THE USE OF ELECTROCATALYSIS AS A TOOL FOR ORGANIC SYNTHESIS

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ABSTRACT

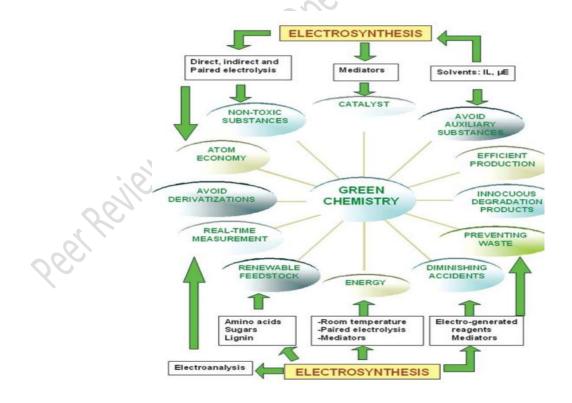
Electrocatalysis has emerged as a powerful tool in organic synthesis, offering new avenues for efficient and sustainable transformations. This review explores the use of electrocatalysis as a versatile methodology to drive organic reactions and highlights its potential in addressing challenges in synthetic chemistry. Electrocatalysis involves the use of catalysts to facilitate electron transfer processes, enabling the activation of organic molecules and the promotion of selective bond formation. The review discusses different types of electrocatalysts, including transition metals, metal complexes, and organic catalysts, and their applications in various organic transformations. Key aspects of electrocatalysis, such as the design of catalytic systems, electrode materials, and reaction conditions, are examined. The use of different electrochemical techniques, such as voltammetry and electrolysis, is also discussed, highlighting their significance in controlling reaction pathways and achieving high selectivity. The review presents a comprehensive overview of the applications of electrocatalysis in organic synthesis, including C-C and C-X bond formation, oxidation and reduction reactions, and functional group transformations. It showcases the ability of electrocatalytic methods to access challenging chemical transformations with high efficiency and selectivity.

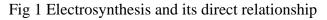
INTRODUCTION

Organic synthesis plays a pivotal role in the development of new pharmaceuticals, agrochemicals, and functional materials. Traditional organic synthesis often relies on the use of stoichiometric reagents and harsh reaction conditions, resulting in issues of waste generation, energy consumption, and environmental impact. In recent years, the field of catalysis has witnessed significant advancements, offering more efficient and sustainable alternatives for organic transformations. Electrocatalysis has emerged as a powerful tool within the realm of catalysis, utilizing electrical energy to drive chemical reactions. It involves the use of catalysts to facilitate electron transfer processes, enabling the activation of organic molecules and promoting selective bond formation. By harnessing the power of electrochemistry, electrocatalysis offers

new opportunities for controlled and sustainable organic synthesis. One of the key advantages of electrocatalysis is its ability to operate under mild reaction conditions. The use of low potentials and room temperature or mild heating allows for the activation of organic substrates without resorting to high temperatures, strong acids or bases, or hazardous reagents. This not only improves the energy efficiency of the reaction but also enhances the selectivity, as milder conditions often lead to fewer undesired side reactions(Vereshchagin, A. N. et al,2008).

Electrocatalysis also enables the selective functionalization of organic molecules, facilitating the formation of challenging chemical bonds. Through careful selection and design of catalysts, electrocatalytic methods have been successfully applied in C-C and C-X bond formation, oxidation and reduction reactions, and diverse functional group transformations. This broad applicability highlights the versatility and potential of electrocatalysis in synthetic chemistry. Furthermore, electrocatalysis offers unique opportunities for sustainable synthesis. The ability to use renewable energy sources, such as solar or wind power, to drive electrochemical reactions provides an environmentally friendly and sustainable approach to organic synthesis. Additionally, the ability to perform reactions in water or other benign solvents further reduces the environmental impact of the process.





In this context, this review aims to provide an overview of the use of electrocatalysis as a tool for organic synthesis. It explores the different types of electrocatalysts, the various electrochemical techniques employed, and the applications of electrocatalysis in different organic transformations. The review also discusses the environmental benefits of electrocatalysis and the challenges and future prospects in the field. By harnessing the power of electrocatalysis, organic chemists can achieve more efficient, selective, and sustainable synthesis routes. The integration of electrocatalytic methods with other catalytic strategies, such as photocatalysis and enzymatic catalysis, further expands the toolbox for synthetic chemists. The continued development of electrocatalysis holds great promise for advancing the field of organic synthesis towards greener and more sustainable practices.

