

Advances in Catalysis

催化进展

甲醇羰基化、微结构反应器与
Phillips烯烃聚合催化剂工业应用的进展

BRUCE C. GATES, HELMUT KNÖZINGER

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BRUCE C. GATES
HELMUT KNÖZINGER

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导 读

这一卷《催化进展》主要从3个专题对催化剂研究发展进行了评述,包括甲醇的羰基化反应,微结构催化反应器,以及负载型菲利普铬系催化剂与乙烯聚合的工业应用。

第1章,英国谢菲尔德大学化学系安东尼·海因斯(Anthony Haynes)综述了甲醇羰基化制备乙酸催化剂发展的历史以及工业化具体生产流程。均相催化剂中 Monsanto 工艺是最为成功的例子之一,该催化剂优异了活性与选择性,在过去60年中成为优选的工艺,该工艺中催化体系主要为铑/碘配合物。作者对该类催化体系进行了详细剖析,包括催化反应的机理、碘盐以及膦配体衍生物对催化剂的作用,催化效率改善和提高等。反应机理中阐述了铑配合物的阴离子中间体,探讨了配体对改变催化反应机理的重要性。同时,文中对高聚物负载型的铑配合物催化剂潜在应用和产业化价值进行了必要介绍和探讨。Monsanto 公司在发展铑配合物催化剂体系的同时,发现了铱配位催化剂体系,研究结果显示铱配位催化剂与铑催化剂体系具有相似的催化活性。文中对铱催化剂的早期研究、工业化过程、一价铱与三价铱配合物催化反应机理进行了较为全面的描述(铱是地壳中除放射性元素外丰度最低的金属,这也许是铱催化剂体系没有工业化的真正原因——导读专家注)。由于近年来计算机运算速度的快速提高,产生了许多计算方法并应用于催化剂体系中机理研究,而且计算模拟对结构模型、催化剂中间态与过渡态等能量对比容易,还可以预测振动频率并与实验光谱数据进行比较,可以获得非常多的数据,相关的研究也体现在甲醇羰基化计算化学研究中。

此外,对于甲醇羰基化研究还涌现了其他金属和配体衍生化的催化剂体系,用于提高催化工艺和降低催化成本的研究,这些研究推动了催化工艺的改进和催化性能的提高,降低了生产和产品纯化的成本,提高了工业附加值和效益。针对工业操作过程中一些重要问题,特别是催化剂活性提高、催化过程稳定性与产品选择性等问题关键方法进行了阐述,并对合成乙酸及其衍生物的其他工业途径进行了必要的阐述。

第2章是洛桑理工学院化学和化学工程研究所阿尔伯特·任柯诺(Albert Renken)和索科洛娃·奇异—明斯克(Liubov Kiwi—Minsker)撰写的关于微结构催化反应器的综述。将原材料通过催化工艺转化为产品的生产过程中,催化过程可以使反应物以较高的选择性转化为所需要产品,并最大限度地降低废物生成,如何做到生态保护与高效节能仍然是关键技术问题。为了全面解释催化活性调控与选择性的本质,设计化学反应器并实现微型调控操作成为催化学者努力

达到的愿望。微结构催化反应器在工艺发展和化学反应中的作用日益受到催化学者的重视,通常在低于毫米尺度范围内进行设计,其比表面积是传统反应器两倍还多。文中对微反应器设计与操作基本原理进行阐述的同时,阐述了热质传输的重要价值和解决思路。对于各种不同的微反应器设计,利用相对比较成功的例子,介绍了各自设计的关键技术和特性。目前,微结构反应器设计中如何将催化剂镶嵌在反应器中仍然是瓶颈问题,作者利用大量篇幅阐述了不同的解决方案和实施方法。微反应器的小尺度优势是便于反应条件控制和反应均一性,反应物在反应过程中扩散时间短;然而,质量传递仍会影响反应速度和效率。与传统反应器比,微反应器反应效率高,传热问题更加重要;但是,利用该特点,也许可以促进高放热或高吸热的快速反应,例如,考虑使用积热型微反应器进行高温、高压以及高反应物浓度的反应,以便减少反应器的体积和催化剂的使用量。在这类反应器设计中,需要更多研究,实现等温条件与短时间停留,优化反应器内的接触时间控制,避免副反应发生。微反应器设计不能个别条件上孤立考虑,需要发展合理催化剂设计和反应器设计,两者需要协同考虑和相互促进地发展,实现反应条件的精确调节与控制反应高效和产物的高选择性。配以工程传质和传热问题的解决,才可能使得反应器具有实际意义和使用价值。总之,微反应器研究发展依赖研究深入,推动催化剂设计和选择反应的发展,提高反应效率和产品附加值,并基于此获得微反应器技术成熟和广泛应用。

