Review

Advanced Silica-Based Catalysts from Rice Husk Ash for CO_2 Fixation: Synergistic Effects of Metal Oxides and Ionic Liquids in Glycerol Carbonate Production

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Advanced Silica-Based Catalysts from Rice Husk Ash for CO_2 Fixation: Synergistic Effects of Metal Oxides and Ionic Liquids in Glycerol Carbonate Production

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Abstract

This review explores the catalytic conversion of carbon dioxide (CO_2) into glycerol carbonate (GC), positioning this pathway as a sustainable strategy that couples environmental mitigation with the valorization of surplus glycerol from biodiesel production. Glycerol carbonate maintains extensive industrial utility as a green solvent, chemical intermediate, and functional component in polymers, cosmetics, and packaging. Distinct from prior literature, this study systematically integrates the evaluation of catalysts derived from agro-industrial waste and hybrid catalytic systems, correlating their structural architectures with catalytic efficiency. The review evaluates diverse catalytic frameworks, with a primary focus on heterogeneous systems. Silica-based materials are highlighted, particularly those synthesized from rice husk ash, an abundant amorphous silica source. The sol–gel method is identified as a robust route for engineering porous matrices with high surface areas and tunable structural properties. Furthermore, the doping of silica with metal oxides, such as niobium oxide (Nb_2O_5) and nickel oxide (NiO), is discussed as a strategic approach to introduce synergistic acid–base sites and redox properties that facilitate CO_2 activation. The integration of ionic liquids into hybrid systems is also examined as a promising frontier to enhance reaction kinetics and selectivity. Finally, this review delineates the nexus between agro-industrial waste management and the reduction of greenhouse gas emissions, proposing a circular economy framework for the biodiesel value chain.

Keywords: carbon dioxide conversion; glycerol carbonate; heterogeneous catalysis; circular economy

1. Introduction

The continuous increase in carbon dioxide (CO_2) emissions, mainly resulting from the burning of fossil fuels, industrial processes, deforestation, and intensive agricultural activities, has intensified environmental impacts and the global climate crisis [1,2]. The accumulation of CO_2 in the atmosphere enhances the greenhouse effect, accelerates global warming, and is associated with extreme climate events such as glacier melting, ocean acidification, and biodiversity loss [3–5]. In this context, the development of technologies for CO_2 reduction, capture, and reuse has become a strategic priority across different industrial sectors.

At the same time, the growth in biofuel production, especially biodiesel, has resulted in a large availability of glycerol as a byproduct of oil transesterification [6,7]. In this context, both crude glycerol and purified glycerol are coproducts of the biodiesel manufacturing process; however, crude glycerol has a lower purity level, typically in the range of 60–80%, compared to refined or synthetic glycerol. This lower purity limits its application in sectors that require a high degree of specification, such as the food, pharmaceutical, and cosmetic industries [8–10]. Although biodiesel's main advantage is its lower CO_2 emissions compared to fossil fuels [11,12], the accumulation of crude

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glycerol generates economic and environmental challenges, including treatment costs and pollution risks [13].

The catalytic conversion of glycerol with CO_2 to obtain glycerol carbonate stands out as a strategy to reduce the carbon intensity of industrial processes and strengthen the biodiesel value chain [14]. It has been investigated through both direct routes between glycerol and CO_2 and indirect pathways, such as epoxidation followed by carbonation, including the application of continuous reactors [15,16].

From an industrial perspective, glycerol carbonate is notable for its multifunctional character and low toxicity, acting as a humectant, green solvent, and precursor of polyurethanes and polymers applicable to food and packaging [17–20]. Its application contributes to the replacement of fossil-based inputs and to the reduction of waste and emissions in the food supply chain. Figure 1 shows these and other diverse applications of this compound.

