

BIENNIAL REPORT

1993-95



NATIONAL CHEMICAL LABORATORY

PUNE, INDIA

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Cover photos

(From upper left clockwise)

1. The new catalyst manufacturing pilot plant facility.
2. *Beck Bond*, a polyurethane-based water-proofing compound, commercialized by Dr. Beck & Co., Pune.
3. The jewel-like design is a computer-simulated structure of a zeolite catalyst, used in industrially important reactions.

NATIONAL CHEMICAL LABORATORY

DR. HOMI BHABHA ROAD

PUNE 411008

INDIA

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PREFACE

There has been a change in our research policy in the recent years; the new policy reflects the changes in our national economic and industrial policy that were introduced some time ago. This policy has been evolving over the past few years and is now taking a tangible shape. In this new policy, there is an increasing emphasis on accountability and self-reliance. Being able to earn on's own funds is no longer an activity to be frowned upon, but a respectable pursuit. In the present climate, scientists would be expected to make their activities profitable in two ways : they should add to the pool of knowledge - the additions should be to the core of the pool and not to the periphery; and they should aim at getting returns in terms of money, for the simple reason that pursuit of research is a costly one, one that needs expensive equipment and instrumentation.

The present report reflects these changes to some extent. We have made original additions to the pool of knowledge and we have also earned money in the process through sponsorship projects and consultancies. We have published close to 600 papers during the two-year period covered by this report, with the area of Polymer Chemistry and Engineering topping the list with 125 papers. It is an impressive figure and shows that we have been able to keep our output in basic research at a high level without sacrificing the quality. Our inventive vigour is reflected in the patents granted to us. The reports tells us that we were granted 17 Indian patents and 15 foreign ones, of which 12 were US patents. This, again, has been a sparkling performance, especially the bagging of 12 US patents, which are the hardest to get in the world. This also points to another heartening development, namely, the increase in awareness of the importance of patenting inventions. This awareness is high among our colleagues, and there is

every hope that our successes in patenting will show a steep upward trend in future. This success leads to yet another fact that should bring us considerable cheer: the awards system initiated by the NCL Research Foundation has proved to be a powerful driving force. It has added an extra dimension to our basic research and patenting activities.

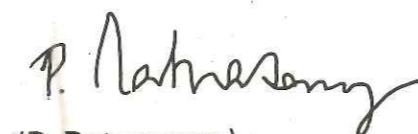
The report of the research during the period, which occupies the first 50 pages or so, discloses that our research endeavour is knowledge-oriented and also product/process-oriented. At least one item in it (oxygen-enriching membranes) shows that societal needs are also among our priorities.

It is now common knowledge that the budget allocation in future will be based not on the number of research proposals that a division submits, but on research performance and research quality. We have to gear ourselves to these new demands on our ingenuity. I have no doubt at all that our reports for the coming years will mirror the success that I am sure we will enjoy in reorienting our priorities to match these demands.

The preparation and release of this biennial report has been delayed by several months, owing to circumstances beyond control. Perhaps, this is the last report to appear in the present format. The format in which the next and the subsequent reports will be prepared will be in keeping with the shift in our research policy with its new priorities.

With this brief introduction, I take pleasure in releasing the report for 1993-95.

April 1996


(P. Ratnasamy)

CATALYSIS AND CATALYTIC PROCESSES

CATALYSIS AND CATALYTIC PROCESSES

◆ Linear Alkyl Benzene (LAB)

A new NCL process for the manufacture of LAB by alkylation of benzene with mixed olefins, using a zeolite catalyst, is undergoing pilot plant trials at Reliance Industries Ltd, Patalganga. The catalyst loaded in the pilot plant has been manufactured indigenously by the process based on NCL know-how. The catalyst has been designed to replace the corrosive and environmentally unsafe HF that is currently being used in the alkylation process.

