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applied heterogeneous catalysis

**design · manufacture
use of solid catalysts**

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Institut Français du Pétrole

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Foreword by

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foreword

I could justify the publication of this book, as it is customary to do, by the considerable importance that heterogeneous catalysis and solid catalysts have earned today in both scientific and industrial circles. Although this would be in keeping with reality, it would be only a very imperfect reflection of the goal set by the authors and of the impact of their contribution to scientific, technical and industrial progress in this field. Indeed, it is not an exaggeration to say that such a book is highly original because it fills a void at long last.

Until now, there has been no clear, logical, and abundantly illustrated book giving a methodology for the development of catalysts in the most comprehensive sense of the term, namely that of products capable of performing the many tasks required by industrial practice. Never before, to my knowledge, has a team with such a range of competence in the field of kinetics, heterogeneous catalysis reactions, the design, preparation and production of catalysts and the optimization of their implementation in industrial plants been assembled. And never before has such a team undertaken to compile in a single document the entire scope and wealth of its experience gleaned from long and perseverant work.

To carry out this enormous task, there had to be a combination of favorable circumstances, although this did not occur merely by chance. First of all, there had to be a major objective worthy of rallying and motivating research scientists and engineers with a variety of technical backgrounds. The goal was to give France, and especially its petroleum and petrochemical industry, a technical tool in keeping with its needs and with the position it deserved in the world after having been eclipsed in the Second World War. Pushing catalysts and catalytic processes, given their importance, to the height of their performance was a key to achieving this goal. Willpower and perseverance were needed to overcome the hurdles of this task.

Everything started with a small but enthusiastic group which, in the early 1950s, undertook the scientific and rational development of refining and chemical processes, with the resolution and determination to sustain the venture to the very end, that is until industrial installations had been designed, built and put into operation based on the research that had been done. Whereas this little band was sustained by its faith in the fundamentals and in the mobilization of the most advanced scientific knowledge and know-how, the concrete fact of having to solve the problems inherent in industrial progress was a further and deciding condition of success.

No weakness, no dodging the issue or diversionary tactic can be tolerated when the proper running of large plants with costly investments, an essential element of a country's industry and economy, is at stake. The task was a difficult one, consisting of endowing a single product with a multitude of characteristics of a very different and sometimes contradictory nature, and with optimum effectiveness so as to obtain the highest possible

performances. For not only did a catalyst have to be active, selective, stable under conditions of operating temperature and pressure, and insensitive to various impurities, but it also had to be regenerable after any eventual contamination. It had to be mechanically solid, that is able to resist being crushed under its own weight. It had to resist attrition resulting from operating stresses. And it also had to cause the least possible pressure drop and be as inexpensive as possible.

This objective, which would apparently seem like trying to square the circle, is not only accessible, but the authors of this book guide us through the systematics enabling us to reach it. The different properties required involve a multitude of disciplines and methods such as: (1) selection of the catalyst support, its nature, structure and texture; (2) choice of active agents and precursors to be used so that they can be produced in the desired form; (3) choice of multifunctional promoters and stabilizers, including the nature and stability of state of the active agent, the temperature stabilization of the support, etc.; (4) conditions of precipitation, maturation, impregnation, drying, calcining, etc., corresponding to as many stages governed by well defined parameters which have to be meticulously controlled; (5) conditions and precautions for implementation.

Therefore, it was quite evident that the path to success lay in creating a team working closely together and consisting of specialists in such different disciplines as solid physico-chemistry, mineral chemistry preparation, heterogeneous-catalysis kinetics and chemical engineering. Only such an integrated team would first be able to determine and then develop the correlations linking catalytic properties, solid properties and preparation parameters. As it turned out, these correlations proved to be an effective guide in optimizing a catalyst as it was being developed.

