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UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION
Vienna International Centre, P.O. Box 300, 1400 Vienna, Austria
Tel: (+43-1) 26026-0 · www.unido.org · unido@unido.org

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DEVELOPMENT OF NOVEL SHAPE SELECTIVE ZEOLITE CATALYSTS

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INDIA

Final Report*

Prepared for the Government of the Republic of India
by the United Nations Industrial Development Organization,
acting as executing agency for the United Nations Development Programme

Based on the work of Mr. Sargis Khoobiar
Expert in Catalytic Reactions, Chemistry and Engineering

Backstopping officer: M. Derrough, Industrial Operations Technology Division

United Nations Industrial Development Organization
Vienna

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I visited NCL in March 1989 and I was happy to see that NCL had made significant advances since my last visit in November 1987. The purpose of my visit was to study NCL's short term and long term research which is funded by UNDP. Specifically, an attempt was made to study zeolite catalyst development and method of its testing in small and large scale reactors. Small scale reactors are used for rapid catalyst testing at the exploratory level. Large scale reactors are used for engineering research, for the development of flow sheets and processes scale up, which are essential objectives in the commercialization of novel processes and comparing them with competing technology. This review is also extended to NCL past developed processes and the possibility of commercialization outside India.

NCL's research effort in petroleum and petrochemicals is approximately equal to a medium-size company in U.S.A., Europe and Japan. For the purpose of evaluation, the essential element of a well-run research organization will be described and NCL will be compared with such an organization.

For the development of a successful catalytic commercial process by any research organization, there are two essential prerequisites: 1. Having an active catalysis group equipped with critical equipment and 2. having a qualified and well-equipped infrastructure that brings novel discovery to commercial reality.

Development of a Successful Commercial Process

In order for a novel discovery to become a commercial reality, a total commitment is required between chemical manufacturing, research and development. Information on the discovery, raw materials and marketing must be shared by all interested groups. A successful research organization will have three essential sections active in research and development:

1. Exploratory (catalysis)
2. Engineering Development (infrastructure)
3. Process Development (infrastructure)

Exploratory Section

This is the core of research, where ideas are conceived, initial trials are made, and data for the preliminary flow sheets is obtained. Detailed experimentation is performed to define the process conditions.

Engineering Development Section

Engineering Development involves two groups. One deals with economic development, competitive economics, and marketing. The second group deals with flow sheet development, plant automation, systems analysis, hazard analysis, as well as other plant requirements. It is the function of this section to confirm a commercial discovery, define the flow sheet, and critical points for a successful process.

Process Development

This section gets its initial data for a discovery with commercial potential from the exploratory section, flow sheet, and critical process variables from the engineering section. They try, experimentally, to scale up exploratory work to commercial designs as specified by the engineering development section. This section performs details and accurate experimentation to bring an exploratory discovery to commercial reality. An oversight or error by process development could have catastrophic results. Research cost in this section is 5 to 15 times that of exploratory group for any project. Therefore, the research in this section is limited to discovery with commercial potential and not to any novel idea.

Ultimately, the engineering development section uses data obtained by the process development section for final process economic and process flow sheet.

The Engineering Development section performs a careful evaluation of the process and its competitive economics, recommends a process to manufacturing with a detailed process flow sheet, description, critical process variables, limitations, and future expansion.

The engineering section of the manufacturing company studies the recommended process and compares it with a competing process offered by others for licensing. The engineering group of the manufacturing section recommends the best process in their evaluation to their management, regardless of whether the process was discovered internally or externally. The manufacturing company builds the best available process.

To achieve technical and commercial success through research and development, total coordination, flow of information and, mutual assistance between these various groups is essential.

NCL has an excellent heterogenous catalysis section. They have had remarkable success in zeolite catalysts as reported by CTA as follows:

<u>Accomplishments</u>

1. A series of faujasites, containing various amounts of Fe in the framework has been synthesised. The presence of Fe in the framework has been demonstrated by more than one technique. Some extra framework Fe is also present. Catalytic activities have been determined.

2. Two new zeolite structures (UN-1 and UN-2) have been made at NCL. Preliminary characterizations show that both materials are so-called "small pore" zeolites; that is, the pores are surrounded by 8 overlapping MO4 tetrahedra (where M = Si or Al).