◆ Auto Exhaust Catalytic Converter for Two/Four-Stroke Engine Vehicles

The second phase of work on the development of a catalytic converter for two/four-stroke engine vehicles, sponsored by DST, New Delhi, has been taken up as an inter-institutional project. Both noble metal and non-noble metal-based catalysts are to be deposited on monoliths of 400 cpsi cell density, developed and made at BHEL, Bangalore. The process for coating non-noble metal oxides on the support has been optimized. Some of the coated formulations are undergoing trials at the Automobile Research Association of India, Pune, in their engine dynamometer set-up, with both leaded and unleaded gasolines. The entire development, together with field trials, is expected to be completed by the end of 1996.

◆ Selective Oxidation of Benzene to Maleic Anhydride

A promoted V-Mo catalyst, developed for the selective oxidation of benzene to maleic anhydride, has been successfully tested. The aim is to replace the existing imported catalyst by an improved indigenous catalyst.

◆ Dehydrogenation of Cyclohexanol to Cyclohexanone

This project, sponsored by Gujarat State Fertilizer Corporation (GSFC), is for the development of suitable catalyst formulations for low temperature as well as high temperature operations at GSFC. A Cu-based catalyst, with the desired activity and selectivity in the temperature range 230-300°C for the dehydrogenation, has been successfully scaled up to bench scale and tested for a long duration to establish its stability; it is now available to GSFC for commercial trials. Another high temperature catalyst is undergoing bench-scale trials in the laboratory.

◆ Augmentation of Aromatics Production

In the manufacture of benzene, toluene and xylenes (BTX) by reforming of naphtha, the reformat is extracted to separate the BTX from the raffinate, which is composed of branched alkanes which cannot be reformed further. The reformat has a low octane number and is a relatively low value by-product. In a project funded by BPCL, Bombay, NCL has developed a zeolite-based bench-scale process to convert raffinates into aromatics and LPG. This process significantly improves the overall economics of aromatics production. The yield of aromatics and LPG, based on raffinate, is about 40% each. The catalyst has a long life.

◆ Oxidation of Hydrocarbons Over Vanadium Molecular Sieves

A project, funded by IFCPAR, New Delhi, and aimed at the synthesis of novel vanadium molecular sieves, identification of the nature and location of vanadium in them and their catalytic applications in selective oxidation reactions, has been initiated in collaboration with University of Paris, France. Two novel vanadosilicates (VAPO-31 and V-Al-β) have been synthesized. Physicochemical studies have revealed that the vanadium in the vanadosilicate lattice is incorporated mostly as V⁵⁺.

◆ Coupling of Exothermic and Endothermic Reaction in the Conversion of Methane and Ethane

The conventional conversions of methane to syngas (CO + H₂) by steam and/or CO₂ reforming, and of ethane to ethylene by thermal cracking are highly endothermic and hence highly energy intensive. Recent processes based on catalytic oxidative conversion require little or no external energy, but are very difficult to operate because of the difficulties associated with the removal of heat from the reactor (a small decrease in selectivity can cause a large increase in the heat produced); this also makes them highly hazardous. Because of the concern about the present energy crisis and environmental problems, it is necessary to develop processes for valorization of methane/natural gas that are highly energy efficient, are safe to operate and require little or no external energy. To achieve this goal, the exothermic and the endothermic reactions have been successfully coupled by carrying them out simultaneously over the same catalyst.

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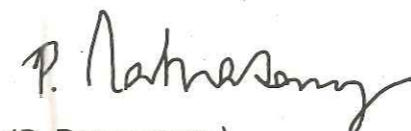
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◆ **Coupling of Oxidative Methane-to-Syngas Conversion and Steam and/or CO₂ Reforming of Methane**

A highly energy-efficient process based on a Ni-based catalyst on a commercial support and a feed comprising methane, O₂, H₂O and CO₂ has been developed. The formation of inactive Ni-silicate/aluminate phases is prevented in the novel catalyst preparation method.

The conversion is achieved by the coupling of endothermic steam and CO₂ reforming with the exothermic oxidative conversion reactions over the same catalyst. High conversion and selectivity (>95%) are the prominent features of this process. The productivity for CO and H₂ can be varied by varying the H₂/CO ratio from 1.5 to 2.5.

By manipulating the process conditions, it is also possible to make the process almost thermoneutral, mildly exothermic or mildly endothermic. Because of a very small adiabatic temperature rise it is possible to use a simple adiabatic reactor for the process.