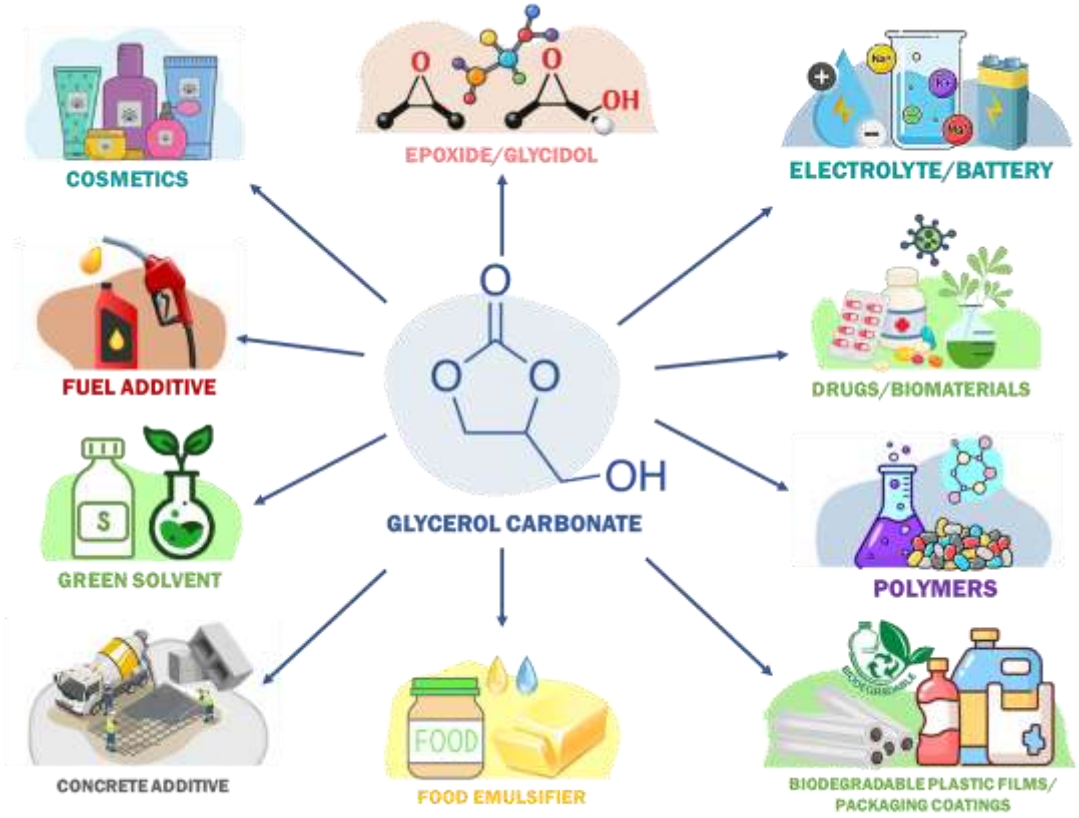

Figure 1. Applications of glycerol carbonate.

In summary, this work aims to discuss the catalytic conversion of CO_2 into glycerol carbonate, highlighting more efficient, stable, and environmentally friendly heterogeneous materials. It seeks to contribute to integration and value creation in strategic industrial sectors, strengthening a technological and sustainable approach.

Despite advances in heterogeneous catalysis, limitations such as the low solubility of CO_2 in glycerol and the thermal stability of catalysts still persist. This review emphasizes how the incorporation of ionic liquids and metal doping with niobium and nickel can overcome these barriers and optimize catalytic performance.

2. Carbon Dioxide (CO_2)

In the contemporary context, the increasing concentration of CO_2 in the atmosphere has become one of the main drivers of the global environmental crisis, largely intensified by anthropogenic

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activities. The burning of fossil fuels for energy generation, industrial processes that emit large volumes of CO_2, deforestation that reduces carbon sequestration capacity, and intensive agricultural practices significantly contribute to this atmospheric accumulation [1,2]. Excess CO_2 intensifies the greenhouse effect by trapping heat in the atmosphere, promoting a rise in the global average temperature, with particularly pronounced impacts in polar regions [3,4]. The resulting ice melt reduces Earth's albedo, increases solar radiation absorption, and reinforces global warming [5], triggering systemic effects such as extreme climate events, ocean acidification, biodiversity loss, cryosphere degradation, and environmental instabilities on a global scale.

One of the most promising uses of CO_2 is in the production of synthetic fuels, such as methanol and e-diesel [21–23]. These fuels can replace fossil-based alternatives in sectors that are difficult to decarbonize, such as aviation and maritime transport, offering a more sustainable mobility solution [24,25].

In the plastics and polymers sector, significant advances have been made in converting CO_2 into polycarbonates and polyurethanes. These materials can be used in packaging, insulating foams, and automotive components, promoting more sustainable alternatives compared to traditional petrochemicals [26–29].

Another example is the chemical fixation of CO_2, in which catalysts play a crucial role, as they enable the activation of CO_2, a thermodynamically stable and chemically inert compound, thus making its conversion into value-added products feasible [30–32]. Reactions such as cycloaddition with epoxides to form cyclic carbonates, carboxylation of organic substrates, and selective hydrogenation are examples of processes that depend on efficient catalysts to occur under mild temperature and pressure conditions [15,16].

Among the most explored catalysts for this purpose are materials functionalized with ionic liquids (ILs) and ionic solids (ISs), which exhibit high performance in CO_2 capture and activation [33]. This efficiency is associated with the presence of amine groups, whose lone pair of electrons on nitrogen interacts with the electrophilic carbon of CO_2, favoring its activation through the formation of species such as carbamates or dipolar intermediates resulting from the nucleophilic addition of the amine to the CO_2 molecule [34–36]. This interaction increases CO_2 polarization and lowers the energy barrier of subsequent steps, facilitating its insertion into organic substrates [37].

Additionally, the basic character of amines enhances CO_2 affinity and selective adsorption on the catalytic surface, increasing the local concentration of the reactant [38]. When these amine-based systems are supported on mesoporous materials or combined with metal centers, a cooperative effect is established between basic sites (CO_2 activation) and acidic or metallic sites (substrate activation), resulting in greater catalytic efficiency, selectivity, and yield in conversion reactions under mild conditions [34,39–41].

