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# **CATALYST DESIGN**

## **Progress and Perspectives**

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# PREFACE

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The idea for this book originated at a symposium on catalyst design at the Annual Meeting of the American Institute of Chemical Engineers in New Orleans, November 1981, followed by another one in San Francisco, November 1984. The large interest in the subject motivated the participants of the San Francisco symposium to address the topic of catalyst design by preparing a self-contained book. For this purpose, we met at the Cypress Inn, Carmel, California in June 1985 (see the photograph facing the title page) to discuss how this could best be accomplished. One decision we made was to add a chapter on zeolites, which is now part of this book.

Catalysis is a complex field that has, to date, largely defied attempts toward a systematic approach to the design of new catalysts (at least in the sense that systematic design is practiced in some other disciplines). Nevertheless we felt that a growing body of knowledge exists which can be utilized as the basis for the design of new catalysts, supplemented with the customary enlightened empiricism. We felt the need to provide some firm basis for the hope that someday catalysts could be designed in a more effective manner. However, we were also careful to avoid overstating the current capabilities of the field. Hence the subtitle of the book.

The bulk of the catalytic literature deals with the description and elucidation of catalysts and catalytic reactions. A smaller but rapidly growing literature deals with the preparation of catalysts; several recent symposia were devoted to this subject of widespread interest. In comparison, the literature that is specifically devoted to catalyst design is of a relatively recent vintage, even though the desire for catalyst design has at least implicitly motivated quite a lot of research.

This book addresses various aspects of catalyst design, from different points of view, reflecting the backgrounds and interests of the authors of

the individual chapters. Nevertheless, an attempt was made to integrate these chapters with a common thread: we set out to critically examine our knowledge base for its suitability for the rational design of workable catalysts and to point out gaps in our existing knowledge which we felt are worthwhile filling.

The book follows a bottoms-up structure, beginning with a microscopic view of catalytic surfaces: G. A. Somorjai's chapter deals with the effects of surface morphology and surface composition on catalyst performance, how our current understanding can be used, and how it could be extended to make it more useful for catalyst design.

The chapter by B. C. Gates addresses the opportunities in catalyst design via anchoring organometallic complexes to solid surfaces, together with the problems that need to be solved before such catalysts have the stability required for commercial applications.

Metal catalysts are usually dispersed over large-surface-area supports; a relatively new and quite lively area of research deals with the interactions between these metals and supports. The chapter by A. T. Bell discusses how supported metal systems might lend themselves to a more systematic design effort.

Surface-catalyzed reactions consist of a number of sequential or parallel elementary steps; catalytic selectivity can often be manipulated by selectively influencing these molecular pathways. The chapter by M. Boudart, drawing a parallel with homogeneous reaction systems, addresses the opportunities for designing selective heterogeneous catalysts by interfering with the appropriate elementary steps.

Zeolite-based catalysts are of major industrial interest; their unique selectivity combines the chemical selectivity of their active sites with the shape selectivity of their highly organized but molecular-size pore structures. Our understanding of shape-selective catalysis is now considerable; the chapter by W. O. Haag and N. Y. Chen attempts to organize this field from the standpoint of catalyst design.

Chemical reaction engineering, a relatively young discipline that deals with the mathematical modeling of catalytic systems, has already demonstrated its utility in the design and optimization of industrial catalysts. The chapter by R. Aris discusses how chemical reactivity and transport phenomena (diffusion, flow) can be coupled into quantitative models and how these can be used for the optimal design of porous catalysts. The theory is now useful enough to promise a broader applicability than heretofore achieved.

The book concludes with a chapter by J. Wei on the design of a specific, large-scale catalyst used for the removal of metal impurities from distillation residues in oil refineries. These catalysts operate under extreme conditions of temperature, pressure, and feed impurities. The combination of our understanding in catalytic chemistry and reaction engineering appears useful for the purposeful manipulation of catalyst design variables to achieve longer catalyst life, improved selectivity, and improved activity.

Although the book covers a wide area, including many aspects of chemistry and chemical engineering, it nevertheless omits some important aspects of catalysis. Notable among these is the absence of quantum-chemical considerations of molecular interactions with surfaces. Just as in homogeneous systems, the molecular-level, quantum-chemical interpretation of heterogeneous catalysis offers great promise and is thus of considerable research interest, but despite rapid advances in that area, it has not yet demonstrated its applicability to the design of working catalysts. The ongoing revolution in supercomputers will soon have a large impact here.

