocarbon fuels. Methods such as dehydration, hydrogenolysis, hydrogenation, decarbonylation and decarboxylation are employed to eliminate oxygen functionalities. Further hydrogenation of aldolic products generates longer-chain alkanes of better quality (De *et al.,* 2015).

Fusion of furfural with acetone results in the formation of significant organic compounds, 4- (2-furyl)-3-buten-2-one (FAc or C8) and 1,4-pentadien-3-one-1,5-di-2-furanyl (F2Ac, F-Ac-F or C13). These compounds, which are not very soluble in water, require subsequent hydrogenation and hydrodeoxygenation reactions to generate longer-chain alkanes of better quality, as well as increasing the solubility of the products (Huber *et al*., 2006).

Low-temperature hydrogenation is used to stabilize the F-Ac-F dimer, generating hydrogenated F-Ac-F (H-FAF) to prevent polymerization due to its unsaturated bonds. This step also prevents unwanted blockage of the reactor during subsequent hydrodeoxygenation (Xing *et al.,* 2010).

High-temperature hydrodeoxygenation involves the reaction of products with hydrogen, removing oxygen in the form of water, generating saturated hydrocarbons such as Tridecane, ideal for aviation fuels, without loss of carbon. It is important to note that hydrodeoxygenation uses a bifunctional catalyst with metal and acid sites. The successive hydrogenation and hydrodeoxygenation phases play an essential role in the saturation of carbon-carbon double bonds (Olcay *et al.,* 2013).

2.4 Reaction chemistry

Huber *et al.* (2005) suggested, through the aldol condensation reaction, a procedure with three consecutive steps to produce these fuels from furfural and acetone. The steps are: (i) deprotonation, where the basic sites remove α -H from the adsorbed ketones, forming a type of surface enolate; (ii) nucleophilic addition reaction between the enolate and the carbonyl carbon of the adsorbed ketones, resulting in the formation of an aldol adduct; and (iii) reprotonation, where the oxygen atom accepts the proton transferred back from the surface, forming an intermediate, 4- (2-furyl)-4-hydroxy-2-butanone (F-OH). This intermediate then undergoes dehydration to form the unsaturated product 4-(2-furyl)-3-buten-2-one (FAc) or C8. Reacting with a second furfural molecule, the products can form 1,4-pentadien-3-one-1,5-di-2-furanyl (F-Ac-F or F2Ac) or C13, via α-H abstraction from FAc. It is noted that the concentration of basic sites and the strength of the basicity distribution play crucial roles in this reaction (Kong *et al.,* 2021; He *et al.,* 2021). Figure 2 shows the reaction mechanism.

Figure 2 - Mechanism of the aldol condensation reaction

After the aldol condensation step, the resulting products are subjected to subsequent reactions, namely low-temperature hydrogenation and high-temperature hydrodeoxygenation (Olcay *et al.,* 2013). The subsequent reactions are observed in the Figure 3:

7H₂

Low-temperature hydrogenation

1,4-pentadien-3-one-1,5-di-2-furanyl (F-Ac-F or F2Ac) or C13

Hydrogenated F-Ac-F (H-FAF)

High-temperature hydrodeoxygenation

Figure 3 - Reaction hydrogenation and hydrodeoxygenation.

Initially, aldol condensation was performed using homogeneous catalysts, such as aqueous NaOH solutions. However, this method faced several problems, such as challenges in recovery and corrosion (Faba *et al.,* 2012). Therefore, preliminary studies on the action and effectiveness of new catalysts are essential to make the furfural-acetone aldol condensation reaction viable and profitable in the long term, since the optimization of the transformation of reactants into products is fully related to the efficiency of the catalyst.

3. Catalysts and reaction conditions

The well-known aldol condensation reaction between aldehydes and ketones takes place in the presence of acidic or basic catalysts. However, in industrial context, these processes generate large quantities of wastewater, requiring additional treatment and incurring high costs. To mitigate this problem, the replacement of liquid catalysts with solid ones has attracted growing interest. Solid catalysts have environmental and process benefits, standing out for their ease of separation and reuse in multiple reaction cycles, which differentiates them from homogeneous catalysts (Arhzaf *et al*., 2021).

