SOLID BASE CATALYSIS

A NEW FRONTIER IN INDUSTRIAL SUSTAINABILITY

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Bentham Books

Solid Base Catalysis: A New Frontier in Industrial Sustainability

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ISBN (Online): 979-8-89881-147-1

ISBN (Print): 979-8-89881-148-8

ISBN (Paperback): 979-8-89881-149-5

Published by Bentham Science Publishers Pte. Ltd. Singapore, in collaboration with
Eureka Conferences, USA.

First published in 2025.

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PREFACE

In recent years, the field of solid base catalysis has emerged as a groundbreaking area of research and application, driven by the growing demand for sustainable industrial processes. "***Solid Base Catalysis: A New Frontier in Industrial Sustainability***" aims to capture this dynamic and rapidly evolving domain, highlighting both recent advancements and the potential future directions of solid base catalysis.

Solid base catalysts have garnered significant attention due to their ability to facilitate a wide range of chemical reactions with high efficiency and selectivity. This versatility makes them pivotal in numerous industrial applications, from organic synthesis to biomass conversion, and even in the development of eco-friendly processes. The advancements in this field not only promise enhanced performance but also align with the broader goal of achieving sustainability in chemical manufacturing.

The book is structured to provide a comprehensive overview of the current state-of-the-art in solid base catalysis. The first chapter delves into the latest methods for synthesizing and characterizing these catalysts, offering insights into various techniques such as precipitation, sol-gel methods, and template-assisted approaches. This foundational knowledge sets the stage for subsequent discussions on more specialized applications.

Following the introduction, each chapter explores different facets of solid base catalysis, reflecting its diverse applications. From supported solid-phase catalysis in organic synthesis to innovative approaches for biomass utilization and chemical conversions, the book covers a broad spectrum of topics. Chapters dedicated to biomass oxidation, multicomponent reactions, and drug synthesis illustrate the practical implications of solid base catalysis in addressing real-world challenges.

Significant emphasis is placed on recent advancements in the field. Detailed discussions on the catalytic mechanisms, optimization strategies, and the development of novel materials offer readers a deep understanding of current trends. Chapters on C-C, C-N, and C-S bond formations, as well as reduction reactions, showcase the versatility and potential of solid base catalysts in a variety of chemical transformations.

As we move towards a more sustainable future, the role of solid base catalysis becomes increasingly critical. The final chapters reflect on the historical context and recent progress, providing a comprehensive perspective on how these catalysts contribute to greener and more efficient chemical processes.

We extend our gratitude to the contributors of this volume, whose expertise and dedication have made this book possible. Their insights and research are a testament to the vibrant and continually advancing field of solid base catalysis. We also hope that this book serves as a valuable resource for researchers, practitioners, and students alike, fostering further innovation and application in the realm of industrial sustainability.

It is our belief that the knowledge presented in these pages will inspire new ideas and drive continued progress in the field of solid base catalysis, ultimately contributing to a more sustainable and efficient chemical industry.

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CHAPTER 1

Recent Methods for the Synthesis and Characterization of Solid-base Catalysts

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Abstract: Solid-base catalysts have acquired significant attention due to their essential role in various chemical processes, including catalytic transformations, environmental remediation, and energy conversion. This chapter highlights a comprehensive overview of current approaches to the synthesis, characterization, innovative methods, and techniques employed in the development of solid-base catalysts. A wide array of methodologies has emerged, ranging from traditional techniques to cutting-edge approaches, facilitating the design and optimization of solid-base catalysts. The synthesis section discusses novel approaches such as sol-gel hydrothermal, nanoparticle immobilization, impregnation, metal-organic framework, fly ash technique, template-assisted techniques, and conventional methods like generation of the basic site by pretreatment at high temperature. Each technique offers unique advantages in controlling catalyst morphology, composition, and surface properties. Furthermore, recent developments in characterization techniques like X-ray diffraction, Fourier-transform infrared spectroscopic, NMR, Temperature programmed desorption, microscopic and surface analysis methods such as indicator method, *etc*., have enabled detailed insights into the physicochemical properties and active sites of catalysts and structure-activity relationships governing the catalytic performance of solid-base materials. Moreover, computational methods play an essential role in predicting and optimizing the catalytic performance of these materials. By summarizing these recent methodologies, this chapter aims to provide valuable insights into the advancements in solid-base catalyst development, paving the way for enhanced catalytic efficiency and sustainability in various chemical processes.

Keywords: Morphological analysis, Solid-base catalysts, Spectroscopic techniques, Structural analysis, Synthetic methods.

INTRODUCTION

Solid base catalysts are materials that can donate electrons or are capable of abstracting protons from reactant molecules based on their types, like Bronsted-

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Lowry Bases, Arrhenius bases, and Lewis bases. Metal oxides, Hydroxides, Carbonates, and supported alkali/alkaline earth metals are examples of Solid base catalysts used for many base-catalyzed reactions [1-4]. Solid base catalysts can be synthesized through conventional methods like basic site generation by pretreatment at high temperatures, precipitation methods, nanoparticle immobilization, and impregnation. Recently, several advanced methods like metal-organic frameworks [5,6], sol-gel, hydrothermal method, one-pot synthesis, [7, 8], and Fly ash have been widely used for the synthesis of solid base catalysts [8-14].

It is crucial to determine the main characteristics of surfaces to predict their potential as base catalysts. This can be easily achieved by having control over crucial parameters which optimize the catalytic process. The number of basic sites, its origin, and its strength are the main factors that define the basicity of any solid base catalyst. Over the past decade, significant advancement has been made in the methods pertaining to the characterization of solid base catalysts. The structural analysis of the base catalyst uses the Brunauer Emmett-Teller technique and X-ray diffraction analysis. Thermal analysis is conducted using Thermogravimetric Analysis (TGA), Differential Scanning Calorimetry (DSC), Differential Thermal Analysis (DTA), Temperature-Programmed Reduction (TPR), and Temperature-Programmed Desorption (TPD) techniques. Spectroscopy techniques include FT-IR and NMR spectroscopy.

This chapter illustrates the impact of new supports, on the one hand, recent catalyst synthesis methods and characterization techniques of solid base catalysts are also discussed.

Synthetic Methods of Solid Base Catalysts

Generation of the Basic Site by Pretreatment at High Temperature

Usually, the surfaces of basic materials are layered with water and CO_2 in the air. Because of that reason, they do not exhibit their catalytic activities. The basic site on the surface of various materials can be generated by high-temperature pretreatment. The high-temperature pretreatment removes surface species like water, CO_2, and O_2 and allows the materials to exhibit basic properties and promote base-catalyzed reactions. The temperature required for the removal of adsorbed material is the decomposition temperature of hydroxide, carbonate, and peroxide.

The nature of the generated basic site depends on the severity of pretreatment. The molecules that are covering the surface are desorbed when the pre-treatment temperature increases, which is depicted in Fig. (**1**). For example, MgO pretreated at a temperature below 723 K has relatively very little activity for migration of double bond of 1- butene, but when the temperature rises above 723 K, it shows great activity that is up to a maximum of 873 K [15].

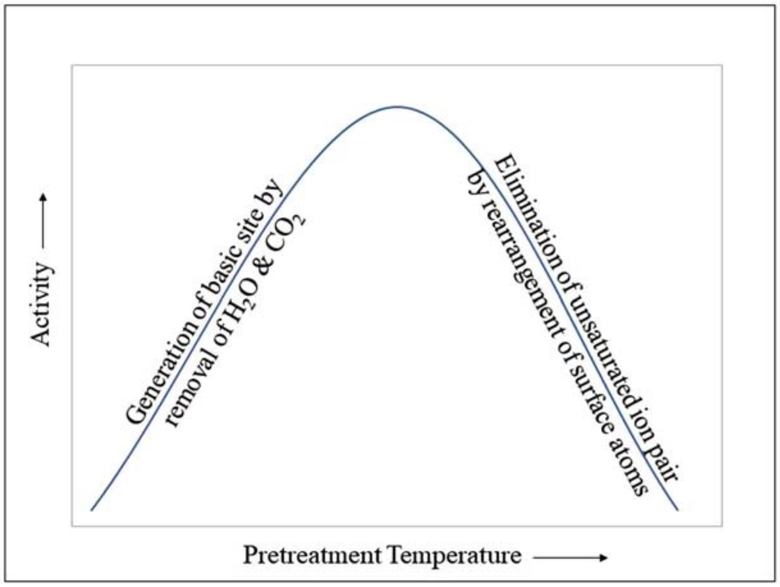

Fig. (1). Effect of pretreatment temperature on basic site.