There are some additional qualifiers that need to be mentioned. The book is not a comprehensive review of all aspects of catalyst design or catalyst selection. For example, the catalytic literature contains numerous time-tested semiempirical rules and observations for catalyst selection (volcano plots for various reaction systems, *d*-electron character selection rules, etc.). These are not systematically reviewed. Instead, we emphasize more recent developments in selected areas, as enumerated above.

We also would like to acknowledge previous attempts at classifying catalysts and catalytic phenomena from the standpoint of catalyst selection and design. The recent book of Trimm (*Design of Industrial Catalysts*, Elsevier, New York, 1980) comes readily to mind.

Although the book is restricted to the discussion of heterogeneous catalytic systems, in several areas we have employed homogeneous concepts. Nevertheless, the design of homogeneous catalysts per se is not covered. Another omitted area is enzymatic catalysis, which is largely homogeneous in nature.

The book is intended primarily for researchers and practitioners in the field of catalysis. We hope that the researcher will be motivated to push catalytic science closer to the point where the ab initio, rational design of catalysts becomes a reality; we also hope that the industrial practitioner will find ideas in this book to shorten the tedious empirical work that is required today for the development of commercial catalysts. Although we had no intention of writing a textbook, we nevertheless hope that advanced

graduate students will find this volume helpful as supplementary reading to textbooks on applied heterogeneous catalysis.

After having completed this manuscript, we have asked the question once again: "Can heterogeneous catalysts be designed on a rational basis?" The answer remains: not yet, at least not to a satisfactory extent. Progress toward that objective remains one of the prime research goals of the field and we expect continued significant developments in the years to come. It is also clear, however, that the growing body of catalytic knowledge is eminently suitable for the design of at least some aspects of catalytic performance; this belief united us in our efforts to publish this text.

L. L. HEGEDUS

*Columbia, Maryland*

*February 1987*

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# INTRODUCTION

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## 1. ECONOMICS

The economic importance of catalysis is quite significant but hard to quantify in a precise manner because of the diversity of the products and processes that employ catalysts. As an example, let us examine the use of catalysts in conjunction with automotive transportation.

A wide variety of catalytically produced components are being used in automobiles today (the "organic" content of an automobile is about 13% of its weight). These include various polymers for structural components, paints, synthetic fibers, and synthetic elastomers, in addition to various solvents, detergents, and other chemicals employed in the manufacturing process. The polymeric materials are produced from monomers that are the products of a chain of catalytic processes beginning with raw materials such as crude oil and natural gas.

Catalysts are also widely used for refining crude oil into automotive fuels; various refinery streams may pass through a number of large-scale catalytic processes.

Finally, each carbon atom of the gasoline (in the form of uncombusted hydrocarbons or carbon oxides) passes once again through a catalyst that is used for the control of automotive emissions.

The above example for the interwoven nature of catalytic technologies illustrates the complexity associated with quantifying their economic impact. Nevertheless, a variety of breakdowns can be made. It is of interest to contemplate the following facts.

It has been estimated that about 90% of the currently practiced chemical and refinery processes are catalytic in nature. The worldwide usage of catalysts in 1984 cost approximately \$2.7 billion (Strategic Analysis, Inc., 1985). Fifty-three percent was consumed by the United States, 17% by western Europe, and the remaining 30% by the rest of the world. Three main areas of application can be identified: catalysts for the manufacture of chemicals (43% of the total), catalysts for petroleum refining (35%), and catalysts for emission control (22%).

The three largest uses of catalysts in the United States are for automobile emission control (about \$445 million in 1984, including noble metals, or about \$285 million, excluding noble metals), for fluid catalytic cracking (\$255 million), and for a variety of polymerization catalysts (\$235 million). Table 1 lists the U.S. catalyst markets in more detail. The numbers add up to a U.S. catalyst consumption in 1984 worth \$1.33 billion. The U.S. Department of Commerce (1986) estimates that the dollar volume of product shipments in 1984 in the categories "Petroleum Refining" and "Petrochemicals" was \$176 billion and \$83 billion, respectively. We can divide the sum of these items (as they are largely catalytically produced) by the 1984 U.S. catalyst sales to arrive at \$195 worth of products for each dollar of catalyst consumed. This illustrates the tremendous economic leverage of catalytic technologies.