The integration of carbon capture, conversion, and reuse technologies can not only mitigate emissions but also stimulate new sustainable and innovative business models aligned with the Sustainable Development Goals (SDGs), particularly SDG 3 (Good Health and Well-Being), SDG 9 (Industry, Innovation and Infrastructure), SDG 12 (Responsible Consumption and Production), SDG 13 (Climate Action), SDG 14 (Life Below Water), and SDG 15 (Life on Land) [42,43].

The CO_2 molecule exhibits high thermodynamic stability and low intrinsic reactivity, factors that limit its transformation and directly influence its performance in catalytic processes [44,45]. CO_2 has a linear geometry (O=C=O), resulting from sp hybridization of carbon, which evenly distributes electron pairs and gives the molecule a zero dipole moment [46,47]. In catalysis, the high stability of CO_2 leads to large activation barriers and thermodynamically unfavorable equilibria for valorization reactions, such as cyclic carbonate formation and reduction processes [48].

In heterogeneous systems, adsorption on metal surfaces or oxides, especially at acid/base sites, promotes polarization of the C=O bonds [49,50]. In homogeneous systems, coordination to metal centers facilitates its insertion into metal/hydride or metal/carbon bonds [51,52]. In reduction reactions, coupled electron and proton transfer is crucial to enhance selectivity and efficiency [53,54].

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3. Glycerol in the Biodiesel Production Chain

Biodiesel, obtained mainly through the transesterification of vegetable oils or animal fats, is a renewable biofuel of significant relevance to the global energy matrix [55–57]. In Brazil, it stands out as strategic due to the country's agricultural base, infrastructure, and public policies such as the National Program for the Production and Use of Biodiesel (PNPB) [58], with production exceeding 6 billion liters in 2023 and ranking third worldwide [59], driven by the mandatory B14 blend and the upcoming B20 mandate [60]. However, the sector's expansion intensifies glycerol generation, whose accumulation devalues the product in the market and may cause environmental impacts, reinforcing the need for valorization strategies [61–63].

Glycerol, a byproduct of this reaction, accounts for approximately 10% of the total mass generated in the process [64–68]. Also known as glycerin, glycerol ($C_3H_8O_3$) is an organic compound belonging to the alcohol class, specifically a triol, due to the presence of three hydroxyl (-OH) groups in its molecular structure [69,70].

It appears as a colorless, viscous, hygroscopic liquid with a sweet taste and is completely miscible in water [71,72]. In addition to being obtained through the transesterification reaction in biodiesel production, glycerol can naturally occur in oils and fats in the form of triglycerides [73,74]. Due to its physicochemical properties, such as high polarity, elevated boiling point, and strong hydrogen-bonding capacity, glycerol has wide applications across different industrial sectors [75,76].

In the cosmetic industry, it acts as a humectant and emollient in formulations of creams, lotions, and soaps [77,78]. In the food sector, it is used as a multifunctional additive (classified as E422), performing preservative, sweetening, and humectant functions [79,80]. In the pharmaceutical field, it is employed in the formulation of syrups, gelatin capsules, and suppositories due to its biocompatibility and safety for human use [81].

Additionally, glycerol constitutes an important raw material in the chemical industry, being used in the synthesis of resins, plasticizers, solvents, and explosives, such as nitroglycerin [82]. However, the high availability of crude glycerol, typically impure and containing contaminants from biodiesel production, limits its direct use and reinforces the need for technological routes that add value to this compound.

One of the most favorable alternatives is microbial fermentation to obtain 1,3-propanediol (1,3-PDO), a monomer used in the production of polymers such as polytrimethylene terephthalate (PTT) [83]. Another possibility is the production of acrylic acid, which can be obtained from the selective oxidation of glycerol using heterogeneous catalysts, such as mixed vanadium and tungsten oxides. This product is widely used in the manufacture of plastics, paints, and adhesives [84–86]. Finally, another alternative is the cycloaddition reaction of CO_2 to form glycerol carbonates, a process that combines residual glycerol valorization with CO_2 mitigation [87–89].

4. Chemical Fixation of CO_2 for Glycerol Carbonate (GC)

Cyclic carbonates stand out as high value-added derivatives obtained from CO_2, with applications ranging from green solvents and intermediates for polymer synthesis to electrolytes for lithium-ion batteries and coordinating agents in catalytic systems [90,91].

Their production occurs mainly through the reaction of CO_2 with epoxides or alcohols, using basic catalysts, Lewis acids, or bifunctional systems capable of promoting the simultaneous activation of the reactants, an essential step given the thermodynamic limitations associated with CO_2 insertion [92,93]. In addition, these compounds are characterized by low toxicity, high chemical stability, and good biodegradability.