Coluccia and Tench presented a model to study the impact of pretreatment temperature on metal oxide surface, specifically MgO, which is shown in Fig. (**2**). On the surface, there are different coordination numbers of MgO ion couples after high-temperature application to remove adsorbed molecules. Low coordination number sites are located close to edges, corners, and surfaces with a high Miller index. An incease in the pretreatment temperature generates different basic sites corresponding to ion pairs with varying coordination numbers. The most reactive site is the three-fold Mg^{2+}-three-fold O^{2-} ion pair, which is very unstable and has a tendency to reorganize or vanish at high temperatures. Despite its great adsorption of carbon dioxide and water, this pair is also very reactive.

Precipitation

The precipitation method for solid base catalysts involves the formation of solid catalysts by inducing the precipitation of metal ions or compounds from a solution. This process starts with creating a uniform mixture, followed by changes in temperature and the addition of acidic or alkaline solutions. Through chemical reactions, soluble ions in the solution interact with another reagent to produce insoluble solid compounds. Factors like temperature, pH, composition of raw

CHAPTER 2

Recent Advances in Supported Solid Phase Catalysis for Organic Synthesis

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Abstract: Solid-phase-supported catalysis has become a key method in organic synthesis over the last two decades, offering significant advantages in expediting both synthesis and purification. This chapter will explore the types of solid phases used in metal catalysis and the various approaches employed for organic transformations.

Keywords: Catalysis, Organic synthesis, Polymer catalyst, Solid-phase.

INTRODUCTION

R.B. Merrifield pioneered solid-phase organic synthesis and developed this technique for peptide synthesis in the 1960s [1]. In 1984, he received a Nobel Prize in chemistry for his pioneering work [2]. Since then, this field has had unbelievable interest [3 - 11]. Although the concept of solid phase-supported catalysis for organic synthesis was conceived in the 1940s, it gained importance after the Merrifield solid-phase peptide synthesis. Solid phase-supported catalysis for organic synthesis involves one reactant in an immobilized phase. It has become an important method for organic transformation due to its ability to expedite synthesis and purification and the removal of immobilized products easily by simple filtration [12]. Organic transformations, typically completed in multiple steps, can be carried out in a single step using solid-phase-supported catalysis. Another advantage of this technique is reduced side reactions, ease of handling, low susceptibility to moisture, cost-effectiveness, stability, high surface area, and reusability. These supported catalysts are stable because they are chemically bound to the solid support, allowing for easy removal after the

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reaction [13 - 15]. Moreover, they are utilized in industries for carrying redox reactions and carbon-carbon bond formation. These distinctive features make them ideal catalysts for facilitating green synthetic transformations, as they address issues such as pollution, chemical disposal, and sustainability [16 - 18]. Fig. (**1**) depicts the advantage of a solid-supported catalyst.

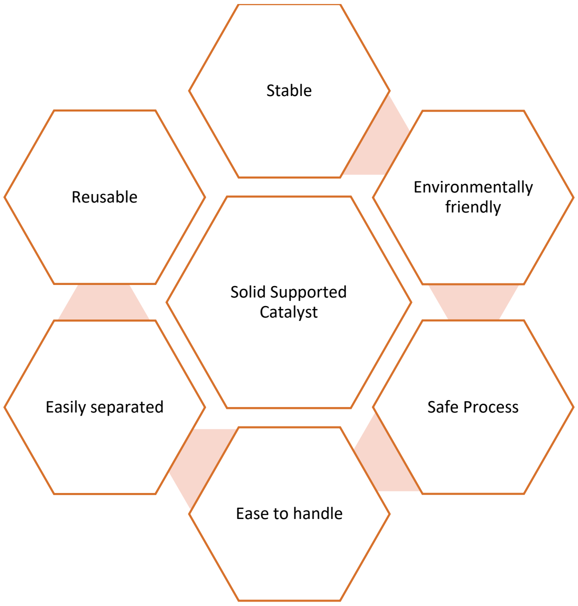

Fig. (1). Advantage of solid-supported catalyst.

These distinctive features make them ideal catalysts for green synthesis, as they help mitigate pollution, waste disposal, and sustainability challenges. Table **1** summarizes the various solid support catalysts used for organic transformation.

Various Types of Solid Supported Transition Metal Catalyst

Transition metal catalysts like palladium, ruthenium, rhodium, cobalt, and copper are supported on various heterogeneous solid surfaces and are used for various organic transformations. The noteworthy advantages of immobilized catalysts on solid support that make them a versatile and innovative tool in organic chemistry include reusability, ease of handling, and isolation from products. The different types of transition metal catalysts supported on solid phase and the various approaches used for organic transformations are discussed below:

Reaction Catalyzed by Solid Supported Palladium

The transition metal, palladium, is most extensively used as a catalyst in organic reactions. The Pd complexes are successful in homogeneous catalysis. However, removing the catalyst from the reaction mixture is difficult, limiting its use. This problem is overcome by using solid-supported palladium. The notable advantage of solid-supported palladium catalysts is that they are easy to prepare, recyclable, and have no leaching of Pd from support. These features increase the efficiency of solid-supported palladium catalysts. Palladium has the ability to change its oxidation state easily, thereby making it a versatile catalyst. Also, it shows high tolerance towards various functional groups and can withstand the reaction conditions. It also shows high selectivity, thereby facilitating the desired bond formation. Some of the organic reaction catalysts by solid-supported palladium are discussed herein:

Table 1. Different types of solid support for catalysis.

Types of Solid Support in Catalysis	Features	Examples	References
Metal Oxides	• Provide thermal and chemical stability to the catalyst • Easy to prepare.	TiO_2, CeO_2, SiO_2.	[19 - 21]
Zeolites	• They can catalyze hydrocarbon reactions due to the presence of Bronsted acid site on them. • Effective catalyst for gas phase reaction above 300 °C • Highly porous and stable.	Aluminosilicate material.	[22 - 24]
Carboneous Material	• They are carbon nanostructures • Porous and high surface area • The surface of carbonaceous material can be modified.	Graphene, graphite, Black carbon, Graphite oxide, Activated Carbon and carbon fibre.	[25 - 27]
Polymeric compounds	• They are employed for a metal catalyst • Improves stability of catalyst by preventing sintering.	Polyorganophosphazenes, styrene/divinylbenzene copolymers, dendrimers, fibers, and biopolymers.	[28, 29]

In Suzuki–Miyaura C-C cross-coupling reaction, aryl halides and arylboronic acids react to form biphenyls (40-99%). The efficient catalytic system involves a Pd complex with ligands having phosphorous and nitrogen. These ligands stabilize the complex and play a crucial role in controlling its activity and

CHAPTER 3

Advancing Biomass Utilization: Chemical Conversions, Catalysis, and Energy Applications

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Abstract: The use of biomass for chemical changes and catalysis is one of the causes that may mitigate the depletion of fossil fuels and environmental concerns. Biomass, which is a product of photosynthesis and the only carbon-based renewable resource, can be engineered into useful liquid fuels and other fine chemicals. In this regard, this innovation is of high importance in the promotion of carbon neutralization and sustainable green industrial development. Biomass catalytic conversion encompasses several steps in the chemical processes oriented towards the production of useful chemicals and fuels with less energy effort. The search for new materials and technologies for catalysts is an important factor in increasing the efficiency of biomass processing. Innovative catalysts can be a game changer for new processes, including lignin degradation, cellulose hydrolysis, biofuel production, *etc*., in the efforts to provide cleaner energy. Research is currently being carried out to develop catalysts that are both highly selective and environmentally friendly and improve the conditions under which biomass is converted. It aims to offer a complete understanding of the chemical processes that underlie bioenergy use, while providing innovative ideas for surface and interface designs. Ultimately, converting biomass into forms amenable for use besides supporting energy security can assist in addressing some of the urgent social ills that come with reliance on fossil fuel energy sources.

Keywords: Biomass, Heterogeneous catalysts, Solid catalysis, Sustainability.