In addition, solid catalysts with acid-base properties are standing out as a promising alternative route in the aldol condensation of furfural-acetone, presenting themselves as a viable and more efficient option (Nguyen *et al.,* 2016).

Thus, the search for optimized production on an industrial scale, with greater conversion of furfuraldehyde, represents a current challenge that has driven the exploration of several heterogeneous catalysts. Table 1 summarizes some recent studies investigating various catalysts and operating conditions.

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1 **Table 1 – Different types of catalysts currently developed.**

 $\begin{array}{c} 2 \\ 3 \end{array}$

Note: P= pressure, T= temperature. Conversion (%) to furfural

4 5

Kong *et al.* (2021) aiming to produce alternative fuels, addressed the synthesis of ZnO-Al₂O₃ catalysts promoted by $La_2O_2CO_3$ through furfural-acetone aldol condensation. These catalysts were compared with La_2O_3 and $ZnO-Al_2O_3$ catalysts with different concentrations and molar ratios. The authors emphasized that the $La_2O_2CO_3$ -promoted catalysts synthesized contained greater quantities of medium-strength basic sites, which were fundamental to the reaction, rather than strong basic sites. This property led to a furfural conversion rate of 93.9% at 100 °C, with 43.1% selectivity for C8 products and 56.9% selectivity for C13 products.

Korolova *et al.* (2023) examined the kinetics of the aldol condensation between furfural and acetone, evaluating the performance of rehydrated MgAl hydrotalcite in the reaction. They investigated temperature, furfural/acetone ratio, catalyst concentration/weight, and their influence on the reaction. The experiments showed that increasing the reaction temperature, catalyst loading, or acetone in the mixture increased furfural conversion after 180 min. In contrast, lower temperatures or reduced catalyst loading caused greater catalyst deactivation, especially at low furfural consumption rates. The authors report that this deactivation started through two parallel processes at high furfural concentrations: furfural-acetone aldol condensation and the Cannizzaro reaction forming furfuryl alcohol and furfurylic acid. Experimentally calculated kinetic curves confirmed that changes in the variables significantly impacted the reaction rate.

The work of Korolova *et al*. (2023) is similar to the methodologies and catalysts of Hora *et al.* (2014), However, the conversion of furfural was relatively higher in Hora's work, which can be justified by the difference in temperature, amount of catalyst and molar ratio of the reactants, since these are fundamental factors in this reaction as reported by Korolova *et al*. (2023).

Faria *et al.* (2018) investigated organic catalysts with different basicity levels, such as Piperazine (PIP), 3-Aminopropyltriethoxysilane (APTES), and 1,5,7-Triazabicyclo[4.4.0]dec-5-ene (TBD), in the aldol condensation reaction. The study highlighted TBD as the most efficient catalyst, promoting a significant transformation of furfural and exhibiting enhanced selectivity for the FAc compound over PIP and APTES. They reported that this performance is related to the higher basicity of TBD compared to other amines. The experimental results showed the possibility of obtaining a complete conversion of furfural at 75°C in 1 hour.

Fang *et al*. (2020) investigated the aldol condensation reaction between furfural and acetone using the Ca/ZSM-5 catalyst, highlighting the influence of calcination temperature and water dosage on the yield of C8 and C13 adducts. The catalyst calcined at 700 °C showed the best performance, achieving maximum combined yield of C8 and C13 (87.4 mol%), due to the larger specific surface area. Water, although non-reactive, was shown to be essential for the reaction, promoting the conversion of furfural and acetone and maximizing the product yield at 93.3 mol% with a dosage of 3 g (28 wt%). While the selectivity to C8 increased with increasing water dosage, the formation of C13 was favored at lower concentrations, being progressively inhibited at higher dosages.

Parejas *et al*. (2019) investigated mixed oxides of Al-Mg as catalysts for the aldol condensation between furfural and acetone, highlighting the impact of synthetic variables and water on catalytic performance. The reference catalyst, obtained without surfactant, showed that higher temperatures increased the conversion without affecting the selectivity for F_2 Ac. Rehydration of the catalysts reduced their activity, while the addition of water to the reaction medium increased the conversion but decreased the selectivity. Catalysts synthesized with surfactant Pluronic 123 and microwaves showed higher acidity and larger pore sizes, favoring higher F2Ac yields in prolonged reactions.