Never will enough emphasis be placed on the decisive role of industrial testing, with its servitudes and contingencies but also the irreplaceable data it provides. How many times the telephone rang to impart the anxiety of the industrial start-up engineer faced with insufficient desulfurization, the shooting up of thermocouple temperatures in a gasoline reforming reactor, or again the appearance of abnormally high amounts of undesirable products in a hydrogenation plant for the purification of olefins. This shows the degree to which the catalyst is inseparable from the process using it and from the procedures for implementing it, from the time it begins operating until it becomes inactive.

Yet the die is cast long in advance. Like the living cell whose genetic code contains the entire program governing its evolution and behavior, the catalyst pellet must be constructed to effectively fulfill all the functions that will be required of it as well as to face up to any possible deviations in normal operating conditions to which it might be exposed. It is thus apparent that effective forecasting requires being aware of everything that might happen.

Likewise, it can be understood why the little group at the start evolved into a solid and thoroughgoing team capable of standing up to stiff competition. For this was the last criterion for success. Faced with a high level of international competition, in order to gain a foothold and maintain it, the scientific and technical value and the quality of the equipment had to be combined with the capacity for the rapid and coordinated implementation of different disciplines.

Today, proof is there for all to see, as illustrated both by the some 600 installations in the world using the processes and catalysts stemming from this effort and by the development achieved by *Procatalyse*, a company created jointly by the *Institut Français du Pétrole* and *Rhône-Poulenc* in 1959 for the purpose of manufacturing and marketing the catalysts developed by the laboratories of both organizations.

J. LIMIDO

Director of Refining and Petrochemicals, 1964-1979

Institut Français du Pétrole

Currently Director of École Nationale

Supérieure du Pétrole et des Moteurs

preface

This book aims to give the reader the benefits of experience at the *Institut Français du Pétrole (IFP)* in the development of solid catalysts and the refining and petrochemical processes that use those catalysts. Without attempting to offer the last word on fundamentals or to expose the latest theories being recommended in the various fields of catalytic application, the text will concentrate on the joint development of solid catalysts and their associated processes.

Nevertheless, to take into account the developments in the field that have appeared since 1978, some modifications and additions have been made in the English translation, in Chapter 7 dealing with solid characterization and in Example 6. Some of our recent results have been introduced.

Given the importance of solid catalysts and catalytic processes during the past thirty years, it is surprising that so few books have been devoted to the industrialization of catalysts. This neglect is perhaps explained by observing that the catalyst imbues a process with its characteristics and originality, and thus distinguishes the process. Consequently the researchers and licensors concerned with industrial catalysts are reluctant to disclose explicit information about unique characteristics of their catalysts or the particular problems that led to the development of those catalysts.

However, the *Institut Français du Pétrole* also assumes the function of training engineers for industry in parallel with its mandate for process development, so that it is more in keeping for us to discuss the general problems of the conception, preparation, and application of catalysts, so long as we do not infringe on the secrecy agreements under which our processes are licensed.

Accordingly, this book may be considered an *exposé* of IFP's recommended methods for carrying commercial catalysis from research to application in industrial plants. The fields of study spanned by such an *exposé* are necessarily vast, including on the one hand a variety of industries, and on the other hand many different scientific and technical pursuits. The industries include: petroleum refining, petrochemicals, fine chemicals, chemical fertilizers, conversion of coal, pollution control, and so forth; while the scientific and technical pursuits include: inorganic chemistry, organic chemistry, physical chemistry of solids, chemical kinetics, chemical engineering, colloid chemistry, and so forth.

With this kind of complexity, it has been necessary for us to try to classify the problems and to reason out methods for solving the classes of problems. At the same time, it has been necessary to bring to bear basic knowledge from various disciplines in order to exploit the most current theories by choosing from among them those salient points that meet the immediate needs, even when the theory is imperfect and temporary. These, then, are the guidelines we have tried to follow in our approach to industrial solid catalysis at a time when the body of this technology is becoming more and more complex.