INTRODUCTION

Fossil fuels are the major energy source to meet the energy demands worldwide. It is impossible to think about life without fossil fuel resources. Urbanization has led to an increase in the consumption of fossil fuel resources at a much larger scale than their production, which has made it difficult to sustain or rely on natural fossil fuel resources. This urges a strong demand to look for other sustainable and renewable alternatives for energy sources to meet global energy demands. Various growing renewable energy sources are being employed and are

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under research, such as electric, hydroelectric, wind, and solar energy. However, the uses of these resources are limited and they cannot be employed to generate value-added chemicals as they lack carbon content in them. Thus, to meet this criterion, alternatives that are carbon-rich and can be converted to fine chemicals need to be explored. One such great source of carbon from renewable energy sources is biomass. The governments of various countries have motivated industrial companies to work in this area in order to shift from fossil fuel resources to ones that can be generated by biomass resources [1, 2]. Biomass is the only carbon source available in nature that can be converted to produce various chemical compounds; thus, it has gained tremendous attention from researchers in past decades [3]. The only barrier to utilizing it as a precursor for fuel compounds is its effective transformation as it possesses high oxygen content and is quite complex in nature. Thus, effective catalytic routes are required to convert the complex oxygenated species to petrochemical products [4 - 7]. The same approach that is applied to convert fossil fuel resources to petrochemical products cannot be applied to process biomass resources to produce value-added commodities as they differ in their starting materials. In the processing of fossil fuel precursors, isomerization and alkylation are major steps, while for carrying out biomass deoxygenation, hydrolysis and hydrogenolysis are important reactions to start with [6] (Fig. **1**).

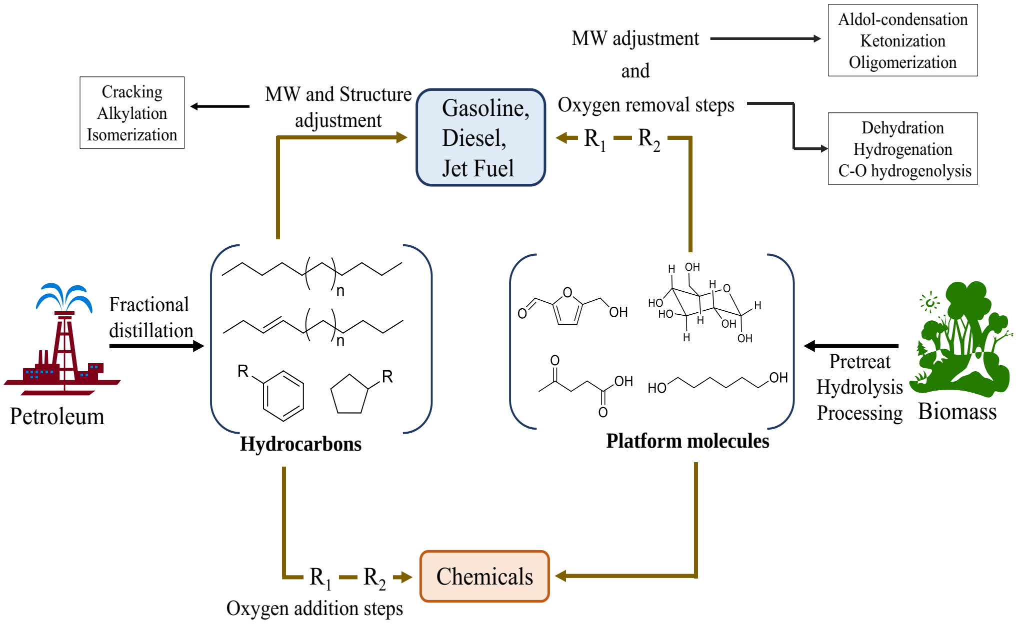

Fig. (1). Important reactions to process value-added products from fossil fuel resources and biomass resources.

To use biomass precursors as potential fuel precursors, the primary step involves breaking down their complex structures into simpler units. Biomass, specifically lignocellulosic biomass, is basically composed of 3 units known as cellulose, hemicellulose, and lignin [8 - 11]. Upon simplifying the complex biomass units *via* pre-treatment (ball milling and hydrolysis), these are sent to biorefinery units where they are converted to materials, energy, and fuel, as required. Upon the destruction of biomass after its preliminary treatment, it is converted into platform molecules based on the utility. These platform molecules or chemical building blocks that carry plenty of functional groups are further processed to a range of value-added products *via* various catalytic routes [12 - 15].

Role of Solid Catalysts

Due to the importance of using renewable feedstock as mentioned above, on the same grounds, catalysis also plays a crucial role from the viewpoint of green chemistry [16]. The role of catalysts in chemical manufacturing of fine chemicals is irreplaceable. However, unfortunately, after even becoming aware of green chemistry principles and the advantages of using solid catalysts, our manufacturing companies are working on the use of homogeneous acid and base catalysts, for instance, mineral acids, like sulfuric acid, hydrochloric acid, and phosphoric acid as acid catalysts; NaOH and KOH as base catalysts; oxidizing agents, $KMnO_4$ and $K_2Cr_2O_7$; reduction using Na, Mg, and Zn metals, *etc*. The use of these chemicals in a stoichiometric ratio generates hazardous and carcinogenic effects; hence, the only alternative to prohibit or reduce the emission of hazardous substances is the use of a heterogeneous catalytic system [17 - 20].

Long back, in the year 1958, researchers began to explore the possibility of using solid materials as acids and bases for catalytic reactions [21]. The basic characteristics of solid alumina were discovered in the 1930s. The history of using solid base catalysts in chemical reactions is amazing. It has been seen that with due course of time, their significance or utilization has increased. Solid catalysts are materials that possess catalytic active sites on their surfaces and are used to catalyze various chemical reactions. Unlike traditional liquid or homogeneous catalysts, solid catalysts are heterogeneous and exist in a solid phase. These catalysts have gained attention in various industrial and laboratory applications due to their advantages, such as ease of separation, reusability, and stability. In the 1990s, numerous metal oxides, such as MgO, CaO, SrO, BaO, LaO, and ZrO were identified as effective solid base catalysts [22, 23]. After the implications of metal oxides as base catalysts, further innovations have led to the emergence of layered double hydroxides and hydrotalcites as solid base catalysts, possessing unique properties [24 - 26].

CHAPTER 4

Oxidation of Biomass to Value-Added Chemicals Using Heterogeneous Solid Base Catalysts: A Recent Advancement

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Abstract: Biomass is considered one of the alternative resources with the greatest potential to compete both as fuels and as chemical intermediates. Biomass-derived molecules like glucose, xylose, HMF, and levulinic acid can be converted to value-added chemicals *via* dehydration, oxidation, isomerization, reforming, and aldol condensation processes using heterogeneous catalysts. The drawbacks of homogeneous catalysts are their low solvent solubility, susceptibility to breakdown under oxidation conditions, and requirement for product separation. Biomass contains a high amount of water, and the formation of water as a byproduct during transformation is a significant obstacle to reaction. Therefore, aqueous phase reaction using heterogeneous catalysts is of major interest. There have been reports of studies employing expensive noble transition metals as catalysts. Non-noble metal oxides are more widely available, less expensive, and have greater thermal stability and poisoning resistance than noble metals. In this chapter, we discuss some noble and non-noble metal heterogeneous catalysts. Heterogeneous metal catalyst involves single metal, bimetallic catalysts, metal oxide, spinel, perovskites, *etc*.

Keywords: Aqueous phase, Biomass, Heterogeneous catalyst, Intermediate, Noble metals.

INTRODUCTION

Biomass, as a sustainable and renewable resource, has gained significant attention in recent years as an alternative feedstock for the production of value-added products [1]. One of the key challenges in biomass valorization is the efficient conversion of biomass-derived compounds into high-value chemicals and fuels. Catalytic processes are crucial in this conversion, enabling selective transformations of biomass components into desired products [2].