The studies by Parejas *et al*. (2019) and Fang *et al*. (2020) highlight the crucial role of water in aldol condensation and product selectivity. Both observed that increasing the amount of water improves the conversion of the reactants and increases the selectivity for the C8 adduct, but significantly reduces the selectivity for the product of greatest interest, F_2Ac .

Arumugam *et al*. (2023) investigated the effect of basicity of potassium-modified MgAl-SBA-15 bifunctional catalysts on the aldol condensation of furfural and acetone under mild conditions. The catalyst with the highest basic loading $(0.27 \text{ mmol g}^{-1})$ achieved 83% furfural conversion and 99% overall selectivity for C8 and C13 products, including FAc-OH, FAc, and F₂Ac. Despite mesopore blocking and reduced accessibility to acidic sites at high potassium loadings, the catalyst demonstrated good regenerability and was reusable for multiple cycles. Despite partial mesopore blocking at high potassium loadings, the material showed good regenerability and reusability. The study reinforces the importance of basicity in catalytic performance under mild conditions.

Like Fang *et al*. (2020), Kikhtvanin *et al*. (2014) investigated the influence of the physicochemical properties of zeolites on catalytic performance in the aldol condensation of furfural and acetone. MFI (medium pore) and MOR (monodimensional wide pore) zeolites showed low conversion \langle 10% at 100 °C). On the other hand, wide-pore zeolites with a three-dimensional structure, in H form, achieved up to 30% conversion in 2 hours. Catalytic activity was affected by characteristics such as crystal structure, acidity and textural properties. The formation of carbonaceous deposits reduced activity, but this was recovered after calcination (>500 °C). As the reaction conditions were different, the best results are those presented by Fang *et al.* (2020).

Arhzaf *et al*. (2021) investigated mixed Mg-Al oxides (MgXAl-O) as catalysts in the aldol condensation of furfural and acetone, evaluating the impact of the Mg/Al ratio, temperature and reaction time. The Mg₃.5Al-O catalyst, with a higher density of basic sites, was the most efficient, presenting a lower apparent activation energy compared to NaOH (55.8 kJ/mol), evidencing greater ease of reaction with heterogeneous catalysis. Temperature and reaction time significantly influenced the conversion and selectivity.

In this way, solid catalysts offer greater efficiency, in addition to facilitating separation and reuse, overcoming the challenges associated with homogeneous catalysts. Therefore, it is essential to analyze different reaction media and reaction parameters to identify the best operational scenarios for the production of renewable fuels, with the aim of optimizing processes and achieving high conversion rates. Likewise, a deeper understanding of current catalyst and product characterization techniques is essential to understand the catalytic action and the formation of the products of interest.

4. Characterization techniques

Catalyst and products characterization is essential to understanding the impact of catalyst type on product formation and increasing the desired selectivity in the chemical reaction.

4.1 Characterization of catalysts

Brunauer-Emmett-Teller (BET), Mass spectrometer (MS) and Energy Dispersive X-ray Spectroscopy (EDX) are traditionally used in catalyst characterization. Recent studies have used these traditional techniques coupled with advanced techniques, such as Field emission scanning electron microscopy (FESEM), X-ray photoelectron spectroscopy (XPS) and Optical Emission Spectroscopy (OES) to obtain high resolution of samples and identification of multicomponents by analysis.

Table 2 summarizes the characterization of the catalysts used for furfural conversion. It is worth noting that the study by Ramos *et al.* (2018) emphasized the chemical composition, surface properties, and morphology of the catalysts, while Kong *et al.* (2021) focused on the detailed structural analysis, surface reactivity, and spectroscopic characteristics of the catalysts. Both studies used several techniques, offering broad knowledge of the material properties.

In a study carried out by Sádaba *et al.* (2011), an inductively coupled plasma mass spectrometer (ICP-MS) was used to examine the chemical composition of solids, unlike Ramos, who used the ICP technique coupled to Induced Plasma Optical Emission Spectroscopy (ICP-OES).