The bibliographic search conducted on February 18, 2026, in electronic databases, including ScienceDirect, Springer Nature, Wiley Online Library, Taylor & Francis, ACS, MDPI, and SciELO, using the terms "Chemical Fixation of CO_2" and "Glycerol Carbonate" in the title, abstract, and keywords, and considering research articles, book chapters, and short communications published from 2020 to 2026, resulted in the data presented in Figure 2.

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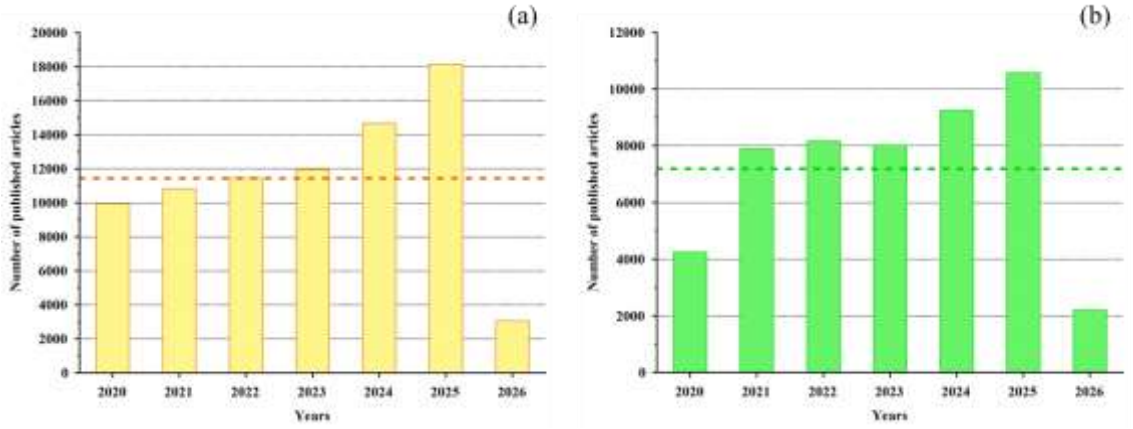

Figure 2. (a) Chemical fixation CO_2 (b) Glycerol carbonate.

The analysis of the bibliographic search reveals a consistent growth in the number of publications related to "Chemical Fixation of CO_2" and "Glycerol Carbonate" over the period from 2020 to 2025. In the case of chemical CO_2 fixation, a continuous upward trend is observed, with a gradual increase between 2020 and 2023 and a more pronounced intensification from 2024 onward, reaching a peak in 2025. This trend reflects the strengthening of scientific and technological interest driven by global discussions on emission mitigation and the development of sustainable pathways.

Similarly, publications on glycerol carbonate show a significant increase from 2020 to 2021, relative stability until 2023, and a new marked rise from 2024 onward, culminating in the highest number of publications in 2025. This pattern highlights its consolidation as a strategic topic linked to glycerol valorization and green chemistry. In both cases, the lower number recorded in 2026 is due to the fact that the survey was conducted at the beginning of the year and therefore does not yet represent the total expected number of publications, confirming the continued expansion of these research areas in the international scientific landscape.

Table 1 presents literature studies addressing the chemical fixation of CO_2 for the production of glycerol carbonate. The systematization of the table highlights different types of catalysts, as well as the reaction conditions, together with their respective performances.

Table 1. Performance of catalysts in the CO_2 fixation reaction with glycerol for the production of glycerol carbonate.

Catalyst (best performance)	Optimal reaction condition			Performance			Reference
	T (ºC)	P (MPa)	t (h)	Selectivity (%)	Yield (%)	Cycles	
Au/MgO	140	0.1	6	-	60	-	[94]
Trietilamina (TEA)	100	2.5	1	87	90	-	[95]
Zn/MCM-41	140/145	0.1	5	98	74	3	[96]
[TMG][TFE]	80	0.1	0.5	95.4	87.7	-	[97]
$ZnWO_4$-ZnO	150	5	6	100	6.5	5	[98]
Amberlite Amb-OH-910-I	115	2	2	-	81	4	[99]
ZnO/SiO_2	140	0.004	6	77.83	64.3	-	[100]
ZIF-67	210	0.3	12	98	29	6	[101]
Zn(OTf)2/1,10-fenantrolina	170	5	48	100	80	5	[102]
PBA Zn(II)-Co(III)	120	2	6	94	81/84	5	[103]
MgO	150	8	24	46.9	12.76	-	[104]
CuO	120	3	5	69.4	61.8	7	[105]
P-DVB-DBUVBI-1	100	2	6	98.9	90	5	[106]

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Mg/Zn/CeO_2	150	4	5	-	58	5	[107]
Cu/In_2O_3/ZnO	150	5	5	64	24.46	5	[108]
0,3ZnO-CaO-SBA-15	110	3	4	76.2	49.3	5	[109]
Pd/CeO_2/TiO_2	150	4	5	60	20.4	5	[110]

*T (temperature), P (pressure), and t (time).