本书的第3章,由美国雪佛龙-菲利普化学公司菲利普研究中心马克思·P. 麦克丹尼尔(Max P. McDaniel)撰写,他对负载型菲利普催化剂以及工业化生产聚烯烃进行了详尽的综述,也是作者本人在1985年《催化进展》(33卷)上关于菲利普催化剂研究和工业化进展的更新。聚烯烃树脂在合成塑料中占有超过一半的使用量,聚乙烯最早作为军工专用产品,工业化的线性聚乙烯使用了60多年。Phillips石油公司偶然发现负载在硅表面的氧化铬能够进行 α -烯烃聚合,成为铬系菲利普催化剂技术的基础,并于20世纪50年代实现产业化。过去50多年里,铬系菲利普催化剂不断改进和完善,特别用于高密度聚乙烯生产和部分低密度聚乙烯,并且具有不可替代的产业地位。铬系菲利普催化剂近年来研究呈现更加活跃的趋势,应该是产业界起了重要作用:欧美对于铬的使用采用了更加严格的限制,菲利普催化剂不可替代的地位使得学者们需要提高铬系催化剂的效率,与此同时,考虑使用菲利普催化剂对所得聚烯烃进行裁剪,获得高性能聚乙烯树脂。在这一章里,作者详尽地阐述了菲利普催化剂中所有涉及的铬配合物的已知信息,更多依据化学知识与工业开发应用关联进行展开,配以实际范例。文中介绍了烯烃聚合的科学常识,包括Ziegler型和茂金属型催化剂,基于此,介绍菲利普催化剂,并对比了不同催化剂体系。文中重点阐述了菲利普催化剂制备过程和各种细节,讨论了催化剂活性中心与各种测试方法,利用实验证据对比了活化中心对于催化性能的影响,展示了大量图表数据凸显聚合物性能对于

催化剂性质的依赖性。麦克丹尼尔引用了超过 700 篇的研究文章及其本人在工业界数十年的工作经验，比较了过去研究中所得结果，甚至包括相互矛盾的研究结果。特别列出了人们在试图阐明铬的中间体和价态的科学难题过程中，菲利普催化剂研究促进了光谱测试技术的发展，然而，部分研究成果却也造成了研究过程的误导；并对于分离和检测铬活性位点的测试方法进行了总结。虽然综述的重点放到催化剂研究和评价，以及催化性能研究上，落脚点还是聚烯烃催化剂以及用于聚烯烃材料制备和产业化。作者本人多年主持 Phillips 石油公司的菲利普催化剂研究，这部分综述从工业界角度全面和系统讨论菲利普催化剂的进展。对于从事聚烯烃研究和产业化的人员，无论知识补充还是技能提高，本章内容都值得认真阅读。

本卷中 3 个章节各有特点，对于从事均相和多相催化研究的学者和产业界人员，可以在具体问题和具体环节中获得直观知识和技能。对于有志于催化研究和产业化的学生和研究人員，及时研究课题差异，亦可通过阅读本书获得催化研究的常规知识、研究方法和思维方法，找到解决未来催化研究的思路。

孙文华

中国科学院化学研究所

前 言

本卷《催化进展》始于 Haynes 对化学在工业中应用最成功的均相催化过程之一：甲醇生成乙酸的羰基化，进行了评述。作者追溯了化学的发展过程和铑配合物催化过程的历史以及有关铑配位催化剂的最新工作。这一章包括了反应的详细机理，阐明了配体与碘共催化的作用，并且介绍了在生产中一些处理工序问题，包括产品的纯化、催化剂回收以及选择性和稳定性。对于溶液中高聚物负载铑配合物催化剂的使用，作者也进行了评述，认为是具有工业应用价值的研究。甲醇的羰基化被认为是一个典型的均相催化过程，作者阐明了催化机理认识的重要性，对化学改性设计的过程改善与催化剂在载体上固定的优势进行了良好评述。尽管目前化学呈现了深奥的定量解释，但在生产过程中仍面临挑战，例如，如何将反应器腐蚀降低到最小以及催化剂的回收与再利用等问题。

Renken 与 Kiwi-Minsker 报道了微观结构性催化反应器。该逻辑性反应器的设计需要低于毫米尺寸的表征手段，具有比表面容积高的特点。首先针对气—固反应，作者进行了微观反应器设计原理的常规介绍。对随机堆积粒子、有序催化床反应器以及多壁催化活性涂层的反应器，描述了不同构型特点和与热量转移的特征。对于反应过程极快，或者放热或吸热很高的反应，小尺度反应器便于于热质传递，提高效率。该章主要部分描述多相反应器，包括气—液—固和液—液—固反应；微结构反应器设计的关键是如何将催化剂注入反应器中。已经成功使用的微结构反应器包括烷烃的部分氧化反应，多种氢化反应以及甲醇蒸汽重整反应。首个微结构反应器中试实验凸显了该领域研究的现状。