Authors	Catalyst	Catalyst
		characterization
		method
Ramos et al. (2018)	Ni and Cu	ICP-OES; FESEM-
	catalysts	EDX; BET; NH_3 -
	supported on	TPD; HAADF-
	$SBA-15$	STEM
Kong et al. (2021)	$ZnO-Al2O3$ catalysts promoted by La ₂ O ₂ CO ₃	XRD ; TPD-CO ₂ ; XPS; IR; TG, BET
Sádaba et al. (2011)	Mg-Zr mixed oxides	ICP-MS; XRD;XPS; HRTEM; EDX
Shao et al. (2023)	Mg-Al-O/AC and Ni/Mg-Zr-O/AC	TPD-CO ₂ , XRD
Korolova et al. (2024)	MOs of MgAl and HTCs	AAS; BET; XRD; SEM; TGA; TPD- CO ₂
Arhzaf et al., (2024)	HTCs intercalated with ion (HTNO3), (HT- $CO3$) and (HT- CH3COO)	XRD; FT-IR; BET

Table 2 - Characterization of catalysts used to furfural conversion.

Note: NH3-TPD: Ammonia temperature programmed desorption. HAADF-STEM: High angle annular dark field scanning transmission electron microscopy. TPD-CO₂: Temperature programmed desorption of carbon dioxide. IR: Infrared spectroscopy. TG: Thermogravimetric analysis. HRTEM: High-resolution transmission electron microscopy. SEM: Scanning electron microscopy. TGA: Thermogravimetric Analysis. XRD: X-ray diffraction

According to Adachi *et al.* (2004), FESEM-EDX refers to the coupling of the two instrumental analysis techniques. By combining the two techniques it is possible to examine individual particles at high resolution, allowing not only the analysis of isolated particles, but also facilitating the determination of the elemental composition in specific regions of the material.

As reported by Douvris *et al.* (2023), the widespread use of Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES) for elemental analysis is due to its ability to measure multiple elements simultaneously. Likewise, X-ray diffraction spectroscopy (XRD) is an extremely efficient method used to identify materials and determine their crystallographic phase. A privilege of this technique is the ability to handle small amounts of material, often down to less than a gram, making it versatile for various research applications (Bergslien, 2022).

As mentioned by Ghobeira *et al.* (2022), XPS is a sophisticated analytical technique used to qualitatively and quantitatively analyze the chemical states in the upper surface layers of materials, within a few nanometers. This method accurately analyzes the element composition of the surface, being able to distinguish different chemical bonds existing in the surface.

Atomic absorption spectrometry (AAS) is widely recognized as a sensitive and effective analytical technique for the determination of metals in various samples, reaching detection levels in the picogram range. Its use is consolidated in research laboratories and in sectors such as food, environmental, pharmaceutical and petroleum, due to its ability to be applied in a wide variety of samples and analytical contexts (Butcher, 2005; Ferreira *et al.,* 2018).

Similarly, Fourier transform infrared spectroscopy (FTIR) is being widely used for catalyst characterization. As mentioned by Jafari *et al.* (2017) and Nirmala *et al.* (2022), the technique is based on the absorption of light in the infrared region of the electromagnetic spectrum by the molecules of the sample, generating a spectrum that acts as a fingerprint. The vibration frequencies of the atomic bonds are represented as peaks in the spectrum, allowing detailed identification of the molecular composition of the analyzed material. This technique allows the identification and quantification of the different components present in the sample in a precise manner.

The BET technique is used to determine the specific surface area of materials, especially porous solids such as zeolites, catalysts, adsorbents and materials with nanometer-scale porosity. Alternative methods for analyzing the surface area and volume of nanoporous materials, such as non-local density functional theory (NLDFT) and Dubinin's pore volume filling theory (DR), have been widely used due to their effectiveness in differentiating micro-, meso- and macroporous structures. NLDFT is available in commercial software systems and internationally standardized (ISO 15901-3), while Dubinin's approach is particularly useful for materials with a uniform distribution of micropores. The inclusion of these methodologies complements the traditional analysis carried out by BET, extending the tools for detailed characterization of catalysts and other porous solids (Thommes *et al.,* 2015).

Surface acidity, crucial in catalysis, is evaluated using Temperature-Programmed Desorption with Ammonia (NH₃-TPD), a technique specifically designed to investigate surface acidity in solid materials, especially catalysts and porous substances. The study of Ramos *et al.* (2018) employed HAADF-STEM for high-resolution observations, delving into the morphology of materials.