From Table 1, it can be observed that the catalytic performance in the conversion of CO_2 into glycerol carbonate (GC) is strongly related to the nature of the catalyst and the operational conditions employed, especially temperature, pressure, and reaction time. Homogeneous systems or catalysts functionalized with well-defined basic sites, such as triethylamine [95] and guanidine-based ionic liquids [97], achieve high yields (≈ 90% and ≈ 87.7%, respectively) under relatively mild conditions, demonstrating high catalytic efficiency.

Similarly, functionalized polymers also reach high yield and selectivity values (≈ 90% and 98.9%), indicating that catalysts with accessible basic sites favor the simultaneous activation of CO_2 and glycerol [106]. In addition, mixed oxide systems have shown superior performance compared to isolated oxides, as evidenced by the hybrid Ni/Nb system [109]. In this case, the combination of metallic species suggests a bifunctional activation mechanism in which Ni contributes redox and/or basic sites associated with glycerol activation, while Nb, due to its strong Lewis acidity, acts in stabilizing reaction intermediates such as carbamates, promoting reaction progression and increasing yield.

In contrast, several simple or mixed metal oxides exhibit moderate or low yields, even under more severe pressure and temperature conditions, suggesting limitations associated with CO_2 activation and insufficient synergy between acidic and basic sites [98,104]. Pure MgO achieved a yield of only 12.76%, whereas the combined Au/MgO system reached 87%, demonstrating that the isolated presence of basic sites is not sufficient to promote high catalytic efficiency [104]. These results indicate that factors such as adequate metal dispersion and higher surface area, features that can be enhanced through the use of supports such as rice husk ash-derived silica, play a decisive role in, as they improve accessibility to active sites and optimize the utilization of the reaction performance catalytic phase.

Moreover, structured materials such as MOFs and mesoporous supports can provide high selectivity; however, this does not always translate into high yields, highlighting that overall efficiency depends on the balance among structure, composition, and reaction conditions. In the specific case of mesoporous supports, a frequently critical aspect is the stability of active species against leaching, particularly in systems operating in liquid media [111]. In this regard, systems employing rice husk ash-derived silica obtained via the sol–gel method tend to promote stronger anchoring of metallic species within the matrix, favoring stronger interactions between the active phase and the support and reducing metal loss compared to simpler doping methods. This contributes to greater catalytic stability and reuse potential.

Various catalytic approaches have been explored for the conversion of CO_2 into glycerol carbonate, including supported metal oxides (CaO, MgO, Nb_2O_5, NiO), functionalized mesoporous materials (such as SBA-15 and MCM-41), graphene-based composites, and catalysts doped with metal cations to tune acid–base and redox properties [104,112]. The incorporation of ionic liquids (ILs), particularly imidazolium-based ([BMIM]Br, [EMIM]Cl) and quaternary ammonium (TBAB) species, is also noteworthy. These are generally applied by impregnation onto supports such as silica, SBA-15, or alumina, forming hybrid heterogeneous systems [39,113]. In these materials, ILs enhance CO_2 solubility in the reaction medium and promote its activation through the interaction of the anion (Br^-, Cl^-) with the electrophilic carbon, in addition to assisting in intermediate stabilization and reactive species activation [36]. This combined action improves the interaction between reactants and active sites, resulting in higher conversion rates and selectivity toward glycerol carbonate.

5. Advances in Heterogeneous Catalysts for Glycerol Carbonate Synthesis

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5.1. Silica-Based Materials as Catalytic Supports

The use of alternative silica sources has gained relevance in the context of sustainable chemistry, especially when associated with the valorization of agro-industrial residues. In this scenario, rice husk ash (RHA) stands out as a strategic raw material, since rice production is a pillar of the global agri-food sector and generates large volumes of husk as a byproduct, which, after controlled combustion, may contain more than 90% amorphous SiO_2 [114,115].

Brazil ranks 11th in the world in rice production [116] and presents a promising outlook: the National Supply Company projects, for the 2024/2025 harvest, a production of 12.1 million tons, driven by the expansion of cultivated area and favorable profitability [117]. Rio Grande do Sul leads national production, followed by Santa Catarina and Mato Grosso [118], maintaining an average of 8 million tons per harvest, even in the face of the floods of 2023 and 2024 [117,119]. For every ton of processed rice, approximately 200 kg of husk are generated, and about 20% of this mass is converted into ash after combustion [120], representing an environmental liability that can be transformed into a high value-added resource.

RHA is predominantly composed of silica (80–95%) and may contain alumina, iron oxides, calcium, magnesium, sodium, and potassium, depending on combustion conditions [115]. The silica morphology may vary between crystalline and amorphous forms, the latter being more desirable due to its higher reactivity and better performance in industrial applications [121]. Combustion temperature is decisive: above 700 °C, the formation of crystalline phases such as cristobalite and tridymite, less reactive forms, is favored [122], whereas controlled combustion between 500–700 °C promotes amorphous silica with suitable pozzolanic properties [123].