McDaniel 对由负载型菲利普催化剂烯烃聚合的化学与工业研究进行了详细综述。这一章是对他于 1985 年出版的《催化进展》(33 卷)一书中该领域里程碑作品的升级。对比这两章内容可以看到该技术日益增长的重要性，世界上 40% (约合每年 1.2 万吨) 的高密度聚乙烯采用该催化剂生产。该章涵盖了烯烃聚合科学，包括 Ziegler 型和茂金属型催化剂与菲利普催化剂的对比，菲利普催化剂在制备、活化以及性能方面的详细介绍；并采用大量图表数据显示聚合物性质是如何依赖催化剂性质，以及之间变化的影响。本章评述了催化化学的深入发展，包括已知的和未知的，并对加工过程问题进行了总结。该章在技术方面进行了详尽的评述，对该领域工作人员而言是必不可少的指南教程。

B. C. GATES
F. C. JENTOFT
H. KNÖZINGER

(孙文华 译)

CONTRIBUTORS

Anthony Haynes

Department of Chemistry, University of Sheffield, Sheffield,
United Kingdom

Lioubov Kiwi-Minsker

Institute of Chemical Sciences and Engineering, Ecole polytechnique
fédérale de Lausanne, Switzerland

Max P. McDaniel

Chevron-Phillips Chemical Co., Phillips Research Center, Bartlesville,
OK, USA

Albert Renken

Institute of Chemical Sciences and Engineering, Ecole polytechnique
fédérale de Lausanne, Switzerland

PREFACE

This volume of the *Advances* begins with an assessment by Haynes of the chemistry of one of the most successful homogeneous catalytic processes applied in industry: the carbonylation of methanol to give acetic acid. The author traces the development of the chemistry and the history of the processes catalyzed by rhodium complexes as well as more recent work with iridium complex catalysts. The chapter includes detailed mechanisms of the reaction, elucidation of the roles of ligands and of iodide cocatalysts, and statements of engineering issues affecting the processes, such as product purification and recycle and catalyst selectivity and stability. Polymer-supported rhodium complex catalysts analogous to those used in solution are also assessed—these too have found industrial application. Methanol carbonylation can be considered a prototypical homogeneous catalytic process, illustrating the value of mechanistic understanding, process improvements associated with modifications of the chemistry, and the benefits of anchoring the catalyst to a solid support. The understanding of the chemistry is deep and quantitative, but challenges remain concerning process engineering issues, such as corrosion minimization and catalyst recycle and reclamation.

Renken and Kiwi-Minsker report on microstructured catalytic reactors. The intricate reactor designs are characterized by submillimeter dimensions and thus high surface-to-volume ratios. The authors provide a general introduction to the design principles of microstructured reactors and focus first on gas–solid reactions. Reactors containing randomly packed particles or a structured bed of catalyst are distinguished from those with a catalytically active wall coating, and the mass and heat transfer characteristics of these configurations are described. The high heat and mass transfer rates that can be achieved as result of the small dimensions are most beneficial for reactions that proceed rapidly and are highly exo- or endothermic. A section of the chapter is devoted to reactors for multiphase reactions, including gas–liquid–solid and liquid–liquid–solid reactions. One of the major challenges in realizing the designs of microstructured reactors is the incorporation of the catalyst into the reactor. Examples of reactions for which microstructured reactors have been used successfully include the partial oxidation of hydrocarbons, various hydrogenation reactions, and methanol steam reforming.

The state of this field is indicated by the mention of the first pilot plant experiments done with microstructured reactors.

McDaniel provides an exhaustive summary of the chemistry and technology of olefin polymerization catalyzed by supported Phillips-type catalysts. This chapter is an update of McDaniel's landmark contribution on this topic, published in the *Advances* in 1985 (Vol. 33). A comparison of the two chapters demonstrates the growing importance of this technology, which is now used to manufacture 40% of the world's supply of high-density polyethylene, corresponding to 12 million kg per annum. This treatise covers the science of olefin polymerization, including comparisons of Ziegler-type and metallocene catalysts with the Phillips-type catalysts, with details of the preparation of Phillips-type catalysts, activation, and performance, bolstered by extensive tables and figures of data, including many showing how the properties of the polymer depend on the catalyst properties. This chapter provides an in-depth assessment of the catalytic chemistry, including what is understood and what is not, as well as a summary of the major engineering issues. The chapter is remarkably detailed in its evaluation of the technology, being an indispensable guide for anyone working in the field.