4.2 Product characterization

The technique traditionally used by researchers to analyze the products of the aldol reaction between furfural and acetone is gas chromatography (GC). However, more advanced techniques are being coupled to GC for more accurate quantitative and qualitative results. Table 3 presents some techniques for characterizing the products.

Table 3 – Techniques for product characterization.

Note: FAc: 4-(2-furyl) -3-buten-2-one (or C8). F2Ac: 1,4-pentadien-3-one-1,5-di-2-furanyl (or C13). GC: Gas chromatography. GC-FID: Gas chromatography-flame ionization detector. GC-MS: Gas Chromatography-mass spectrometry.

In studies conducted by Ramos *et al.* (2018), the products were characterized by gas chromatography (GC). In addition to the previous analysis, Kong *et al* (2021) used GC coupled to a flame ionization detector (FID) and capillary column, as this technique is commonly used for the analysis of organic compounds, providing information on the identity and concentration of the products.

Jing *et al*. (2019) used the same technique as Kong *et al*. (2021) for quantitative analysis. However, the author chose to use gas chromatography-mass spectrometry (GC-MS) for quantitative analysis, as it is possible to identify compounds based on their mass-charge relationship and compare them with a database of known spectra, allowing the identification of the components present in the sample. Similar to other authors, Kikhtyanin *et al*. (2018) characterize the reaction products using GC with a flame ionization detector and capillary column (HP5).

As noted, these methods are widely used to characterize the aldol condensation products furfural-acetone. They are constantly reproduced by several authors, demonstrating the effectiveness of these characterization methods.

5. Challenges and future perspectives

To face the challenges and future perspectives related to aldol condensation between furfuryl and acetone, it is essential to analyze the complexities inherent to this reaction process. Advancement in this area presents promising opportunities, but also faces obstacles that require indepth investigations and innovative strategies to overcome.

5.1 Furfural

Lignocellulosic biomass is a promising feedstock for sustainable biofuel production. The major challenge is pretreatment and hydrolysis, which are major obstacles to the economic viability of this strategy. Therefore, research is focused on optimizing these processes to improve biomass efficiency in biofuel production (Alonso *et al.,* 2010). High biomass conversion is directly related to efficient furfural production, especially in lignocellulosic biorefineries.

However, the commercial viability of furfural faces challenges due to pricing issues and market instability. Reducing the current price of furfural requires technological advances, as conversion technologies involve energy-intensive distillation steps. Competitive industrial-scale production requires innovations that can process diverse lignocellulosic biomass sources, which have varying compositions and impurities. This will ensure a stable supply of feedstock, increase process flexibility, and reduce production costs. Although furfural industries exist, they are underdeveloped and require significant advances to gain prominence (Mariscal *et al.,* 2016).

5.2 Catalysts

To improve catalytic processes, strategies such as refining formulations, replacing noble metals with more economical alternatives, and introducing gas co-feeds are crucial. Although the causes of catalyst deactivation are known and regeneration procedures exist, many reactions still lack a complete understanding of the deterioration processes. Detailed information is essential to design more stable catalysts, prevent or reduce deactivation, and develop reactivation protocols. However, long-term research under real reaction conditions is often lacking. Evaluating catalyst stability using real furfural solutions, directly from biomass and with authentic impurities, can provide valuable insights, especially with non-distilled furfural (Mariscal *et al.,* 2016).

Heterogeneous-based catalysts are commonly used in the condensation of aldol with furanic compounds, but due to the accumulation of heavy compounds, they suffer deactivation. To address this, novel deposition-resistant catalysts are needed, achievable through optimization of structures and surface properties. On the other hand, research about heterogeneous acid catalysts for aldol condensation with furanic compounds is limited compared to the extensive study of base catalysts (He *et al.,* 2021). Thus, new catalysts are being developed for application in aldol condensation reactions, with the aim of producing renewable fuels from furfural and other raw materials of biological origin.

Zhang *et al.* (2024) highlight the importance of aldol condensation and hydroxyalkylation/alkylation reaction in the production of high-density fuels from biomass, using $Fe₃O₄@SiO₂@F-SO₃H$ catalyst, with superacidic and hydrophobic characteristics for high-density biofuels. The aldol self-condensation of cyclohexanone, studied by the authors, resulted in a biofuel with a density of 0.887 g/mL, 13.7% higher than aviation kerosene. The cross-condensation between cyclohexanone and cyclopentanone produced a hydrocarbon with high density (0.881 g/mL) and a very low freezing point (<-75 °C), due to the formation of asymmetric cyclopentylcyclohexane.