SCOPE OF THE RESEARCH

The scope of the research on the use of electrocatalysis as a tool for organic synthesis is broad and encompasses various aspects. Some key areas within the scope of this research may include(Wang, Y. et al,2008):

Electrocatalytic reactions: Investigating different types of organic reactions that can be facilitated or enhanced by electrocatalysis, such as C-C bond formation, C-X bond formation, oxidation reactions, reduction reactions, and functional group transformations. Exploring the mechanisms and reaction pathways involved in these electrocatalytic transformations.

Catalyst design and development: Exploring different types of catalysts, including transition metals, metal complexes, and organic catalysts, that can effectively promote electrocatalytic reactions. Investigating the factors influencing catalyst activity, selectivity, and stability, such as ligand design, coordination environment, and electronic properties.

Electrochemical techniques: Exploring various electrochemical techniques employed in electrocatalysis, including voltammetry, electrolysis, and photoelectrochemistry. Understanding the principles and parameters governing these techniques and their influence on reaction outcomes, selectivity, and efficiency.

Reaction optimization: Investigating the optimization of reaction conditions, such as electrode materials, potential range, solvent systems, and reaction parameters, to maximize the efficiency, selectivity, and scalability of electrocatalytic transformations. Exploring the use of

renewable energy sources, such as solar or wind power, for sustainable electrochemical reactions.

Mechanistic studies: Investigating the mechanistic aspects of electrocatalytic reactions, including electron transfer processes, intermediates, and reaction pathways. Utilizing techniques such as spectroscopy, electrochemistry, and computational methods to elucidate reaction mechanisms and understand the factors influencing selectivity and reactivity.

Synthetic applications: Exploring the practical applications of electrocatalysis in organic synthesis, including the synthesis of complex organic molecules, natural product synthesis, and functional material synthesis. Assessing the advantages and limitations of electrocatalytic methods compared to traditional synthetic approaches.

Sustainability and environmental impact: Evaluating the environmental benefits of electrocatalysis, such as reduced waste generation, milder reaction conditions, and the use of renewable energy sources. Assessing the sustainability of electrocatalytic processes in terms of atom economy, energy efficiency, and environmental footprint.

The scope of the research may also include discussions on challenges and future prospects in the field, such as catalyst design, scalability of electrochemical processes, integration with other catalytic strategies, and potential applications in industry and pharmaceutical synthesis.

Organic synthesis

Organic synthesis is the branch of chemistry concerned with the construction of organic compounds through chemical reactions. It involves the manipulation of organic molecules to create new compounds with desired properties and functionalities. Organic synthesis plays a vital role in various scientific disciplines, including medicinal chemistry, materials science, and agrochemical research.

The goal of organic synthesis is to design and execute efficient and selective routes to target compounds. It involves the selection of appropriate starting materials, the design of reaction sequences, and the optimization of reaction conditions to achieve the desired transformations. Organic synthesis often requires the use of reagents, catalysts, and specific reaction conditions to control the stereochemistry, regiochemistry, and overall efficiency of the synthesis.(Garjonyt, R. et al,1998).

Several methods and strategies are employed in organic synthesis, including:

Functional Group Transformations: Organic molecules can undergo a variety of chemical reactions to introduce or modify functional groups. Examples include oxidation, reduction, hydrolysis, esterification, and substitution reactions.

Carbon-Carbon Bond Formation: The construction of carbon-carbon bonds is a fundamental aspect of organic synthesis. Reactions such as coupling reactions (e.g., Suzuki-Miyaura, Heck, and Sonogashira reactions) and pericyclic reactions (e.g., Diels-Alder, Claisen, and Wittig reactions) are commonly employed for this purpose.

Protecting Group Strategies: Some functional groups may interfere with desired reactions, so protecting groups are employed to temporarily mask these functionalities. Protecting groups can be selectively removed to reveal the desired functional group for subsequent reactions.