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Over the past ten years, the public and commercial sectors have significantly supported scientific research and process evolution for synthesizing chemicals from sugars derived from lignocellulosic biomass [3]. New methods for synthetic biology and metabolic engineering, as well as new chemical and biological pathways and homogeneous and heterogeneous catalysts, have been developed as a result of this progress, allowing the conversion of waste products like glycerol, lignin, and sugars into useful chemicals and hydrocarbon fuels [4]. Inexpensive feeds and low-quality heat drive large-scale C-C coupling, hydrogenation, and deoxygenation processes. However, chemical catalysis requires clean feed streams to prevent catalyst deactivation [5]. Corn-derived sugar, sucrose (sugar cane), lignocellulosic sugars (glucose, xylose), and glycerol are examples of renewable resources that can be used to produce industrial chemicals and fuels [6]. Carbohydrates from C3 to C6 are available from these sources as basic ingredients. Biodiesel may also be made from vegetable and palm oils [7]. C–C coupling is well-established through methods like Methanol-To-Gasoline (MTG) and Fischer–Tropsch, and large-scale heterogeneous hydrogenation, deoxygenation, and decarboxylation reactors are widely utilized in the chemical and petroleum-based fuel sectors [8]. Theoretical modeling has been used to study the reaction mechanism of molecules formed from biomass on the surface of metal particles. The development of multifunctional catalysts that enable the intensification of processes has led to significant improvements in efficiency and product yields [9]. Catalyst modification considers the effects of water, porosity, hydrophilic, acidic properties of supports, and catalyst deactivation caused by organic pollutants found in bio-sourced raw materials [10].

Two key processes for the biomass valorization process are esterification reactions and catalytic selective oxidation. Alcohols and other oxygen-containing molecules generated from biomass can be selectively oxidized to produce useful compounds, including aldehydes, ketones, carboxylic acids, and esters [11]. However, esterification makes it easier to produce esters from acids and alcohols obtained from biomass. These esters can be used in a variety of industrial applications, such as flavouring agents, solvents, and precursors for biodiesel [12]. Demand for sustainable and renewable alternatives to fossil fuels has increased due to the global depletion of fossil fuels. These alternatives are used to produce energy, chemicals, and fuels [13]. Although solar, wind, hydropower, and geothermal energy are examples of renewable energy sources, biomass is the only readily accessible renewable source of carbon needed for producing fuels and chemicals [14]. Currently, the majority of industrial processes rely on fossil fuels like petroleum and other fossil resources. However, there is more than enough biomass available right now to meet the needs of the modern chemical industry in terms of raw materials [15].

Review articles on bio-sourced chemicals have been published in large quantities. A wide variety of biomass feedstocks and reaction types are covered in these articles. They focus on specific feedstocks like cellulose, hemicelluloses and pentoses, glycerol, 5-hydroxymethylfurfural, carbohydrates, triglycerides, lignin, and lignocellulose [16]. A few review articles also discuss certain types of reactions, including oxidation, telomerization, hydrogenolysis/dehydroxylation, and metathesis [17]. Catalysts tailored to the biomass-to-chemical value chain have received relatively less attention. Besides these pure chemicals, consideration will also be given to metal-catalyzed processes that yield a mixture of chemicals that can be utilized to produce high-tonnage end products, such as plasticizers, paints, foams, resins, surfactants, and paper additives [18].

This chapter focuses on the application of both noble and non-noble metal catalysts to the selective oxidation and esterification of chemicals produced from biomass. It discusses the fundamentals of catalyst design, reaction mechanisms, and process optimization for the effective conversion of biomass to value-added products.

Catalyst Design and Synthesis

The conversion of biomass into value-added chemicals is a sustainable solution to environmental and resource challenges, with heterogeneous solid base catalysts playing a key role in oxidation processes. These catalysts are valued for their efficiency, reusability, and selectivity, making their design crucial for optimizing reactions. Factors, such as active site selection, support materials, and properties like porosity and thermal stability, significantly impact the performance of catalysts [19]. Enhancements in catalytic efficiency are achieved through the incorporation of alkali metals, rare-earth elements, or metal oxides. Advanced synthesis techniques, such as sol-gel and hydrothermal methods, enable precise control over catalyst structure. Innovations like bifunctional active sites and nanostructures further enhance reaction rates and selectivity for products like lactic acid, vanillin, and HMF. Developing cost-effective and eco-friendly catalysts is essential for advancing biomass oxidation on an industrial scale [20]. Gold (Au), platinum (Pt), and palladium (Pd) are noble metals known for their excellent conversion efficiency and selectivity across various oxidation processes. These metals can activate oxygen-containing functional groups in biomass-derived molecules, converting them into useful products [21]. Transition metals, such as nickel (Ni), cobalt (Co), and iron (Fe), have been widely explored as non-noble metal catalysts for biomass valorization due to their cost-effectiveness and abundance compared to noble metals [22, 23]. The preparation of these catalysts involves diverse methods, including deposition-precipitation, impregnation, and sol-gel techniques [24]. To enhance catalytic performance, these materials are

CHAPTER 5

Solid Base Catalysts in Multicomponent Reactions: A Green Pathway to Heterocyclic Compounds

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Abstract: Solid base catalysis has emerged as a promising strategy for driving Multicomponent Reactions (MCRs) in organic synthesis. MCRs, involving more than two starting reagents, occur under specific reaction conditions, yielding a single product embodying key features of the starting materials. These reactions play a crucial role in modern drug discovery, offering a rich origin of molecular variation and enabling quick, automatic, and scalable production of organic molecules. Solid base catalysts can facilitate various reactions, including condensation, rearrangement, and hydrogenation, making them valuable across different industries. This chapter provides a comprehensive overview of recent trends in solid base-catalyzed MCRs for eco-compatible heterocyclic synthesis, emphasizing their potential to advance environmentally conscious and highly effective synthetic methodologies in organic chemistry.

Keywords: Catalysis, Heterocycle, Multicomponent reactions, Solid base catalyst.

INTRODUCTION

The history of solid base catalysts is intertwined with the broader development of heterogeneous catalysis and the quest for more efficient and sustainable chemical processes.[1-3] While the concept of solid base catalysis has ancient roots, its systematic exploration and application in modern chemistry began to gain momentum in the 20th century [4]. Early investigations into solid base catalysis primarily focused on metal oxides and hydroxides, which exhibited basic properties due to the presence of surface oxygen ions. One of the pioneering studies in this field dates back to the 1920s when researchers explored the catalytic properties of metal oxides such as MgO (Magnesium Oxide) and CaO (Calcium Oxide) [5, 6]. Through the basic sites of metal oxides on their surface,

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various organic reactions, including aldol condensation, Michael addition, and Knoevenagel condensation, were promoted [7, 8].

A new class of solid base catalysts emerged in the 1950s and 1960s due to the advent of zeolites [9-11]. Zeolites are crystalline aluminosilicate distinctive materials with nanoporous structures and high surface areas. The presence of basic sites within the zeolite framework, typically associated with bridging oxygen atoms, endowed them with solid base catalytic properties [12-14]. Zeolites proved to be efficient catalysts for an extensive scope of reactions, counting isomerization, alkylation, and cracking, in both petrochemical and fine chemical industries [15, 16]. In the following decades, researchers continued to explore and expand the repertoire of solid base catalysts. Metal oxides, like hydrotalcite (layered double hydroxides) and mixed metal oxides, gained attention for their tunable basicity and catalytic activity in various organic transformations [17]. Mesoporous resources, like mesoporous silica and metal oxide, also emerged as promising solid base catalysts due to their large surface areas and interstitial volumes, which facilitated efficient mass transfer and catalytic activities [18-23].

The development of nanoporous materials and advances in nanotechnology further propelled the field of solid-base catalysis [24]. Nanostructured catalysts, including metal nanoparticles supported on solid bases, exhibited enhanced catalytic performance and more selectivity when compared to their larger equivalents [25]. The design and synthesis of tailored solid base catalysts with specific pore structures, surface chemistries, and functionalities became a research focus for optimizing catalytic efficiency and selectivity in various chemical processes [26, 27].

In recent years, the application of solid base catalysts has extended beyond traditional organic synthesis to areas such as biomass conversion, environmental remediation, and renewable energy [28-33]. Solid base catalysts perform a key role in promoting sustainable and environmentally friendly procedures by enabling the efficient utilization of renewable feedstocks, reducing energy consumption, and minimizing waste generation [34-36].

Recognition of certain materials as solid base catalysts is based on the following key factors [4, 37-40].