Li *et al.* (2025) synthesized metal oxide catalysts by the sol-gel method and investigated their performance in the production of aviation biofuel precursors from biomass-derived ketones, such as cyclopentanone, cyclohexanone, and 2-pentanone. The results demonstrated that copper-doped metal oxide catalysts (Cu/Al_2O_3) showed excellent efficiency in the catalytic conversion of these ketones into biofuel precursors, highlighting their potential for applications in sustainable aviation.

Raguindin *et al.* (2025) developed an integrated approach to produce 1-octanol, a thirdgeneration biofuel, from biomass-derived furfural and acetone. The two-step one-pot process achieved a yield of 64.8%, combining high energy efficiency and productivity. The multifunctional catalytic system, composed of $Ni/Al₂O₃-ZrO₂$ and a solid acid additive (KCC-1-APSO₃H), proved to be a sustainable and economical alternative to traditional catalysts, standing out for its selectivity and industrial viability.

De Paz Carmona *et al.* (2024) analyzed commercial sulfide catalysts (Mo/Al₂O₃, NiMo/Al₂O₃, $CoMo/Al₂O₃$ and $CoMo/TiO₂$) for the co-hydroprocessing of aldol condensation adducts with atmospheric gas oil, highlighting their application in biomass-derived biofuels. Noble metal catalysts, such as Pt/Al_2O_3 and Pd/Al_2O_3 , demonstrated high efficiency in the conversion of adducts into alkanes (n-octane and n-tridecane) under mild conditions. The hydrotreatment of the adducts, due to their furan-rich composition, resulted in higher production of n-octane and n-tridecane, especially in mixtures with specific proportions. ATR-IR analyses confirmed deoxygenation as the main catalytic pathway promoted by $CoMo/Al_2O_3$ and $CoMo/TiO_2$.

The references presented demonstrate significant progress in the development of catalysts for the production of renewable fuels. From compounds such as furfural and acetone, different types of fuels can be obtained through aldol condensation, reinforcing the importance of global efforts to create efficient alternatives to petroleum derivatives.

5.3 Separation and Reaction kinetics

Obtaining a highly selective synthesis of the final product in heterogeneous catalysis processes is essential and requires both the improvement of catalyst structures and the control of the distribution of active sites. An in-depth study of the separation and purification steps for target products is essential in order to align the process with practical applications. In general, condensation adducts are separated by extraction and/or distillation, however these steps depend on the boiling point and polarity of the products and solvents. The appropriate selection of solvents is essential to ensure effective separation. The efficiency and economic viability of the process are crucial and affect the practical use of the separation techniques developed (He *et al.,* 2021).

Similarly, the importance of understanding the mechanisms involved in bio-based chemical conversions is highlighted in order to improve conversion processes and guide experimental research. As the complexity of a reaction system increases, especially in real systems with multiple side reactions, the interactions between different reactants can deviate significantly from the reaction pathway observed in model compounds. Therefore, there is a great need for further studies to gain insights into catalytic mechanisms The analysis of kinetics plays an essential role in the optimization of conversion processes and in the design of pilot-scale reactors. In conclusion, a deeper understanding of the processes as well as the mechanisms involved in the reactions is essential, as it provides a solid basis for interpreting the behavior of the systems under real conditions (Wu *et al.,* 2016).

5.4 New means of reaction

Biomass, as a renewable resource, has great potential to meet the growing energy demand in a sustainable manner. Its use enables the development of technological alternatives capable of replacing energy sources derived from non-renewable resources, such as oil. In this scenario, the application of innovative methods to convert residual biomass into fuels suitable for aviation stands out as a promising solution. With the reduction of fossil fuel reserves, the conversion of aldehydes and ketones into aviation fuels has aroused significant interest in the scientific community (Shao *et al.,* 2023).