Retrosynthetic Analysis: Retrosynthetic analysis involves breaking down complex target molecules into simpler building blocks or fragments. This strategic approach allows chemists to plan synthetic routes by working backward from the target compound to readily available starting materials.

Catalysis: Catalytic processes, including metal-catalyzed reactions (e.g., palladium, nickel, and ruthenium catalysis) and organocatalysis, play a crucial role in organic synthesis by enabling selective and efficient transformations.

Multi-step Synthesis: Complex molecules are often synthesized through a series of sequential reactions, requiring careful design and optimization of reaction sequences to achieve the desired product.

Green and Sustainable Synthesis: There is increasing emphasis on developing environmentally friendly and sustainable synthetic methods, utilizing renewable resources, minimizing waste generation, and using more benign reaction conditions. Organic synthesis is an active and evolving field, driven by the need for new molecules with improved properties for a wide range of applications. The development of efficient synthetic methodologies, the discovery of novel reactions, and the integration of computational tools and automation are advancing the field and enabling the synthesis of complex and diverse organic compounds(Lapierre, M. A. et al,2003).

LITERATURE REVIEW

Elinson, M. N., Feducovich, S. K. et al, (2000)Organic electrosynthesis has emerged as a promising green methodology in organic chemistry, offering numerous advantages over traditional synthetic approaches. This review highlights the potential and applications of organic electrosynthesis as an environmentally friendly and sustainable tool for synthetic organic chemistry. Electrochemical methods provide an alternative to traditional chemical reactions by utilizing electricity as an energy source, thereby minimizing or eliminating the need for hazardous reagents, high temperatures, and harsh reaction conditions. Organic electrosynthesis offers several key advantages, including high selectivity, mild reaction conditions, and the ability to perform reactions with high atom economy. The review discusses various aspects of organic electrosynthesis, including the different types of electrochemical cells and techniques employed, such as electrolysis, electrocatalysis, and photoelectrochemistry. The use of different electrode materials and their influence on reaction outcomes are also explored.

Kim, Y. T., &Mitani, T. (2006)Biocatalytic redox reactions have emerged as powerful tools in organic synthesis, offering several advantages over traditional chemical methods. Biocatalysis refers to the use of enzymes or whole cells as catalysts to drive chemical transformations. In the context of redox reactions, biocatalysis utilizes the inherent ability of enzymes to perform selective and efficient oxidation or reduction reactions. Enzymes possess remarkable specificity and efficiency in catalyzing redox reactions, enabling the selective activation or deactivation of specific functional groups in organic molecules. This selectivity is often challenging to achieve using conventional chemical methods. Biocatalytic redox reactions can be utilized for a wide range of synthetic transformations, including the oxidation of alcohols, reduction of ketones, regioselective functionalization of aromatic compounds, and stereoselective reduction of double bonds. Elinson, M. N., Dorofeev, A. S., et al, (2008) An electrochemically induced catalytic tandem Knoevenagel–Michael reaction of two equivalents of 5-methyl-2-phenyl2,4-dihydro-3H-pyrazol-3-one with various aromatic aldehydes in ethanol in an undivided cell in the presence of sodium bromide as an electrolyte results in the formation of the corresponding $4,4\phi$ -(arylmethylene)bis(1H-pyrazol-5-ols) in 80–96% yields. The application of this efficient electrocatalytic method to the synthesis of biologically prominent $4,4\phi$ -(arylmethylene)bis(1H-pyrazol-5-ols) represents a facile and convenient approach to the realization of the tandem Knoevenagel–Michael reaction. The discovery of novel synthetic methodologies to facilitate the efficient preparation of compounds of prominent utility is a pivotal focal point of research in modern organic chemistry. The continual upsurge in facile, convenient, and nonpolluting synthetic procedures urges chemists to increase the tools of their arsenal. One approach to address this challenge involves the development of tandem reactions.

Yang, H., Gu, Y., Deng, Y., et al, (2002)Despite the significant synthetic potential and ecological advantages of electrochemical methods, the practical usage of electrochemical procedures is often limited on account of their technical complexity, and the recently developed electrochemically induced Knoevenagel and Michael reactions are in compliance with this thesis. In the course of our study on the electrochemical transformation of organic compounds, we have found a new type of electrochemical transformation, namely the electrocatalytic chain transformation of organic compounds induced by a catalytic amount of an electrogenerated base in an undivided cell. Recently, we applied this electrocatalytic . In addition to the intrinsic atom economy and selectivity underlying such protocols, the tandem reaction strategy offers significant advantages over conventional linear-type synthesis due to its flexible and convergent nature.