1. **Surface characterization:** Various analytical methods, including changes in the color of acid-base indicators, acidic molecule adsorption, and spectroscopic techniques (such as UV, IR, XPS, ESR, *etc.*), detect the occurrence of basic sites on the surfaces of these substances.
2. **Correlation with catalytic activity:** The effectiveness of these materials in catalysis shows a clear connection to the quantity and potency of basic sites

they possess. Moreover, the active sites can be negatively impacted by acidic molecules, including hydrochloric acid, water, and carbon dioxide, rendering them less effective.

3. **Comparable activity to homogeneous catalysts:** These materials demonstrate catalytic behaviors akin to those observed in homogeneous basic catalysts involved in "base-catalyzed reactions" within homogeneous reactions. The proposed mechanistic pathway for reactions taking place on their surfaces closely mimics those found in homogeneous basic solutions.
4. **Insight into mechanism:** Research into reaction mechanisms, yield distributions, and surface species spectral analysis suggests the participation of anionic intermediates in these reactions.

The list of some common solid-base catalysts used in organic synthesis is given below:

1. Alkali metal hydroxides (*e.g.*, KOH, NaOH)
2. Carbonates of alkali metals (*e.g.*, K_2CO_3, Na_2CO_3)
3. Hydrides of alkali metals (*e.g.* KH, NaH)
4. Alkali metal alkoxides (*e.g.*, KO^{t}Bu, NaOEt)
5. Alumina (aluminum oxide)
6. Silica gel
7. Zeolites (*e.g.*, zeolites (alkali ion-added): Cs_2O/Cs–X, Zeolites (alkali ion-exchanged): Na–X, Cs–X, imide and nitride of lanthanide on zeolite)
8. Ion exchange resins
9. Metallic oxides (*e.g.*, MgO, CaO, and BaO).
10. Clay minerals (*e.g.*, hydrotalcite, chrysolite, sepiolite)
11. Non-oxide (*e.g.*, KF impregnated on alumina, metal oxynitrides: VALPON ALPON)
12. Nanoparticles, *etc*

These solid base catalysts are often employed in various organic transformations, including multicomponent reactions, due to their ability to facilitate reactions efficiently under mild conditions [41-45].

Sr No.	Solid Base Catalysts	Liquid Base Catalysts
1	Neutralizing the reaction mixture is unnecessary, reducing waste generation.	In liquid base-catalyzed reactions, the necessity to neutralize the base results in waste production.
2	High-temperature reactions are possible without limitations.	Liquid base-catalyzed reactions are limited to temperatures below boiling point due to solvent use.

CHAPTER 6

Historical and Recent Advances in Solid Base Catalysts for Drug Synthesis

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Abstract: Pines and Haag were the first to demonstrate the use of heterogeneous base catalysts in the isomerization of alkenes using sodium metal dispersed on alumina. Following this, a number of work has been performed on solid bases as catalyst materials. Metal oxides, zeolites, supported alkali metal complexes, clay minerals, waste solid base catalysts, mesoporous solid base catalysts, *etc*., are examples of solid base catalysts. Precipitation, co-precipitation, sol-gel, hydrothermal, impregnation, vapor phase deposition, and sonochemical techniques can all be used to create them. The majority of materials referred to as solid bases exhibit catalytic activity when water and carbon dioxide are removed from their surfaces. The degree of pre-treatment conditions influences the surface basic sites' characteristics. In addition to eliminating carbon dioxide and water, pre-treatment involves the rearrangement of bulk and surface atoms, which modifies the kind and quantity of basic sites as the pre-treatment temperature rises. Double bond isomerization, the addition of anion and proton to various double bonds and alcohol decomposition, hydrogenation, amination, dehydrocyclodimerization, aldol addition, nitroaldol reaction, michael addition, conjugate addition of alcohol, cyanoethylation, and Tischenko reaction are among the reactions that solid base catalysts can catalyze. Solid base catalysts play a crucial function in industrial processes due to their varied application.

Keywords: Alcohol decomposition, Catalytic activities, Conjugate addition of alcohol, Cyanoethylation, Double bond isomerization, Heterogeneous base catalyst, Isomerization of alkenes, Sodium metal dispersed on alumina, Solid base catalysts, Tischenko reaction.

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INTRODUCTION

The development of efficient and sustainable catalysts is a crucial aspect of modern organic synthesis for various industrial processes, including the pharmaceutical industry [1]. Traditional acids and bases catalysts pose significant environmental issues due to their toxicity, waste generation, and high energy consumption. As a result, there is a growing need for eco-friendly and sustainable chemical processes, driving researchers to seek out effective and environmentally friendly replacement of conventional acids and bases in reactions [2]. Reducing environmental impact and boosting sustainability in organic processes requires substituting solid catalysts for liquid acids and bases [3]. Solid base catalysts have emerged as a promising class of materials, offering advantages such as recyclability, reusability, and reduced environmental impact [2]. This highlights the significance of catalysts in enabling efficient and sustainable production of various essential products, including medicines, that are vital to our well-being and daily lives [4]. The most notable heterogeneous basic catalysts are typically derived from three main categories: alkaline earth metals, transition metals, and metal oxides. These classes of materials have shown significant potential in facilitating various chemical reactions as effective heterogeneous basic catalysts [5]. The ability of solid base catalysts to effectively catalyze the isomerization of unsaturated molecules containing heteroatoms, such as N and O, gives them an edge over solid acid catalysts. Solid base catalysts are a useful option for these particular reactions since they are less prone to deactivation (or poisoning) when exposed to heteroatom-containing reactants, compared to solid acid catalysts [6].

Catalysts

A catalyst is a material that, depending on the reaction, can speed up or slow down a reaction's pace, while staying in the same composition once the reaction is complete. It modifies the reaction's activation energy without changing the equilibrium. Catalysts are effective for reactions that are thermodynamically feasible, such as those with a negative change in Gibb’s free energy under the given reaction conditions. When selecting a catalyst for a reaction, the focus often lies in its ability to influence, rather than enhance, the reaction rate. These principles apply universally across various types of catalytic reactions, whether they are homogeneous, heterogeneous, or enzymatic. Catalysts are commonly classified as either solid acid or solid base catalysts [7].

Solid Base Catalyst

A solid base catalyst is a type of catalyst that operates by donating electrons and displaying basic properties due to the presence of exposed oxygen atoms on its surface. These oxygen atoms interact attractively with Hydrogen ions (H^+), enabling the abstraction of H^+ from strongly acidic molecules [8].

Solid Base Catalysts Preparation Methods

Precipitation and Co-precipitation

This method involves separating the desired substance from a uniform mixture by inducing precipitation. The precipitation process is initiated by manipulating temperature conditions and introducing acidic or alkaline solutions, causing the substance of interest to precipitate out of the solution, allowing for its isolation and collection. Various factors that influence the formation of the precipitate include temperature, pH levels, the composition of raw materials, type of solvent used, *etc*. As a result, the transition from a liquid solution to a solid state unfolds in four distinct stages [9].

Sol-Gel Method

The first step in the sol-gel process is the formation of a stable colloidal solution (sol) that has very pure finely distributed particles. The sol is formed by hydrolysis and partial condensation of the precursor, and then it passes through polycondensation to produce a gel. Evaporation is used to remove extra liquid from the gel, producing a dry product called a xerogel. After that, this xerogel is heated to enhance its qualities and provide the finished product with the necessary features [10].

Hydrothermal Method

In a hydrothermal process, a homogeneous aqueous solution, including alkali and precursor chemicals, is heated to a high temperature in a sealed autoclave for a predetermined amount of time. This controlled environment facilitates chemical reactions and material formation, allowing for the synthesis of unique materials with specific properties. The materials dissolve and then reform into crystals under the influence of the elevated temperature and pressure. The resulting solid material is recovered through filtration, which is then rinsed and subsequently dried [10].