The world of catalyst manufacture is a complex one; it includes (a) companies that primarily sell catalysts to others and (b) companies that are captive users of catalysts they manufacture. In the former category, Stinson (1986) lists W. R. Grace, Engelhard, and Harshaw-Filtrol as the three largest suppliers based on their estimated dollar sales, making appropriate reservations for accuracy. Stinson's article lists the major U.S. catalyst suppliers to the chemical and petroleum refining industries.

Catalysts are typical specialty chemicals sold mostly on the basis of performance and involving a high level of contact between manufacturers and users. Since margins are driven primarily by performance advantage and only secondarily by cost, the catalyst industry tends to be highly research-intensive, often spending over 10% of catalyst sales on research (Stone, 1985). Catalyst developers are diverse in nature; they include major end-users such as oil and chemical companies (which may have both captive and merchant catalyst businesses), process developers, and specialized catalyst companies. The industry is highly proprietary in nature; composition and method of manufacture of complex industrial catalysts are rarely published. Nevertheless, there are references that allow a glimpse into this

**TABLE 1. U.S. Consumption of Catalysts in 1984, Including Captive Consumption**

	Consumption (\$ Million)
<b>Chemical Processing<sup>a</sup></b>	
Polymerization	235
Organic syntheses	85
Oxidation, ammoxidation, and oxychlorination	80
Hydrogen, ammonia, and methanol syntheses	50
Hydrogenation	30
Dehydrogenation	10
Total	490
<b>Petroleum Refining<sup>a,b</sup></b>	
Catalytic cracking	255
Hydrotreating	75
Hydrocracking	40
Catalytic reforming	20
Total	390
<b>Emission Control<sup>c</sup></b>	
Automotive <sup>c,d</sup>	445
Other <sup>d</sup>	5
Total	450
<b>Total Catalyst Consumption</b>	<b>1330</b>

<sup>a</sup>Strategic Analysis, Inc. (1985).<sup>b</sup>Excluding \$155 million for HF and H<sub>2</sub>SO<sub>4</sub> alkylation catalysts.<sup>c</sup>Including an estimated \$160 million for noble metals.<sup>d</sup>Estimated.

aspect of catalysis (e.g., Thomas, 1970; Sittig, 1978; Stiles, 1983). An interesting listing of commercial catalysts for the petroleum refining industry has been published recently (Aalund, 1984).

## 2. TECHNOLOGY

Catalytic technologies continue to advance rapidly, driven by strong economic pressures to improve the selectivity, activity, lifetime, and cost of catalysts. In a typical process, the catalyst is surrounded by expensive

process steps aimed at processing the feed to the catalytic reactor or at processing the effluent from it. Thus, more selective, more active, and more durable catalysts have a large impact not only on feedstock utilization but also on overall plant capital and operating economics. Correspondingly, catalyst research activities are divided between those aimed at new catalyst-process combinations and those aimed at new or improved catalysts for existing processes.

Let us consider an example to illustrate the dynamic nature of commercial catalyst research and development. In the late 1960s and early 1970s it became evident that the emissions of automobiles would have to be considerably reduced in order to meet mounting public demands for strict emission standards. It also became evident that mere modifications to the gasoline-powered internal combustion engine as it existed at that time (or, as a matter of fact, as it exists today) were incapable of meeting these demands. Therefore, catalytic aftertreatment emerged as the only promising technology. As no catalyst existed at that time which could meet the required activity and durability specifications, a new catalyst-process combination had to be developed. A major industry-wide research effort was mounted which, in the case of General Motors alone, involved the testing of over 1500 catalyst formulations, submitted by some 82 prospective catalyst manufacturers, involving over 5000 General Motors employees and 22 million test miles before the catalyst was commercialized in the fall of 1974 (Hegedus and Gumbleton, 1980).

Once commercialized, further development of the catalyst continued: Among other factors, the need for improved fuel economy placed new demands on catalyst performance. Hence, improved catalysts emerged for an existing process. This example is especially interesting because the cycle did not stop there: new, more stringent environmental regulations required the catalytic control of not only hydrocarbons and carbon monoxide but also of nitrogen oxides. A new catalyst-process combination emerged in 1978 to meet this new demand: three-way catalysts (which can simultaneously oxidize hydrocarbons and carbon monoxide while reducing nitrogen oxides) in a catalytic converter controlled by an electronic computer to keep the feed stoichiometry in the desired range (some of the related developments were reviewed by Hegedus and Gumbleton, 1980). The cycle still continues: present-day demands on the catalyst require continued improvements in conversion performance, reactor volume, noble metal usage, durability, cost, and some other parameters. Thus, the research effort goes on at a high level, aimed yet again at improved catalysts for an existing process.