◆ **Coupling of Oxidative Dehydrogenation and Thermal Cracking of Ethane to Ethylene**

In the NCL process, thermal cracking of ethane is carried out simultaneously with the oxidative dehydrogenation of ethane over a catalyst. By the coupling of the endothermic and exothermic ethane conversion reactions, it is possible to convert ethane to ethylene with more than 70% conversion and more than 80% selectivity in an energy-efficient and safe manner, with very little expenditure of external energy. In this case also, the process can be made nearly thermoneutral, mildly exothermic or mildly endothermic; and the problems of heat removal from the reactor are eliminated.

◆ **Phenol by Cleavage of Cumene Hydroperoxide**

In the process for the manufacture of phenol by the cumene route, cumene hydroperoxide is decomposed to phenol and acetone in the presence of sulphuric acid. Subsequent neutralization and distillation lead to the desired product. Exploratory studies have been carried out using solid catalysts with encouraging results.

◆ **Transalkylation of Cumene Heavy Ends**

The manufacture of phenol by the cumene route generates heavy ends containing about 80% diisopropyl benzene (DIPB) along with low boilers and heavy residues.

Currently DIPB is being disposed off as fuel. A catalyst developed on a laboratory scale at NCL is capable of converting DIPB to cumene by reaction with benzene. The process development work at laboratory scale has been completed and its scale up to 10 tonnes per day has been conceptualized. An economic analysis shows that the process is commercially viable. Hindustan Organic Chemicals Ltd., Cochin, the sponsors, have planned to install a 10-TPD pilot plant for carrying out trials.

◆ **Maleic Anhydride to Tetrahydrofuran / Butanediol / Butyrolactone**

Tetrahydrofuran (THF), butanediol (BDO) and gamma butyrolactone (GBL) are important industrial chemicals with wide ranging applications in plasticizers, detergents and solvents. A catalytic process for the conversion of maleic anhydride to THF, BDO and GBL has been developed; the unique features of this process are the high selectivity of the catalyst for THF and the flexibility that enables the production of GBL or BDO by tailoring reaction conditions and the catalyst. A pilot plant is being setup in NCL for evaluation of the catalyst and collecting design and scale-up data for this process, which is superior to the state-of-the-art Davy McKee process and is far more versatile.

◆ **Biphasic Catalysis for Hydroformylation**

Homogeneous catalysis by water soluble complexes in a biphasic system has many important applications in industry, as it simplifies the separation of catalysts and products. NCL has developed a new catalyst system for hydroformylation of olefins using a water soluble rhodium catalyst which offers ten to fifteen times higher activity, compared to the state-of-the-art biphasic catalysts. This catalyst can be directly used in hydroformylation processes for alcohols in the C₆-C₂₀ range, which have important applications in plasticizers, perfumes and detergents.

The new catalyst system developed at NCL is based on a novel concept of interfacial catalysis using catalyst-binding ligands. This major discovery has been published in the 9 February 1995 issue of *Nature* (p.501). This approach leads to a several-fold rate enhancement in homogeneous catalysis by water-soluble complexes.

This work forms a part of the collaborative project between NCL, Pune, and ENSIGC, Toulouse, France, supported by the Indo-French Centre for Promotion of Advanced Research (IFCPAR).

◆ **Process for Ibuprofen via Carbonylation of 1-(4-isobutylphenyl) Ethanol (IBPE)**

Ibuprofen is an antiinflammatory and analgesic drug belonging to the family of aryl propionic acids. It is currently manufactured by a process involving at least six steps. Such a multi-step synthesis produces large amounts of undesirable by-products that are harmful to the environment. NCL has developed an ecofriendly laboratory-scale catalytic process for ibuprofen involving carbonylation of IBPE. The process consists of three main steps; acylation, catalytic hydrogenation and catalytic carbonylation. The first two steps have been standardized on a 10 g/batch scale. Work on the last step is in progress. The catalytic system developed at NCL gives over 90% conversion of IBPE and over 95% selectivity for ibuprofen.