The presence of pores in the mesoporous range (2–50 nm) ensures greater accessibility to active sites, reduces internal diffusion resistance, and promotes better utilization of the active phase, contributing to higher catalytic activity and system efficiency [124–127]. These structural and chemical characteristics enable various RHA applications in sectors such as civil construction, the chemical industry, and agriculture [128], including high-performance concretes and cements [129], advanced ceramics, catalysts, and the synthesis of nanostructured silica.

Studies have demonstrated its efficiency in removing heavy metals, synthetic dyes, organic compounds, and nutrients such as phosphorus and nitrogen from wastewater [130,131], both in its natural form and when chemically modified [128,132]. It can also act in coagulation–flocculation processes [133] and as a catalyst support in advanced oxidation processes, such as photocatalysis [134].

In the synthesis of mesoporous materials, MCM-41-RHA and SBA-15-RHA have been produced for solid hydrogen storage, showing good adsorption performance with nickel impregnation up to 10%, particularly in the 5% sample, which maintained stability after five cycles [135]. Similarly, RHA was employed in the synthesis of ZSM-5 zeolite combined with fly ash; when modified with iron/copper, the zeolite achieved 99% degradation of tetracycline in 10 minutes via a Fenton-type oxidation process [136]. In aquaculture, its application as a silicon fertilizer and heavy metal adsorbent increased dissolved silicate concentration and achieved mercury removal of up to 97.41% after NaOH modification [137].

In the energy field, RHA has been investigated as a heterogeneous catalyst in biodiesel production. A yield of 89.97% was obtained in the transesterification of waste oil using catalysts containing 10% strontium oxide calcined at 900 °C, under conditions of 65 °C, an oil/methanol ratio of 1:18, 6% catalyst loading, and 1 hour of reaction, meeting international quality standards [138].

For CO_2 related applications, RHA has been combined with ashes from coal co-combustion as a source of silica and alumina in the synthesis of zeolites for CO_2 capture, producing a material with high surface area and significant adsorption capacity [139]. Another study reported silica extracted from RHA converted into aerogel and subsequently functionalized with APTES, resulting in a material with active amine groups capable of adsorbing 0.88 mol of CO_2/kg, demonstrating improved capture performance through combined physisorption and chemisorption mechanisms [140]. The in situ incorporation of RHA in the synthesis of MIL-101(Cr) significantly increased its surface area and

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pore volume, resulting in a 14–27% enhancement in CO_2 adsorption capacity, greater CO_2/N_2 selectivity, and improved overall capture performance compared to the unmodified material [141].

Several studies highlight RHA as a sustainable and low-cost alternative for catalytic and environmental applications [142–144]. One example is its use in crude glycerol purification, enabling its application in higher value-added routes and contributing to the circular economy [145]. Its conversion into mesoporous silica allows the production of catalytic supports with adequate dispersion of active species and tunable acid–base properties. Porosity control is essential, since the high viscosity and molecular size of glycerol may cause diffusion limitations in microporous materials [146].

In this context, silica extracted from RHA presents suitable properties to act as a precursor in the sol–gel technique, a versatile method for obtaining porous materials with high structural homogeneity, in which silica serves as a silicon source for gel formation that, after hydrolysis and condensation steps, generates solids with tunable physicochemical properties [147–149].

5.2. Sol–Gel Route and Silica Functionalization: Structural and Catalytic Influence

Silica-based materials stand out as versatile catalytic supports due to their high surface area, thermal and chemical stability, and the possibility of structural modification through different synthetic routes [150,151]. Among these routes, the sol–gel method is widely applied in obtaining homogeneous matrices with controlled pore distribution and active sites [152,153]. Based on the transition from a colloidal solution (sol) to a three-dimensional crosslinked network (gel), the process occurs through hydrolysis and condensation reactions of metallic precursors, generally alkoxides or salts dissolved in appropriate solvents [154–156].

During hydrolysis, metal-bound hydroxyl groups are formed [157], which subsequently undergo condensation, generating metal–oxygen–metal bonds and consolidating the inorganic structure [155]. Depending on the drying conditions, xerogels, aerogels, or thin films can be obtained [158,159]. Aerogels are characterized by supercritical drying and ultralow density [160], whereas xerogels result from conventional drying with greater structural shrinkage [161]. Hydrogels, formed from hydrophilic polymers, exhibit high water absorption capacity [162].

Among the advantages of the method are low processing temperature, homogeneous distribution of components, and ease of incorporating different species into the inorganic matrix [163,164], factors that broaden its application in sensors, biomaterials, photonics, functional coatings, and electroceramics [165–168].

In catalysis, the sol–gel route enables the production of materials with high dispersion of active phases [169,170]. A high efficiency of TiO_2 nanoparticles synthesized via this method was observed in the degradation of methylene blue (80.89%) and in the oxidation of benzaldehyde to benzoic acid (94%) [84]. Another study reported high adsorption capacity of Pb(II) and Cd(II) by porous SiO_2 nanoparticles [171], as well as the development of a heterogeneous Fenton catalyst based on La-doped $MgFe_2O_4$ with high efficiency in carbamazepine removal [172].