Other examples of significant technological developments in catalysis during the past 20 or so years include (a) zeolite-based fluid cracking catalysts [which, according to Mills (1983) save over \$1 billion per year in imported oil as a result of improved gasoline yields from crude]; (b) a host of imaginative catalytic processes based on ZSM-5, a new zeolite material; (c) a large-scale catalytic technology for the reduction of nitrogen oxide emissions from power plant stack gases currently practiced in Japan and being introduced in West Germany (these are huge monolith-type reactors with up to 1000 m<sup>3</sup> in volume, employing base metal catalysts that selectively react nitrogen oxides with ammonia into nitrogen and water); and (d) a variety of important developments in the partial oxidation of hydrocarbons (e.g., acrylonitrile via ammoxidation, or improved catalysts for the selective oxidation of ethylene). Bimetallic hydrocarbon-reforming catalysts with vastly improved performance have emerged; hydroprocessing catalysts have been developed which can process heavier crudes with higher metals contents; methanol synthesis catalysts have been developed which allow improved process economics; and new polymerization catalysts have emerged which produce high-density and linear low-density polyethylene with substantially reduced capital and operating costs. The list of important recent developments would not be complete without mentioning the substantial catalytic technology base that was generated during the past 10–15 years in the synthetic fuels area. Although many of these processes are not economical at present, they may prove to be important in the future. Among these are several significant new catalysts and processes for the gasification or liquefaction of coal and for the downstream processing of coal-derived gaseous or liquid feedstreams.

### 3. SCIENCE

Catalytic science is in a state of rapid progress (e.g., Somorjai, 1985). Let us review briefly some of the important scientific developments of the past 10–15 years

The evolution of modern instrumental-analytical techniques made a significant impact on the understanding of catalytic behavior. Surface structural information can now be readily gained by low-energy electron diffraction (LEED); surface compositional information by Auger electron spectroscopy (AES); both spatial and compositional information by the scanning Auger microprobe (SAM); and bonding, oxidation state, and related surface-chemical information by x-ray photoelectron spectroscopy

(XPS). Infrared (IR) spectroscopy and Fourier-transform infrared (FTIR) spectroscopy yield qualitative and quantitative information of catalyst-reactant interactions under actual reaction conditions, while modern variants such as photoacoustic infrared spectroscopy (PAS), diffuse reflectance spectroscopy (DRS) and reflectance-absorbance infrared (RAIR) spectroscopy have greatly expanded the utility of this time-tested technique. Vibrational features to which infrared is not sensitive can often be studied by laser Raman spectroscopy (LRS). A somewhat complementary and also relatively new technique for probing molecule-surface interactions is represented by high-resolution electron-energy loss spectroscopy (HREELS). Thermal desorption spectroscopy (TDS), a technique which provides for the time-resolved desorption of surface-bound species, usually into a mass spectrometer, allows the study of the energetics of highly complex, multicomponent catalytic surfaces. Variants of TDS are temperature-programmed desorption (TPD), which employs a carrier gas, and temperature-programmed reduction (TPR), which uses a reducing carrier gas.

In addition to surface-sensitive information, the bulk or near-surface properties of catalysts are also of significant importance. Long-range order can be studied by x-ray diffraction (XRD); shorter-range order can be studied by extended x-ray absorption fine structure (EXAFS) analysis, by transmission electron microscopy (TEM), and by magic angle-spinning nuclear magnetic resonance spectrometry (MASNMR), another new technique that has found important use for the near-order structural analysis of zeolites. The Mössbauer effect has also been successfully employed in catalytic studies to yield information about oxidation states and local coordination numbers.

The rapid penetration of these techniques into industrial catalytic research has completely changed the nature of the way in which new catalysts are being developed today. In addition, modern methods of analysis have proved to be invaluable for ensuring reproducibility and quality control, which are key commercial considerations.