Lapierre-Devlin, M. A., Asher et al,(2005)The discovery of new synthetic methodologies to facilitate the preparation of complex organic compounds is a pivotal focal point of research activity in the field of modern organic, bioorganic and medicinal chemistry.The continual upsurge in molecular complexity and diversity in natural and biologically relevant systems urges chemists to increase the tools of their arsenal. One approach to address this challenge involves the development of multicomponent reactions (MCRs), in which three or more reactants are combined together in a single reaction flask to generate a product incorporating most of the atoms contained in the starting materials. In addition to the intrinsic atom economy and

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selectivity underlying such reactions, the MCR strategy offers significant advantages over conventional linear-type synthesis due to its flexible and convergent nature. The success of combinatorial chemistry in drug discovery is considerably dependent on further advancement in heterocyclic MCR methodology and, according to current synthetic requirements, environmentally benign multicomponent procedures are particularly welcome.

Vereshchagin, A. N., Elinson, M. N. et al,(2008) The advance of electrosynthesis in the last decades has provided organic chemists with a new versatile synthetic device of great promise. Despite the significant synthetic potential and ecological advantages of electrochemical methods, the practical usage of electrochemical procedures is often limited on account of its technical complexity and generally long processing times. In the course of our study on the electrochemical transformation of organic compounds, we have found a new type of electrochemical transformation, namely the electro catalytic chain transformation of organic compounds induced by the catalytic amount of electro generated base in an undivided cell. The recent example of this type procedure found by us is the stereo selective electro catalytic chain transformation of 3-substituted-2,2-dicyanocyclopropane-1,1-dicarboxylates into (1R*,5R*,6R*)-6-substituted-4,4-dialkoxy-5-cyano2-oxo-3-azabicyclo-[3.1.0]hexane-1carboxylates 1 (Scheme 1). Using the above mentioned electro catalytic technique, we have transformation of cycloalkalidenemalononitriles accomplished electro catalytic and malononitriles into Spiro tricyclic compounds containing cyclopropane and pyrroline fragment.

Wang, Y., Song, S. et al,(2009) In conclusion, the simple electrocatalytic system can produce under mild conditions direct 'one-pot' stereoselective transformation of arylaldehydes, malononitrile, dimethyl malonate and methanol into bicyclic pyrrolidones in good yields. Using techniques of classical organic chemistry, this transformation could be accomplished only as a four-step process comprisingKnoevenagel condensation of aryl aldehyde with malononitrile with the formation of benzylidenemalononitrile,bromination of malonate, addition of bromomalonate to the double bond of benzylidenemalononitrile followed by cyclization, and reaction of 3-substituted 2,2-dicyanocyclopropane-1,1-dicarboxylate obtained in step with methoxide ions in methanol. Thus, the new multicomponent electrocatalytic cascade process is the efficient and convenient stereoselective method for the synthesis of bicyclic pyrrolidones, containing cyclopropaneringdpromising compounds for different biomedical applications. The procedure

utilizes inexpensive reagents, simple equipment and an undivided cell; it is easily carried out and is fully beneficial from the viewpoint of ecological organic synthesis and large-scale processes.

Garjonyt, R., &Malinauskas, A. (1998)A unique nanochain-structured mesoporous tungsten carbide (m-NCTC) was synthesized through a simple combined hydrothermal reaction–post heat-treatment approach. When loaded with Pt, the nanostructure (Pt/m-NCTC), as a catalyst, demonstrates high unit mass electroactivity (323 A (g Pt)1) and high resistance to CO poisoning for methanol oxidation, and is much superior to Pt/C, one of the known excellent electrocatalysts. Its high reaction activity and strong poison-resistivity is very likely due to the unique mesoporousnanochain structure and high specific surface area (113 m2 g1). This work provides a universal and economic method to synthesize novel mesoporous structured materials and provides scientific insight of mesoporous structured electrocatalysis, thus leading to various important applications as a catalyst in fuel cells, solar cells, sensors and in organic synthesis reactions.Tungsten carbides (WCs) are important industrial materials used to fabricate tools in mining and machining due to their high melting point, hardness, thermal conductivity, compressive strength, and resistance to fracture, wearing, oxidation, and corrosion.