3. NCL's accomplishments in addition to items 1 and 2 are:

   a. Iron has been incorporated into the framework of ZSM-20. The new zeolite has been characterized. The crystals are 100 percent pure.

   b. Iron has been incorporated into the framework of zeolite beta. Its properties have been characterized. The crystals are 100 percent pure.

   c. NCL has systhesized a high-silica mordenite. High Silica mordenite has great commercial potential. This is the first successful direct systhensis of this material. Its properties have been characterized. The crystals are 100 percent pure.

d. NCL has synthesized a high silica EU-1 zeolite. Again this is the first successful direct synthesis of this material. Its properties has been characterized. The crystals are about 100 percent pure.

e. NCL has found a better and less expensive process to synthesize zeolite beta.

f. The iron-containing beta zeolite may be a better xylene isomerization catalyst than those used commercially today. It combines the advantages of Mobil's HZSM-5 (high ethylbenzene conversion of high LHSV) and Engelhard and UOP's mordenite hydro-isomerization of ethylbenzene to xylenes).

As reported by CTA, the heterogenous catalysis section is strong and innovative. They have some critical equipment needed to identify a novel structure and explore commercial potentials.

NCL's catalyst testing equipment at present is semi-automatic. This has slowed down and restricted their productivity. Complete automation of catalyst testing equipment and supporting analytical equipment will improve NCL's productivity dramatically. It is essential to obtain more funding from the Indian Government and UNDP to eliminate this shortcoming.

NCL's Infrastructure

Comparing NCL's infrastructure with a worldwide competing company, NCL is relatively weak, although it has some outstanding chemical engineers. The weakness is due to factors, as outlined below.

NCL does not have prototype reactors, either adiabatics or isothermal. Prototype reactors, with and without multiple sampling points, are essential for determining commercial reactors, other essential equipment, and realistic economic information. This information is essential in determining the commercial feasibility of the process.

Adiabatic Reactors

An adiabatic reactor with or without multiple sampling points is essential for zeolite catlysis. A prototype, adiabatic reactor with 50, mm., I.D. and 5-10 m. height, is basically a small fraction of a commercial reactor with 10-30 m., I.D., and 5-10 m. height. A prototype adiabatic reactor has to reproduce temperature profile and the product composition of commercial reactors, although its size is 10^{-4}-10^{-6} of commercial reactors.

The heat transfer through reactor walls should be insignificant compared to the heat of reaction as is the case in a commercial reactor. The temperature difference between the inside and the outside of the reactor, through its entire length, should be negligible. This will allow NCL to obtain design information for commercial reactors. Often it becomes essential to confirm some critical points of adiabatic reactors with isothermal reactors. To build an adiabatic reactor with negligible heat transfer through the reactor wall needs temperature monitoring and control inside the reactor and outside of the secondary insulation for the entire length of the reactor. That is why a perfect prototype adiabatic research reactor is the most expensive research reactor to build.

Isothermal Reactor

An isothermal prototype reactor is basically one tube from 10,000-50,000 commercial size tubes. They can operate from room temperature up to 500° C. The reactor is immersed in heat transfer liquid, to obtain isothermality. The basic requirement for an isothermal reactor is opposite of that of the adiabatic reactor. The heat transfers through the reactor wall should exceed, or at least be equal to the heat of reaction. Temperature differences between the inside reactor and the surrounding liquid should be low enough to avoid the run away reaction. This is essential for a highly exothermic oxidation reaction.

NCL does not currently have any isothermal reactor other than ethylene oxide, which is limited to that project. To build a prototype isothermal reactor with multiple sampling points for high temperature operation up to 500° C. requires special design considerations.

Automated adiabatic and isothermal prototype reactors with supporting equipment are essential in the commercialization of a novel discovery. A recycle system may also be required should the process be based on a recycle system.

Industrial, Experience, Process Flow Sheet

The other area of weakness is the absence of extensive commercial manufacturing facilities to use those processes as a guide in the development of flow sheets and economic evaluation. NCL recognizes this problem. Its effect is reduced by cooperation with the manufacturing company, the engineering company, and the private consultant. This is not as critical as having the prototype units.

Conclusion

NCL's research accomplishments in zeolite catalysis has been excellent. I believe they could continue on this path and compete successfully worldwide in zeolite related technology. However, to bring these discoveries to commercial reality, it is imperative to have prototype units of adiabatic and isothermal reactors. The mini prototype units should be automated in order to increase accuracy and efficiency. It would be preferable and more cost effective if the automation included various small reactors of the exploratory group. The Indian Government, UNDP and other sources should finance this equipment as it will help in the industrialization of India and contribute to the advancement of mankind.