The preparation of porous supports for metal oxides via the sol–gel method promotes high dispersion, thermal stability, and structural control of materials [173]. This characteristic has been explored both in the synthesis of catalysts for transesterification in biodiesel production [174,175] and in glycerol valorization toward higher value-added routes [176]. In this context, functionalization of the silica matrix obtained via sol–gel allows the controlled incorporation of metal oxides with tunable acid–base and redox properties, enhancing catalytic performance.

Particularly noteworthy is the introduction of species such as Nb_2O_5 and TiO_2, whose structural and electronic characteristics can modulate acidity, metal dispersion, and metal–support interactions, highlighting the potential of the sol–gel technique as an effective strategy for homogeneous metal incorporation and for the development of advanced catalytic systems.

5.3. Metal Doping: Niobium Oxide (Nb_2O_5), Nickel Oxide (NiO), and Graphene-Based Hybrid Systems

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The doping of silica-based materials with metal oxides has proven to be an efficient strategy to optimize the acid–base, electronic, and structural properties of catalysts applied to the conversion of CO_2 into glycerol carbonate. Among these metals, niobium (Nb) and nickel (Ni) stand out due to their chemical versatility and the broad technological applications associated with their compounds, particularly niobium oxide (Nb_2O_5) and nickel oxide (NiO), which are widely studied as catalytic promoters.

Niobium is a group 5 element with atomic number 41, characterized by high ductility, corrosion resistance, and a metallic gray appearance. Discovered in 1801, it occurs naturally mainly in minerals such as columbite, tantalite and pyrochlore, the latter being the primary industrial source [177,178]. Brazil holds approximately 98% of global niobium reserves, with deposits concentrated in the states of Minas Gerais and Goiás, giving the country a strategic role in the production and development of advanced niobium-based materials [179,180]. In technological applications, niobium is prominent in high-performance alloys, superconductors, electronic devices, and especially as an active component in heterogeneous catalysts [181–183].

Among its compounds, Nb_2O_5 is one of the most relevant due to its high thermal stability, surface acidity, semiconducting behavior, and good dielectric properties [184,185]. This oxide may exhibit different crystalline structures, monoclinic, orthorhombic, or tetragonal, depending on synthesis and calcination conditions. In heterogeneous catalysis, Nb_2O_5 is widely employed in reactions involving Lewis and Brønsted acid sites, such as dehydration, selective oxidation, cracking, and cycloaddition, reinforcing its potential in glycerol carbonation reactions [186–188]. Incorporating Nb_2O_5 into a silica matrix obtained via the sol–gel method enhances active site dispersion, improves mass transport, and promotes the simultaneous adsorption of CO_2 and glycerol key aspects for carbonate intermediate formation.

Similarly, Ni is a group 10 transition metal widely recognized for its corrosion resistance, thermal stability, and electronic and magnetic properties [189–191]. Naturally found in ores such as laterites and sulfides, nickel is globally distributed and extensively mined in countries such as Indonesia, the Philippines, Russia, and Brazil [192–194].

Historically used in metal alloys and stainless steels, nickel also plays a crucial role in heterogeneous catalysis, particularly in hydrogenation, dehydrogenation, cracking, hydrocarbon reforming, and Fischer–Tropsch processes [195,196]. In sustainability-related applications, nickel catalysts have been employed in biomass conversion, biogas reforming, and CO_2 activation due to their ability to facilitate reactions involving C–H and C–C bond transformations [197–199].

NiO, in particular, stands out for its redox properties, thermal stability, and applications in electrocatalysis and energy storage [200,201]. It is widely used in the catalyst industry, battery electrodes, and ceramic films, with coloration ranging from green to black depending on its stoichiometry [202–205]. Synthesis techniques such as sol–gel, coprecipitation, and hydrothermal methods allow control over morphology, surface area, and crystallinity, resulting in more active catalysts that are resistant to sintering, especially when supported on mesoporous silica, alumina, or zeolites [32,206,207].

The combination of Nb_2O_5 and NiO in hybrid systems has proven successful, as it integrates Lewis acidity, redox properties, and thermal stability—features desirable for CO_2 conversion reactions. Simultaneous doping can generate important synergistic effects, increasing active site density, improving CO_2 polarization, and enhancing selectivity toward glycerol carbonate formation.

Recent studies also demonstrate the potential of incorporating graphene or graphene oxides as structuring additives, promoting improved metal dispersion, increased surface area, and enhanced electronic interaction between metallic phases and the support [208]. These hybrid materials offer additional advantages, such as high stability and resistance to deactivation, making them particularly attractive for sustainable CO_2 valorization processes.

Within this context, ionic liquids stand out for their high capacity to solubilize and activate CO_2, especially when containing basic anions. They can act as co-catalysts by stabilizing intermediates and facilitating glycerol activation. When immobilized in matrices containing Nb_2O_5 and NiO, they

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combine the advantages of homogeneous and heterogeneous catalysis, contributing to improved efficiency, selectivity, and stability of the catalytic system in glycerol carbonate formation.