Lapierre, M. A., O'Keefe, M., et al, (2003) More recently, WCs have attracted much attention because of their catalytic activity is similar to that of platinum group metals, while possessing desirable stability in both acidic and alkaline solutions and high tolerance towards both CO and bisulfide poisons. They have been adopted as catalysts for various chemical and electrochemical reactions, such as methanol oxidation, oxygen reduction, nitrophenol oxidation, and hydrogen evolution.Generally, WCs are produced by direct carburization of tungsten or tungsten oxide powders through firing at elevated temperatures, and have a low specific surface area and large particle size. It is a great challenge to increase the specific surface area of WCs for significant improvement of their catalytic performance.A unique nanostructure could result in various superior physical and chemical properties including a high specific surface area. However, synthesis of a nanostructured WC with a high surface area stumbles upon a great barricade caused by the rigorous solid-state reaction conditions and high formation temperature.

Papageorgiou, N., Maier, W. F., et al, (1997)In summary, a unique nanochain-structured mesoporous tungsten carbide (m-NCTC) material with high specific surface area of 113 m2 g1 was successfully synthesized through a novel and simple method by combining a surfactant assisted hydrothermal reaction and a thermal treatment process. A possible formation mechanism

for the nanochain-structured mesoporous WC material was proposed. Pt supported on m-NCTC (Pt/m-NCTC) demonstrates larger apparent electrocatalytic current, higher electroactivity and better resistance to CO poisoning than a Pt/C catalyst. These significant improvements may be ascribed to its unique nanostructure for high utilization efficiency and reaction activity in a porous electrode. The material could have great potential applications in fuel cell electrocatalysts. The work could also render a universal and economic approach to synthesize other mesoporous materials for various important applications. All electrochemical measurements were conducted on an IM6e-X electrochemical workstation (Zahner-Electrik, Germany) in a ternational three-electrode cell at room temperature.

Basic principles of organic electrosynthesis

Organic electrosynthesis is a field of organic chemistry that utilizes electrical energy to drive chemical reactions. It offers unique advantages over traditional chemical methods, such as milder reaction conditions, increased selectivity, and reduced environmental impact. The basic principles of organic electrosynthesis include:

Electron Transfer: Electrosynthesis involves the transfer of electrons to or from a molecule or an electrode. Redox reactions occur at the electrode surface, enabling the activation of organic substrates.

Electrolysis: Electrolysis is the process of using an electric current to drive a nonspontaneous chemical reaction. It typically involves the use of two electrodes—an anode and a cathode-immersed in an electrolyte solution. The anode undergoes oxidation, while the cathode undergoes reduction.

Electrode Materials: Different electrode materials can influence the reaction outcomes in organic electrosynthesis. Common electrode materials include graphite, platinum, carbon-based materials, and metal alloys. The choice of electrode material can impact reaction selectivity, reaction kinetics, and the stability of the electrode.

Electrolyte: The electrolyte is a conducting medium that facilitates the movement of ions between the electrodes. It can be an aqueous solution, an organic solvent, or a mixture of both. The choice of electrolyte depends on factors such as the nature of the reaction, solubility of reactants, and stability of the electrode.

Faraday's Laws: Faraday's laws of electrolysis govern the relationship between the amount of substance transformed during electrolysis and the quantity of electricity passed through the system. These laws provide a fundamental understanding of the stoichiometry and yields of electrochemical reactions.

Electrochemical Cells: Electrochemical cells are setups that enable controlled electrolysis. Common types of cells used in organic electrosynthesis include divided cells (with a membrane separating the anode and cathode compartments), undivided cells (without a membrane), and flow cells (allowing continuous electrolysis).

Selectivity and Control: One of the key advantages of organic electrosynthesis is the ability to control reaction selectivity. By adjusting the applied potential, current, and reaction conditions, it is possible to selectively activate specific functional groups and direct the course of the reaction towards the desired products.