Currently, several reaction methodologies are being developed to maximize product yield and selectivity. The most common approaches focus on the synthesis of new catalysts and supports, in addition to the choice of suitable organic solvents for mass transfer in the proposed reactions. Among the main proposals, heterogeneous base catalysts, acids and bifunctional acid-base catalysts stand out, aiming to maximize the production of intermediates and aviation fuels (Hora *et al.,* 2014; Meemanah *et al*., 2023). Another increasingly discussed proposal is the integration of the aldol condensation and hydrogenation stages, carried out in a single piece of equipment, promoting greater operational efficiency and reducing the energy requirements of the process (Shao *et al.,* 2023).

In addition to aldol condensation, several other reactions have been explored to promote C-C coupling in the production of renewable fuels. Among them, the Diels–Alder reactions, hydroxyalkylation/alkylation, oligomerization, Michael addition, reductive coupling and Simmons– Smith cyclopropanation stand out. These reactions are often followed by hydrodeoxygenation (HDO) steps to obtain the final products (Fang *et al*., 2024).

The catalytic conversion of bio-based chemicals into the aqueous phase presents environmental and economic opportunities for generating high-quality liquid fuels from biomass. Despite the potential, aqueous-phase processing of biobased chemicals poses challenges, and developments in this field are ongoing (Wu *et al.,* 2016).

5.5 Practical applications

The practical viability of high-yield fuels obtained from furan compounds is directly linked to the large-scale, highly efficient and economically viable production of these compounds. Despite numerous studies on synthesizing furanic compounds from biomass, the persistent challenge of costeffective large-scale production remains a significant constraint for potential applications. This highlights the urgency of intensifying research aimed at the synthesis of low-cost furan compounds from lignocelluloses on a larger scale. In the path to converting bio-based furan compounds into high-quality fuels, it is essential to go beyond the aldol condensation approach. Focus should be placed on other efficient carbon chain extension strategies, carefully designed to improve the overall efficiency of the biofuel production process (He *et al.,* 2021).

5.6 Economic Analysis

In addition to developing crucial scientific and technical knowledge, it is necessary to assess commercial viability, encompassing economic and environmental considerations. However, carrying out these analyses requires meticulous care, due to the complexity of the factors involved, such as the logistics of supplying raw materials and transporting products, the quality and volume of competition, price variation forecasts, future fluctuations and possible synergies with other sectors, as well as other relevant variables (Mariscal *et al.,* 2016).

As highlighted by Hakeem *et al*. (2022), techno-economic analysis has become an increasingly relevant tool in assessing the economic viability of biochemical processes that convert biomass into bioproducts, especially in commercial development phases. The most widely used methodology involves software to estimate costs and revenues, helping to predict cash flows and return on investment. The competitiveness of biorefineries can be improved by the co-production of biofuels and high-value chemicals.

Olcay *et al*. (2018) conducted a comprehensive economic analysis of biofuel and chemical production, considering various biomass pretreatment methods. The study evaluated process yields, product portfolios, hydrogen sources, and historical fluctuations in essential commodity prices. Simulations were performed for seven biorefinery models designed to convert red maple wood into products such as furfural, hydroxymethylfurfural, jet fuels, and diesel. This work stands out by integrating techno-economic and environmental assessments, encompassing process simulation, minimum selling price calculations, and greenhouse gas emissions analysis. Fixed capital investments for the biorefineries ranged from US\$459 million to US\$473 million. Among the pretreatment methods evaluated, the use of hydrochloric acid resulted in the lowest selling price for jet fuel, estimated at US\$3.60 per gallon, based on 2018 values.

5. Conclusion

The aldol condensation between furfural and acetone presents a promising pathway for sustainable biofuel production, leveraging renewable feedstocks and innovative catalytic strategies. Advances in catalyst design, including the development of regenerable and highly selective materials, have significantly contributed to enhancing process efficiency and scalability. The optimization of reaction parameters and the integration of advanced catalytic systems underscore the potential of this approach to meet industrial demands.

Furthermore, the continued refinement of characterization techniques has deepened our understanding of catalyst structure and function, enabling the design of materials tailored for highperformance applications. By addressing key challenges such as catalyst stability, selectivity, and process sustainability, ongoing research is paving the way for the industrial deployment of aldol condensation in biofuel production.

This progress marks an important step toward reducing global reliance on fossil fuels, offering a viable alternative that aligns with the goals of a sustainable energy future and the mitigation of climate change. Further interdisciplinary efforts, integrating materials science, chemical engineering, and industrial processes, will be crucial in realizing the full potential of this transformative technology.

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