B.C. GATES
F.C. JENTOFT
H. KNÖZINGER

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Catalytic Methanol Carbonylation

Anthony Haynes

Abstract

The carbonylation of methanol to acetic acid is established as one of the major industrial applications of homogeneous catalysis. Since the introduction of a rhodium-catalyzed process by Monsanto four decades ago, there has been almost continuous activity aimed toward improving the process and gaining a deeper understanding of the underlying chemistry. Strategies to improve catalyst performance and process economics have been developed, resulting in both rhodium- and iridium-catalyzed systems that operate with high activity at reduced water concentration, making product purification less costly. This chapter describes important aspects of the commercial processes as well as potential strategies for enhancing catalyst activity, stability, and selectivity. A particular emphasis is placed on mechanistic aspects, with experimental studies being complemented in recent years by theoretical investigations. Attempts to anchor the rhodium catalyst and to influence activity and selectivity using phosphine ligands are reviewed. Some potential alternative catalytic routes to acetic acid and derivatives are also summarized.

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Department of Chemistry, University of Sheffield, Sheffield, United Kingdom

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ABBREVIATIONS

Ac	acetyl
Ar	aryl
COSMO	conductor-like screening model
dcpb	1,4- <i>bis</i> (dicyclohexylphosphino)butane
DFT	density functional theory
dppb	1,4- <i>bis</i> (diphenylphosphino)butane
dppe	1,2- <i>bis</i> (diphenylphosphino)ethane
dppm	<i>bis</i> (diphenylphosphino)methane
dppp	1,3- <i>bis</i> (diphenylphosphino)propane
dppx	<i>bis</i> (diphenylphosphino) <i>ortho</i> -xylene
dtbpx	<i>bis</i> (di- <i>t</i> -butylphosphino) <i>ortho</i> -xylene
EXAFS	extended X-ray absorption fine structure
HPiR	high-pressure infrared
HPNMR	high-pressure nuclear magnetic resonance
kt/a	kilotonnes per annum
Me	methyl
NHC	<i>N</i> -heterocyclic carbene
PCM	polarizable continuum model
Ph	phenyl
PHIP	<i>para</i> -hydrogen-induced polarization
PVP	polyvinylpyrrolidone
QAS	quaternary ammonium salt
v/v	volume per volume
w/w	weight per weight
WGS	water gas shift

1. INTRODUCTION

1.1. Historical Context

Acetic acid is an important bulk commodity chemical, with world annual production capacity of ~9 million tonnes. Its principal use (~40%) is in the manufacture of vinyl acetate, a monomer of great importance in the

polymer sector. A range of other acetate esters are also significant derivatives, along with monochloroacetic acid which is an important intermediate in the production of pesticides. Dehydration of acetic acid can be used to make acetic anhydride, which is used as an acetylating agent, for example, in the production of cellulose acetate. Acetic acid also finds a major use as a solvent for the oxidation of xylene to terephthalic acid, which is required on a large scale for incorporation into polyethylene terephthalate.

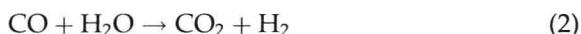
Industrial routes to acetic acid have included oxidation of ethanol derived from fermentation, hydrolysis of acetylene, and the oxidation of hydrocarbons such as butane or naphtha. In the late 1950s, the development of the Wacker process (a $\text{PdCl}_2/\text{CuCl}_2$ -catalyzed oxidation of ethylene) provided a route to acetaldehyde, which could be converted to acetic acid by subsequent oxidation.

The production of acetic acid by carbonylation of methanol (Equation (1)) can also be traced back to the 1950s when Reppe and coworkers at BASF developed a cobalt iodide catalyst that was effective for this reaction at relatively high temperatures and pressures ($\sim 250^\circ\text{C}$, 600 bar) [1,2].



The process based on the cobalt-catalyzed reaction was commercialized by BASF [3,4] but proved not to be so selective as subsequent processes, with an acetic acid yield of 90% (based on methanol feedstock) and 70% (based on CO). The major organic by-products were higher alcohols, aldehydes, and carboxylic acids that required demanding and expensive separation procedures to give acetic acid of sufficient purity.

Much higher catalytic activity and selectivity under milder conditions ($\sim 175^\circ\text{C}$, 30 bar) was revealed by Paulik and Roth at Monsanto, who used an iodide-promoted rhodium complex catalyst [5]. In the same short communication reporting the catalytic reaction, preliminary suggestions concerning the catalytic mechanism were made, and iridium was identified as having comparable activity to rhodium. Monsanto selected the rhodium/iodide catalyst for commercialization, and the first plant based on this technology began production in Texas City in 1970. The so-called Monsanto process became the dominant method for manufacture of acetic acid during the 1970s and 1980s, such that by 1991 it accounted for $\sim 55\%$ of global acetic acid production. It achieved selectivity of $> 99\%$ (based on methanol) but only $\sim 85\%$ based on CO, which is also consumed by the water gas shift (WGS) reaction (Equation (2)):



The process involving the rhodium/iodide combination was operated by other companies under license from Monsanto, including BP Chemicals and Hoechst-Celanese. In 1986, BP Chemicals acquired the licensing rights