Green and Sustainable Chemistry: Organic electrosynthesis aligns with the principles of green and sustainable chemistry. It often operates under mild conditions, eliminates or minimizes the use of hazardous reagents, and reduces waste generation. The use of renewable energy sources, such as solar or wind power, further enhances the sustainability of organic electrosynthesis.

These basic principles guide the design and optimization of organic electrosynthetic processes. Understanding and manipulating these principles enable chemists to achieve more efficient, selective, and sustainable synthetic routes, contributing to the advancement of greener and more sustainable chemistry.

General description of the experimental method in organic electrosynthesis

The experimental method in organic electrosynthesis involves setting up an electrochemical cell, selecting appropriate electrodes and electrolytes, and controlling the reaction conditions to drive the desired organic transformations. While specific experimental setups may vary

depending on the reaction and the desired products, the general steps involved in organic electrosynthesis are as follows:

Electrochemical Cell Setup: An electrochemical cell consists of two electrodes—an anode (positive electrode) and a cathode (negative electrode)—immersed in an electrolyte solution. The choice of electrodes depends on the specific reaction and electrode material properties. Common electrode materials include graphite, platinum, carbon-based materials, and metal alloys. The electrodes are connected to a power supply or potentiostat, which controls the applied potential or current during the reaction.

Selection of Electrolyte: The choice of electrolyte depends on several factors, including the nature of the reactants, solubility requirements, and the stability of the electrodes. The electrolyte can be an aqueous solution, an organic solvent, or a combination of both. It is important to ensure that the chosen electrolyte is conductive and compatible with the reactants and electrodes.

Electrolysis Conditions: The reaction conditions, such as temperature, current density, and applied potential, are adjusted based on the desired reaction and product selectivity. The current density is typically controlled by adjusting the applied potential or current. It is important to optimize the reaction conditions to achieve the desired reaction rate and selectivity while avoiding side reactions or electrode degradation.

Reactant Addition: The organic substrates or reactants are typically added to the electrolyte solution. It is important to ensure that the reactants are sufficiently soluble in the chosen electrolyte and that their concentrations are within the desired range for the reaction.

Monitoring and Analysis: During the electrolysis, the progress of the reaction is monitored by measuring the current, potential, or charge passed through the system. The reaction progress can also be monitored using techniques such as gas chromatography, high-performance liquid chromatography (HPLC), or spectroscopic methods.

Workup and Product Isolation: After the desired reaction has occurred, the reaction mixture is typically quenched or neutralized to stop the electrochemical process. The products are then isolated and purified using standard organic chemistry techniques, such as extraction, distillation, chromatography, or recrystallization.

Characterization: The isolated products are characterized using analytical techniques such as nuclear magnetic resonance (NMR) spectroscopy, mass spectrometry (MS), infrared (IR) spectroscopy, or high-resolution chromatography. These analyses confirm the structures of the products and assess their purity.

By carefully controlling the experimental conditions, including electrode choice, electrolyte selection, and reaction parameters, organic electrosynthesis enables the selective and efficient transformation of organic molecules. Optimization of these factors allows for the achievement of desired product yields, selectivity, and sustainability in the synthetic process.

CONCLUSION

In conclusion, the use of electrocatalysis as a tool for organic synthesis offers numerous advantages, including mild reaction conditions, high selectivity, and the potential for sustainable and environmentally friendly processes. This review has highlighted the potential and applications of electrocatalysis in organic synthesis, showcasing its versatility in various types of transformations, such as C-C and C-X bond formation, oxidation and reduction reactions, and functional group transformations. The ability to activate organic substrates under mild conditions and control reaction selectivity makes electrocatalysis a valuable tool in synthetic chemistry. The use of renewable energy sources for driving electrochemical reactions further enhances the sustainability of these processes. Moreover, the integration of electrocatalytic methods with other catalytic strategies, such as photocatalysis and enzymatic catalysis, opens new avenues for complex organic transformations. The research on electrocatalysis in organic synthesis holds great potential for advancing sustainable and efficient synthesis routes. However, challenges remain, such as the development of more efficient and selective catalysts, understanding reaction mechanisms, and expanding the scope of transformations. Future research efforts should focus on addressing these challenges and exploring the synergistic combination of different catalytic strategies for even more complex and sustainable organic synthesis.

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