Impregnation Method

The impregnation technique involves applying aqueous precursors to a support material, typically an oxide support. The process entails:

1. Contacting the support with a solution containing precursors of the active catalyst elements.

CHAPTER 7

Historical and Recent Advances in Solid Base Catalysts for C-C, C-N, C-S Bond Formation

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Abstract: The formation of C-C and carbon-heteroatom (N and S) represents a fundamental process in organic chemistry, playing a pivotal role in synthesizing a wide array of organic compounds, with applications spanning from pharmaceuticals to materials science. Conventional catalytic approaches for C-C bond formation frequently depend on costly and toxic transition metal catalysts, which raise environmental and economic apprehensions. However, with the advent of metal oxides, ion-exchange resins and graphene-based carbocatalysts present a promising alternative owing to their distinctive attributes, chemical stability, conductivity, and high surface area. In environmentally sustainable and cost-effective processes, this catalytic method efficiently enables the formation of a wide range of C–N and C–S containing compounds across diverse organic synthesis pathways. This book chapter emphasizes the advancement of carbon-carbon and carbon-heteroatom (N & S) formation through various types of solid base catalysts.

Keywords: C-C bond formation, C-N bond formation, C-S bond formation, Coupling reactions, Solid base catalysis.

INTRODUCTION

Solid base catalysts have emerged as essential components in contemporary catalysis, providing environmentally friendly alternatives to conventional acidic catalysts [1]. In contrast to their liquid counterparts, solid base catalysts offer numerous benefits, including ease of separation, recyclability, and reduced environmental footprint [2]. Solid base catalysts are characterized by the existence of basic sites on a solid support [3]. The active sites on solid base catalysts generally comprise basic functional groups, such as hydroxides, oxides, or amine groups, which can accept protons or donate electrons during catalytic reactions [4]. Common materials utilized as solid bases include metal oxides such as MgO,

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CaO, and Al_2O_3, along with various porous materials, such as zeolites and mesoporous silica [5 - 7]. Solid base catalysts facilitate chemical reactions by abstracting protons or donating electron pairs to substrates, thereby initiating catalytic cycles [8]. The basicity of a solid base catalyst is defined by its capability to adsorb and activate reactant molecules, resulting in the formation of intermediates with modified reactivity [9]. Commonly used solid base materials include metal oxides, zeolites, layered double hydroxides, and supported organic bases, each offering unique advantages depending on the specific reaction requirements [10 - 12]. The preparation of solid base catalysts involves chemical composition and customizing the surface properties to enhance catalytic performance. Techniques like impregnation, precipitation, sol-gel, and ion exchange are utilized to deposit basic species onto support materials, or to modify the surface chemistry of the catalyst [13].

Additionally, these catalysts participate in a crucial role in biomass conversion processes, including the production of biodiesel and the upgrading of biomass-derived intermediates into value-added products [14]. Moreover, solid base catalysts find applications in environmental endeavors, such as pollutant degradation, acid gas removal, and water purification, playing a role in reducing environmental pollution, and promoting sustainability [15]. Solid base catalysts are integral to green and sustainable chemistry, providing versatile tools for facilitating efficient, selective, and environmentally benign chemical transformations [16 - 18].

Solid base catalysts facilitate the synthesis of C-C/C-heteroatoms (N and S), which is an essential procedure in the synthesis of many organic molecules with uses in materials science and medicine (Fig. **1**).

Solid base catalysts play a crucial role in the formation of C-C bond reactions, which are primary in organic synthesis for constructing complex molecules. These catalysts facilitate various types of C-C bond formations, including Knoevenagel condensation, aldol condensation, Michael addition, Claisen condensation, and more. Solid base catalysts are involved in some of these reactions and transesterification, enabling the synthesis of pharmaceuticals, biofuels, and fine chemicals [19, 20]. Aldol condensation is a process that produces a *β*-hydroxy carbonyl molecule by condensing two carbonyl compounds with the help of a base catalyst. Solid base catalysts, such as metal oxides (like CaO, MgO) or amine-functionalized materials, which are supported amines on silica, are commonly employed to catalyze this reaction. They deprotonate the α-carbon of one carbonyl compound, enhancing its reactivity towards nucleophilic attack [21]. In the Claisen condensation, two ester molecules undergo condensation to produce a *β*-keto ester with the help of a base catalyst. Solid base catalysts, such as alkali earth metal oxides or alkaline, facilitate this reaction by extracting a proton from one of the ester molecules, thereby enabling nucleophilic attack by the carbonyl group of the other ester [22]. In the Michael addition, a new carbon-carbon bond is formed when a nucleophile is added to an *α,β*-unsaturated carbonyl molecule. Solid base catalysts, such as metal oxides and supported amines, can catalyze Michael addition reactions via activating the nucleophile, and aiding its addition to the electrophilic double bond [23, 24].

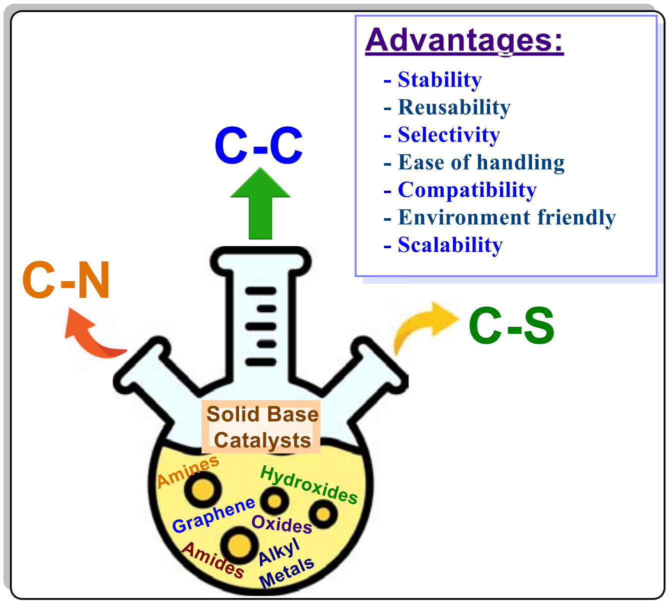

Fig. (1). Solid base catalyzed formation of C-C and C-heteroatoms bond.

In chemistry, a "C–N coupling" refers to a chemical reaction in which a carbon atom forms a bond with a nitrogen atom" [25, 26] In C-N bond formation processes, solid base catalysts have shown great promise as a viable substitute for conventional homogenous catalysts [27, 28]. There are various benefits to solid base catalysts over their liquid equivalents, because they are sustainable, stable, and allow for facile separation [29, 30]. The solid catalyst basic sites aid in the deprotonation process, and the follow-on nucleophile is able to interact with different electrophiles to generate the appropriate C-N bond [31]. Current developments in the sphere of solid base catalysis for the production of carbon-nitrogen bonds have centered on creating new types of catalysts, increasing catalytic efficiency, and broadening the variety of substrates that can be engaged [32 - 34].

CHAPTER 8

Advanced Solid Base Catalysts for Depolymerization of Lignin and Transesterification of Glycerol

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Abstract: Due to environmental concerns and the depletion of fossil resources, the sustainable utilization of biomass for producing valuable chemicals and fuels has gained substantial attention in recent years. Advanced solid base catalysts have emerged as essential tools in this attempt, with high catalytic efficiency, improved selectivity, and low environmental impact. The present chapter provides an in-depth exploration of various advanced solid base catalysts like metal oxides, alkali metal-supported metal oxides, alkaline earth metal oxides, waste solid base catalysts, clay materials, and mesoporous solid base catalysts, *etc*., for transforming biomass and its derivatives into value-added products. Specific processes covered include transesterification of oils, aldol condensation of biomass-derived compounds, depolymerization of lignin, glycerol transesterification for carbonate production, and so on.

Keywords: Aldol condensation, Glycerol carbonate, Glycerol, Lignin depolymerization, Oils, Solid base catalysts, Transesterification.

INTRODUCTION

Feedstocks derived from biomass can be used to produce renewable chemicals and biofuels. They are commonly categorized into three primary groups: lignocellulose, starch, and triglycerides. Lignocellulosic biomass is particularly favored due to its non-competitive nature with food supplies. The conversion process involves various catalytic or chemical treatments, including pyrolysis,

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hydrothermal treatment, liquefaction, hydrolysis, and fermentation. These treatments yield liquid fractions rich in oxygenated molecules like alcohols, aldehydes, acids, and furans, serving as crucial molecules for synthesizing a variety of chemicals and fuels, such as additives, solvents, and monomers.

Currently, there is a predominant focus on converting biomass into liquid fuels, with less emphasis on chemical and fine chemical production. However, the evolving bio-economy emphasizes the importance of co-producing biofuels alongside valuable chemicals. To address this shift, innovative processing strategies for second-generation biomass at unified biorefineries are being proposed. These strategies involve primary treatment, fractionation, and separation, followed by downstream processing into fuels, fuel additives, and chemicals.