5.4. Role of Ionic Liquids and Ionic Solids in the Conversion of CO_2 Into Glycerol Carbonate

Ionic liquids (ILs) and ionic solids constitute distinct classes of compounds that have gained prominence due to their unique physicochemical properties and broad spectrum of technological applications. Initially, these compounds were referred to as "room-temperature molten salts." However, debates regarding the accuracy of the term "molten salts" and even the definition of "room temperature" which varies according to geographic region and season led to the adoption and consolidation of the term "ionic liquids" [209]. ILs are salts composed exclusively of ions that remain liquid at temperatures below 100 °C, with many being liquid at room temperature [37]. They generally consist of bulky organic cations, such as imidazolium, pyridinium, ammonium, or phosphonium, combined with organic or inorganic anions [210,211]. The high structural asymmetry of these ions hinders crystalline packing, which explains their low melting points and pronounced liquid character.

Among their main properties are low vapor pressure, high thermal stability, good ionic conductivity, and excellent solvation capacity [37,212,213]. These characteristics make ILs particularly attractive for applications in organic synthesis, homogeneous and heterogeneous catalysis, selective extractions, electrochemistry, lubrication, and energy storage [214,215]. In the environmental context, several ILs have stood out in CO_2 capture, activation, and conversion, since many contain basic sites capable of strongly interacting with this linear and relatively unreactive molecule [216,217].

The possibility of tailoring the structure of an ionic liquid through the selection of its cation and anion is fundamental for the development of customized catalytic systems. Modifications in the chemical nature of these ions allow control over properties such as polarity, viscosity, acidity, or basicity, thereby adjusting IL performance for specific applications [218–220]. As a result, the use of ILs functionalized with acidic or basic groups has been promoted, showing high performance in esterification, transesterification, and alkylation reactions, and even being employed as catalysts or co-catalysts in biodiesel production [221,222].

In the specific case of glycerol conversion with CO_2 to form glycerol carbonate, ionic liquids can perform multiple catalytic roles [223]. They can promote CO_2 activation through basic anions capable of forming carbonate- or carbamate-type intermediates, thereby increasing system reactivity [224]. Additionally, they improve miscibility between CO_2 and glycerol, minimizing diffusion limitations typical of this reaction. When immobilized on solid supports, ILs can also modify the surface properties of heterogeneous catalysts, adjusting acidity, basicity, and medium polarity [225,226]. Furthermore, they are able to stabilize reactive intermediate species, enabling the process to proceed under milder temperature and pressure conditions [36,225].

Beyond ionic liquids, it is important to distinguish their role from that of ionic solids, which are crystalline compounds composed of three-dimensional networks of strongly bonded cations and anions, such as chlorides, fluorides, sulfates, and metal oxides [227,228]. These materials exhibit high melting points and hardness and are widely used in heterogeneous catalysis, sensors, ceramics, and solid-state batteries [229]. Despite their structural and functional differences, ionic liquids and ionic solids share the presence of strong electrostatic interactions, which facilitates their combination in hybrid systems.

Although ILs are often classified as "green solvents" due to their low volatility, it is important to recognize limitations related to toxicity, synthesis cost, and low biodegradability, factors that still restrict their large-scale application [230].

Indeed, the incorporation of ionic liquids into porous solid supports such as silica, zeolites, modified clays, or carbon-based materials has emerged as an attractive strategy for sustainable catalysis. These hybrid materials combine the chemical versatility of ILs with the structural stability and high surface area of solids, resulting in more selective, less corrosive, and more easily recoverable

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catalysts [231,232]. In the conversion of CO_2 into glycerol carbonate, such systems simultaneously enable CO_2 activation, enhance mass transport, and stabilize active catalytic sites, significantly improving the overall efficiency of the process.

6. Conclusions

The catalytic conversion of CO_2 into glycerol carbonate stands out as a pathway aligned with the principles of the circular economy, as it simultaneously transforms CO_2 emissions and surplus glycerol from the biodiesel chain into a high value-added compound with broad industrial applicability. The analyzed studies demonstrate that process performance is directly related to the nature of the catalyst, the accessibility of active sites, and the reaction conditions employed. In this context, silica-based heterogeneous catalysts particularly those derived from rice husk ash and synthesized via the sol–gel method combined with metal oxide doping and the use of ionic liquids, represent promising alternatives in terms of selectivity, structural stability, and reuse potential.

However, despite laboratory-scale advances, the consolidation of this technology at the industrial level still faces significant challenges. Among the main barriers are the need to ensure catalytic stability over multiple reaction cycles, maintain activity under continuous operating conditions, mitigate diffusion limitations in more concentrated systems, and, above all, address the energy costs associated with CO_2 compression and pressurization. Furthermore, aspects such as catalyst synthesis scalability, economic feasibility of the integrated process, and life cycle assessment still require in-depth investigation. Thus, progress toward industrial application demands not only catalytic improvements but also comprehensive process optimization from technical, economic, and environmental perspectives.

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