Our book is composed of two parts, one concerning the catalyst and the other the

catalyst's application. The first part, after short descriptions of catalytic action and the related kinetic laws, analyzes the sequence of operations that proceed out of one another from the conception of a catalyst to its use in an industrial plant, that is: selecting active agents, formulating a catalyst at various stages of its development, determining the catalytic, mechanical and physico-chemical properties of the various formulations, selecting the optimum catalyst, and putting it to work under applicable conditions of operation in a suitable reactor and flow scheme. The various methods of determining and studying the mechanical and physico-chemical properties of the catalysts are briefly described, and when several methods are available, we have tried to indicate the advantages and disadvantages of each.

We want to emphasize the importance we accord to the correlations that always exist among: (1) the catalytic properties, (2) the physico-chemical characteristics of the solids, and (3) the conditions that govern the various stages of catalyst preparation. When based on the fundamentals of catalytic action, those correlations can be used practically to speed the optimization of the industrial catalyst and the conditions for its use.

The second part of this book is devoted to six concrete examples, five of processes developed by *IFP* and the sixth of a process jointly developed by *IFP* and *Charbonnages de France*, using catalysts that are the result of research carried out jointly in the laboratories of *IFP* and *Rhône-Poulenc* through their subsidiary, *Procatalyse*. These examples have been chosen both because they illustrate the methods described in the first part and because the documentation for thoroughly analyzing this methodology is available.

With all this mind, we expect that this book will be of interest to:

- (a) University researchers, who might use it to better judge the conditions to be met and the problems to be solved as an idea proceeds to its industrial development.
- (b) Industrial researchers, who might not have their own catalyst preparation facilities and are faced with the task of selecting from among commercially available catalysts those that best answer a specific problem.
- (c) Operating engineers and managers, who might find here suggestions for answers to the problems they face in maintaining the smooth functioning of industrial units.
- (d) Teachers, who can find here practical examples to use in training young engineers and researchers.
- (e) Students, who may look forward to working in one of the disciplines represented here.

Finally, it should be understood that this book is a collective work, with contributions from the majority of the engineers in *IFP's* heterogeneous catalysis group. The undersigned authors are only the interpreters for a much larger group of people, who have been working on the development of catalysts and catalytic processes. We also want to thank all those who have helped, but whose contributions may not be so obvious, and we want them to know that our results are available to them for any work or publication that they might wish to undertake in their own respective fields; we want to thank:

- (a) Those colleagues who have left the catalyst group to work elsewhere, e.g. Messrs. M. DERRIEN, P. DUHAUT, and P. NAFFA.

- (b) The engineers and technicians of the physico-chemistry group (particularly Mrs. M. T. CHENEBAUX) for their participation in determining the basic characteristics of our catalytic solids.
- (c) All the technicians, operators and secretaries of the heterogeneous catalysis group, who have worked anonymously but effectively for the renown of *Institut Français du Pétrole* for more than twenty years, and who have contributed to the successful operation of the several hundred units now operating under *IFP* license.
- (d) The students and junior engineers, both foreign and French, who have helped with research studies that permit us to better illustrate the teachings of this book, and who have helped resolve a number of applied and exploratory problems encountered in the course of our research work.
- (e) Our colleagues at *Rhône-Poulenc*, who have played an important role in working out the various catalysts commercialized by *Procatalyse*, particularly those discussed in this book, both in the laboratory and in the development of manufacturing technology.

In addition, we express our gratitude to Mr. René NAVARRE, now deceased, who while he was President Director General of *Institut Français du Pétrole* encouraged us to write this book.

We also want to express our sincere gratitude to Professor JUNGERS, former Chairman of the Scientific Committee of *Institut Français du Pétrole*, for the advice and encouragement he gave us.

Finally, our deepest thanks go to Mr. J. LIMIDO, Director of Refining and Petrochemicals from 1964 to 1979 at *Institut Français du Pétrole*, who created the heterogeneous catalyst group at *IFP* and started the development of its processes and who remains one of the promulgators of the ideas of this book. Mr. LIMIDO, who is currently Director of *École Nationale Supérieure du Pétrole et des Moteurs*, has kindly agreed to write the Foreword to this work, for which we express our thanks.

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