Another area of rapid development in catalytic science is the synthesis and utilization of a number of important zeolites. The technical revolution associated with zeolite-based fluid cracking catalysts has been well documented; subsequent and ongoing work on ZSM-5 and other high silica-alumina-ratio materials has the promise of similar industrial impact. The unique transport properties of zeolites allow researchers to tailor them to achieve high catalytic selectivity (Weisz, 1973). As one of the interesting recent developments, Thomas et al. (1982) discovered ZSM-11 inter-

growths in ZSM-5 zeolite crystals which can only be detected by TEM and which significantly influence the product distribution of methanol conversion to gasoline. While 40 zeolite minerals have been discovered and about 150 synthetic zeolites have been reported to date (Whyte and Dalla Betta, 1982), the majority of commercial catalytic zeolite applications employs only three of these: faujasite (zeolite Y), mordenite, and ZSM-5 (Heinemann, 1981). Thus, the catalytic exploitation of zeolites has a huge untapped potential.

Our understanding of catalytic mechanisms has been considerably enhanced by advances in organometallic homogeneous catalysis (e.g., Maugh, 1983); organometallic analogies of heterogeneous catalysis have also provided much new understanding (e.g., Canning and Madix, 1984). Isotope techniques have continued to represent an important key to the determination of catalytic mechanisms. Metal alloys and bimetallic clusters (Sinfelt, 1979) represent another area of scientific progress with technological potential. In light of these new concepts, the old notions of electronic and geometric factors in catalytic action are undergoing new interpretations (Sachtler, 1983).

Last but not least, important advances are being made in theoretical chemistry with potentially huge impact on catalytic science. Computer-aided materials simulation techniques are evolving (e.g., Goddard, 1985), which may have the ultimate potential for the *ab initio* design of high-selectivity catalytic surfaces. Similarly important future advances in catalytic kinetics are indicated by the increasing availability of supercomputers (e.g., Dagani, 1985) for rate calculations from quantum-mechanical principles.

As a result of this rapidly growing scientific understanding, new catalytic technologies are on the horizon; among these are various C-H bond activation reactions in alkanes, chirally selective catalysts, catalysts for the partial oxidation of methane into more useful chemicals, fuel cells, high-performance emission control catalysts, and a host of new catalytic materials that will be tailored to achieve catalytic selectivity approaching that of biological catalytic systems.

#### 4. CATALYST DESIGN

The development of new or improved catalysts is a tedious, time-consuming process involving a combination of good science, hard work, experience, and considerable serendipity. It would, however, be misleading to claim that catalysts are developed entirely in an empirical way; in fact, progress



in catalytic science is reflected by the decreasing role of empiricism as a function of time. More recently developed catalysts are better understood than their predecessors were at their time of commercialization, and a greater degree of preselection was employed in their development (e.g., Trimm, 1980).

Beyond the obvious driving forces toward a rational basis for catalyst design (i.e., maximum possible catalyst performance and thus improved margins), there are other pragmatic considerations. One of these is the sometimes extraordinary amount of testing required before a workable catalyst is identified and before this catalyst is optimized. As an example, new automobile exhaust catalysts have to pass several stages of laboratory-scale screening, followed by engine dynamometer and vehicle screening experiments by the automobile manufacturers, all involving lengthy durability runs. Once a catalyst has survived these, it enters the certification fleet of the car manufacturer, adding considerable time and expense. The process does not end there; the catalyst manufacturer continues to screen his various manufacturing batches, the car manufacturer faces end-of-the-assembly-line test requirements, and the catalyst, once in the field, is monitored by various agencies for performance in the customers' hands. It is clear that a more rigorous catalyst design procedure, emanating from a deeper understanding of the chemistry, physics, and engineering involved, would be of considerable benefit. Similar examples are provided by various petroleum refining catalysts (e.g., hydroprocessing), where lengthy durability testing requirements tend to limit the number of catalyst configurations that can be considered.

What is needed, then, is a more useful level of detailed understanding of the factors that influence catalyst performance. The list of such items begins at the catalyst surface: We need to have a better understanding of the relationships between the structure and composition of surface and its reactivity. The complexity in practical situations often arises because industrial catalysts may contain quite a large list of modifiers, dopants, selectivity enhancers, structure stabilizers, dispersion stabilizers, and so on, which make the difference between a workable and a nonworkable catalyst. Much of the investigative research to date has been focused on primary catalyst surfaces; chemically modified catalytic surfaces involving several components are still poorly understood. Thus, for a more effective catalyst design, more fundamental studies are needed which involve multicomponent catalytic surfaces.