In response to the demands of the evolving bio-economy, the development of novel catalytic methods is crucial for competitive and efficient production from biomass. While homogeneous catalyst systems have been traditionally utilised for biomass conversion due to cost-effectiveness, there is a notable shift towards exploring heterogeneous catalysts [1]. Solid catalysts offer advantages, such as reusability and the absence of a neutralization step, minimizing waste product formation, representing a significant avenue for environmentally conscious biomass utilization.

Heterogeneous catalysts, particularly solid base catalysts, have the ability to tailor active properties for achieving selective products, making them promising candidates for executing multistep processes. These catalysts enable the creation of robust multifunctional catalysts, facilitating collaboration among active sites across different stages of a given cascade process [2, 3].

In contemporary applications, solid base catalysts, including alkali-modified zeolites and various metal oxides, are widely employed for base-catalyzed reactions in biomass transformation. Aldol condensation, a crucial C—C bond-forming reaction, plays a significant role in biomass utilization for chemical and fuel production. Despite the high hydrogen consumption in deoxygenation steps, aldol condensation allows for the selective synthesis of linear alkanes suitable for liquid fuels.

While approximately 90% of biodiesel is obtained through the transesterification of vegetable oils *via* homogeneous bases as catalysts, the drawbacks in separation and purification steps have led to a growing interest in heterogeneous catalysts. Solid base catalysts, in particular, play a pivotal role in enhancing biodiesel production. This chapter extensively discusses and examines the transesterification of oils and glycerol, depolymerization of lignin, and aldol condensation using solid base catalysts.

CLASSIFICATION, CHARACTERIZATION, AND PROPERTIES OF BIOMASS FEEDSTOCKS

Classification of Biomass

Biomass feedstocks can be broadly classified into three main categories, which are lignocellulosic, animal, and aquatic biomass. Lignocellulosic Biomass (LCB) is the most common and widely researched category, consisting of plant-derived materials, such as wood, crop residues, and dedicated energy crops. Animal biomass includes organic waste from animals, such as manure. In contrast, aquatic biomass refers to materials obtained from aquatic sources like algae and aquatic plants. Lignocellulosic biomass is primarily composed of cellulose, hemicellulose, and lignin, and its composition is shown in Table **1**.

Table 1. Composition of common LCB [4, 5].

Lignocellulosic Biomass (LCB) Material	Cellulose %	Hemicellulose %	Lignin %
Hardwood stems	40–55	24–40	18–25
Softwood stems	45–50	25–35	25–35
Almond shells	29.0–31.1	28.0–38.0	27.7–35
Corncobs	45	35	15
Grasses	25–40	35–50	10–30
Wheat straw	30	50	15
Flax straw	36.70	34.40	28.90
Leaves	15–32	80–85	0
Sugar cane bagasse	32–44	27–32	19–24
Coastal Bermuda grass	25	35.7	6.4
Switch grass	45	31.4	12

Characterization of Biomass Feedstocks

Characterization of biomass feedstocks involves understanding their physical, chemical, and thermal properties [6]. Key physical characteristics include particle size, density, and moisture content, which influence processing. Additionally, chemical analysis examines elemental composition and energy content. Thermal

CHAPTER 9

Solid-Supported Base Catalysis for Carbon-Carbon Bond Formation

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Abstract: Solid-Phase Organic Synthesis (SPOS) has gained prominence after the pioneering work of Merrifield for the development of peptide synthesis using solid-phase catalysis was published in the 1960s. Since then, chemists have shown tremendous interest in expanding this field to develop environment-friendly, cost-effective, and sustainable protocols for synthesizing diverse compounds used in material synthesis. The major emphasis of solid base synthesis is on the growth and emergence of sustainable and green chemistry. This approach largely relies on converting solution-phase reactions into solid-phase processes. Key green chemistry principles that support solid-phase synthesis include reduced use of toxic solvents, fewer reaction steps, improved energy efficiency, and high atom economy. However, the solid-phase approach has high efficiency, excellent selectivity, easy work-up, and good dispersion of active reagent sites. Solid-supported basic catalysis has advanced significantly over the last few decades, and it is widely used in the synthesis of organic molecules, bench-top catalysts, and fine chemicals. An overview of solid basic catalysts and their application in the synthesis of important organic molecules was disclosed in this chapter. The initial section discussed the importance and synthesis of a variety of supported solid base catalysts. The second part discusses the application of solid base catalysts in various organic reactions and evaluates their catalytic performance.

Keywords: Aldol condensation, Coupling reactions, Hydrogenation, Knoevenagel condensation, Michael addition, Solid-supported catalyst.

INTRODUCTION

Catalysis attracts attention due to its wide applicability in numerous chemical production from small scale in research laboratories to large industrial scale [1]. Catalysis has high importance in the manufacturing of our daily life essentials like medicines, polymers, fuels, automotive and so on [2]. Homogeneous catalysts usually dominate over heterogeneous catalysts because of their high solubility and

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reactivity with reactants [3]. In this regard, the main challenge faced by the chemist is to purify the desired product, which is difficult to separate from the catalysts. To overcome this difficulty, an alternative approach has been adopted by designing novel catalysts to replace homogeneous catalysts. Chemists have turned to heterogeneous catalysts, which are easier to separate and recycle. In addition to this, heterogeneous catalysts are easily removed from the product, and their reaction requires a minimum quantity of toxic organic solvents [4]. Thus, approaches using this catalyst are environmentally benign for small-scale synthesis as well as for industrial-level synthesis of organic compounds. The increasing demand for sustainable and green chemistry also led scientists to design new strategies using heterogeneous catalysts instead of conventional homogeneous catalysts.

Among various heterogeneous catalysts, solid base catalysts have attracted more attention due to their valuable properties over their liquid counterparts. Importantly, the solid bases are much less corrosive and cause fewer disposal problems. Solid base-catalyzed reactions generally show higher selectivity towards the targeted product. Further, solid bases are easier to separate from the product and make the recovery of products from reaction systems faster, and subsequently it become facile to recycle the catalysts. Therefore, solid bases provide an environmentally benign and economical pathway for the synthesis of organic compounds [5]. Consequently, the major concern of chemists is to develop heterogeneous catalyst-based methodologies and design new solid base catalysts by altering the solid support along with the structural and morphological characteristics of the catalysts during the reaction.

Pioneering work on solid base catalysts was reported by Pines and Haag in 1958. They designed the new catalyst system by the dispersion of metal Na on Al_2O_3 and evaluated its efficiency in various organic reactions [6]. The results of this catalyst system were amazing enough to encourage the organic chemist to expand new ventures in this field. During the last two decades, solid-phase synthesis has gained tremendous interest among chemists due to its wide applicability [7]. Now, sustainable chemistry has been in demand for the synthesis of a myriad of chemicals through the environment-benign approach, which was used in material chemistry and catalysis. The novel solid-supported basic catalysts, which range from single to multicomponent metal hydroxides as well as oxides (*e.g.*, hydrotalcite), have vast applications in various chemical processes. Till now, the size, texture, morphology, category, and surface area of the particles have been the determining factors for the research on solid base catalysts [8]. The structure and characteristics of alkaline sites on the surface of the developed solid base catalyst have proved its catalytic behavior [9].

The chapter provides a concise outline of the progression in the construction and designing of novel solid basic catalysts as well as their use in a variety of organic reactions such as Knoevenagel reaction, Aldol condensation, Michael addition, double bond migration, transesterification, and hydrogenation (Fig. **1**). This section provides a detailed description of the structure and design of solid base catalysts, along with their controlled synthesis and broad application in various organic reactions. The content described in this chapter will influence the synthetic chemist towards the development of novel solid basic catalysts and it is anticipated that this will open new ventures in the field of sustainable chemistry.

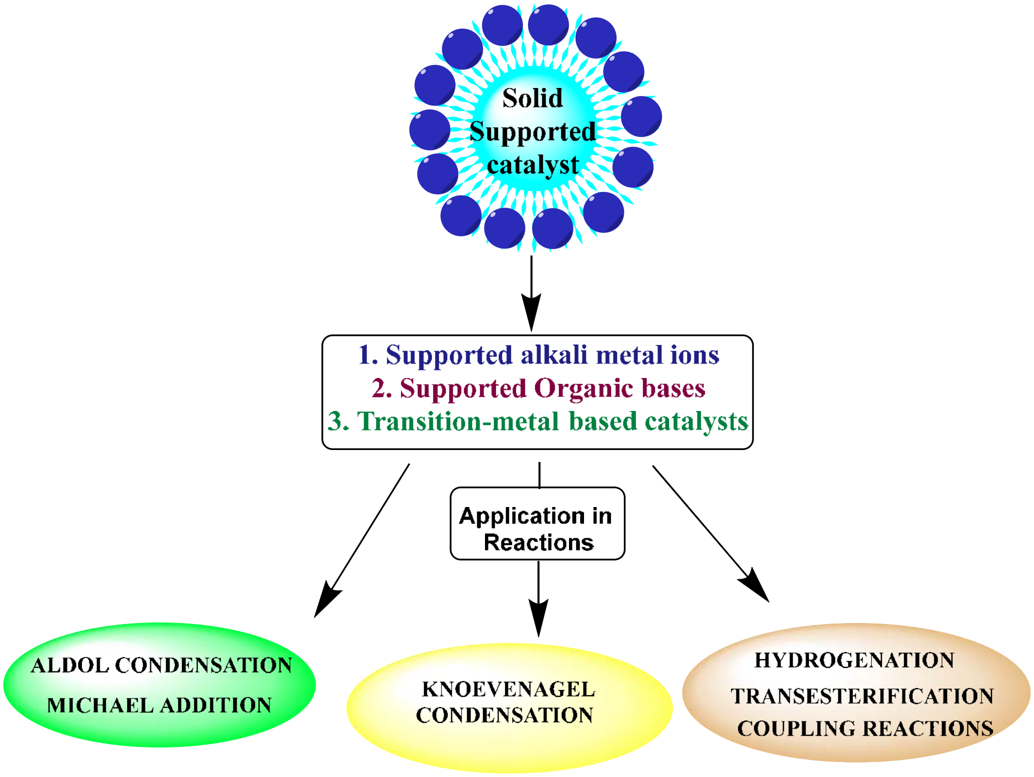

Fig. (1). General overview.

SUPPORTED SOLID BASIC CATALYST

In heterogeneous base catalysis reactions, supported solid basic catalysts play a crucial role. Alkali and alkali earth metals have some basic functional groups incorporated in them, which behave as potent bases and nucleophiles. These metals can also work as solid catalysts or as initiators of anionic chain reactions. In a solid basic catalyst, the basic strength of a solid surface is defined as the ability of the surface to convert an adsorbed neutral acid to its conjugate base

CHAPTER 10

Recent Advances in Solid Base Catalysis for Reduction Reactions

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Abstract: The demand for solid base catalysts has led to the development of green catalytic processes, owing to their advantages such as easy separation, low corrosion, and eco-friendliness. Over the last decade, substantial advancements have occurred in crafting these catalysts, amplifying their utility in organic synthesis, fine chemical production, and environmental catalysis. Consequently, propelling the progress of solid basic catalysts is crucial for both academic exploration and industrial implementation. The book chapter delves into the pivotal role of solid bases in organic reactions, particularly emphasizing their increasing usage in organic synthesis due to their superiority over liquid bases. Solid bases offer easier disposal, simplified separation and retrieval of products, solvents and catalysts, along with being non-corrosive. Moreover, they enable base-catalyzed reactions without the necessity of solvents and even in gas-phase conditions, thereby expanding the scope for discovering innovative reaction systems. Through numerous illustrative examples, this chapter delineates the significant impact of solid base catalysis in reduction reactions. Additionally, it sheds light on recent advancements and future prospects concerning solid base catalysts in reduction reactions.

Keywords: Catalyst, Eco-friendly, Effortless separation, Non-corrosive, Reduction, Solid base.

INTRODUCTION

While extensive research has focused on solid acids, relatively less attention has been directed towards heterogeneous basic catalysts, also known as solid base catalysts. The first pioneering investigation into heterogeneous base catalysts was conducted by Pines and Haag [1].

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They demonstrated that sodium metal dispersed on alumina effectively catalyzed the isomerization of double bonds in alkenes.

In 1972, Tanabe and his team discovered that calcium oxide and magnesium oxide showed a marked increase in catalytic activity for the isomerization of 1-butene, especially after undergoing vacuum pretreatment. This study emphasized the critical role of preparation techniques and pretreatment procedures for base catalysts [2, 3].

Additionally, various techniques were developed to assess the basic characteristics of solid surfaces and to identify reaction intermediates. In 1989, Tanabe and his team released “New Solid Acids and Bases,” summarizing the progress made in this field during the 1970s and 1980s [4].

Carbanions are essential intermediates in many organic reactions, such as isomerization, additions and condensations. They generally form when a base abstracts a proton from a C-H bond in an organic molecule, typically in homogeneous phases.

These organic transformations frequently necessitate a stoichiometric quantity of liquid bases to facilitate the generation of carbanions. However, this process also produces a stoichiometric amount of metal salts as by-products.

There is no necessity for employing a stoichiometric quantity of base compounds. Another notable advantage of solid bases compared to homogeneous bases is the reduced need for solvents. In homogeneous phase reactions, the requirement for both the base catalyst and reactants to be soluble in the same solvent limits the range of suitable solvents. Consequently, this often leads to the use of hazardous solvents like methyl chloride and dimethyl sulfoxide (DMSO) [5].

In reactions catalyzed by solid bases, solvents should only dissolve reactants (and products), allowing for a much wider range of solvent choices. The effects of solvents in solid-base catalyzed reactions differ from those in homogeneous reactions. No waste is produced from solvents like aqueous solutions with organics or organic solvents with alkaline water. Additionally, simplified separation processes reduce energy consumption. Moreover, solid bases can be reused after separation, improving atom economy in catalyst preparation. The absence of solvent selection constraints also increases the potential for discovering new reactions. Bases such as sodium hydroxide create corrosive solutions, necessitating corrosion-resistant materials for reactors and disposal systems. In contrast, this problem is typically absent in systems that use solid base catalysts.

The conversion of homogeneous processes to heterogeneous ones using solid-base (or solid-acid) catalysts is especially significant in the synthesis of fine chemicals and pharmaceuticals. This is because these sectors often produce more waste per unit mass of the desired product when employing stoichiometric or homogeneous catalytic processes. The advantages described earlier make this transformation essential in improving environmental sustainability and efficiency in these industries.

Moreover, solid base catalysts often achieve high activities and selectivity across a variety of reactions. Unlike acid-catalyzed reactions, reactions over basic catalysts typically avoid issues like cooking, which can be a significant problem. Furthermore, functionalized organic compounds like amines interact more with basic sites than with acidic sites. Consequently, these molecules are less likely to be adsorbed onto the surface and can desorb more readily. Consequently, reactions involving these molecules tend to proceed more smoothly over solid bases.

Organometallic compounds, such as Grignard reagents and alkyl lithiums, donate carbanion-like species, facilitating the formation of Si-C bonds through various reaction mechanisms.

$$R_3SiX + R'MgX \longrightarrow R'\text{-}SiR_3 + MgX_2$$

$$R_3SiX + R'Li \longrightarrow R'\text{-}SiR_3 + LiX$$

Once again, the use of organometallic reagents in stoichiometric quantities is necessary. However, solid bases provide alternative pathways to produce the same products in a more straightforward and environmentally friendly manner.

$$H_2SiEt_2 + {}^tBuC{\equiv}CH \longrightarrow {}^tBuC{\equiv}C\text{-}SiEt_2H + H_2$$

$$H_2SiEt_2 + C_6H_5CH_3 \longrightarrow C_6H_5CH_2\text{-}SiEt_2H + H_2$$

These two reactions proceed smoothly in the presence of catalytic systems such as KF/Al_2O_3 and KNH_2/Al_2O_3, respectively [6, 7].

The following are four key reasons certain materials are recognized as solid base catalysts:

1. Surface characterizations employing various methods, including changes in acid-base indicators, adsorption behavior towards acidic molecules, and spectroscopic techniques (such as UV, IR, XPS, ESR), consistently reveal the

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