◆ **Carbonylation Reactions Using Gas-Liquid-Liquid-Solid Catalysis Systems**

A new approach involving a four phase (gas-liquid-liquid-solid) catalytic system for enhancing rates of carbonylation reactions has been developed. It has been found that due to the adhesion properties of the solid catalytic particles at the liquid-liquid interface, the rate of carbonylation reactions is enhanced. This is also a unique example of a catalytic cycle comprising dissolution of the metal and its precipitation. The system not only improves the rates substantially but is also convenient for practical applications.

◆ **Phase-Transfer Catalyzed Carbonylation of Benzyl Chloride to Phenylacetic Acid**

Carbonylation of organic halides using a phase-transfer catalyst produces the corresponding carboxylic acid derivatives in high yields under mild operating conditions. This single-step route avoids the use of toxic NaCN. A bench-scale process has been developed for phenyl acetic acid on a 10g-per-batch scale. It uses a catalyst system based on cobalt carbonyl complex and phosphorus-containing ligands. The selectivity for phenyl acetic acid is between 70 and 80%, and the conversion of benzyl chloride is more than 95%, which make the process economical.

POLYMER SCIENCE AND TECHNOLOGY

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◆ Water-borne Polyurethanes

Solvent-based polyurethanes have well-known applications as coatings and adhesives. In recent years, aqueous polyurethanes have enjoyed a distinction as materials of choice because of the requirements of environmental legislation. Moreover, a wide range of properties characteristic of organic solvent-based systems are achievable with the use of aqueous systems. Water-dispersible cationic and anionic polyurethane ionomers have been synthesized from polyether polyols and diisocyanates using suitable emulsifying agents.

Anionic polyurethane ionomers have been employed as surface coatings. The polyurethane film dries in air in less than four hours at room temperature. The film has good adhesion to substrates like glass, mild steel, aluminum, wood and plastics. It has good flexibility, scratch hardness and resistance to water and mild detergent solutions. Development of formulations and their evaluation for various applications are underway.

◆ High Performance Polymers : Polycarbonates and Polyesters

Poly(arylcarbonate)s are commercially manufactured using environmentally hazardous chemicals like phosgene and chlorinated solvents. NCL has succeeded in making injection-moulding grade polycarbonate by a non-phosgene route based on the carbonate interchange reaction. This ecofriendly process is of strategic importance in the global context.

Traditional synthesis of aromatic polyesters has been based on either phenyl ester of tere/isophthalic acids and bisphenol A (BPA) or BPA-diacetate with tere/isophthalic acids. Both the processes are expensive and recycling of the by-product is difficult. A new melt polycondensation process for the transesterification of DMT/DMI with BPA has been developed. A polyester with intrinsic viscosities greater than 0.4 has been synthesized by this method.

◆ Viscosity Index Improvers

A novel approach using metallocene catalysts has been adopted for the synthesis of ethylene-propylene copolymers for use as viscosity index improvers, in a collaborative programme involving NCL, Indian Oil Corporation, Faridabad, Lubrizol India Ltd.,

Thane, and Centre for High Technology, New Delhi. The copolymers are comparable in performance to commercially available viscosity index improvers. This is mainly due to the unique macromolecular engineering capabilities of metallocenes which are not present in other catalysts. NCL is exploring the possibility of setting up a pilot plant for further work jointly with a major Indian polyolefin manufacturer.

◆ Water-soluble Polymers

Acrylamido methyl propanesulphonic acid (AMPS) is a highly reactive water-soluble monomer containing a pendant sulphonic acid group. Polymers and co-polymers of AMPS have outstanding thermal and metal tolerance properties and show enhanced hydrolytic stability. AMPS is used as a comonomer with acrylonitrile, in the manufacture of acrylic fibre. AMPS homopolymers are used in water treatment plants, paper industry, oil fields, etc. Presently, AMPS's requirement of 500 TPA is being met by imports. The sodium salt of methyl allyl sulphonic acid (Na-MAS), an intermediate in the process for AMPS, is also used as a co-monomer for acrylic fibres.

The synthesis of AMPS comprises two steps : synthesis of NA-MAS and condensation of Na-MAS with acrylonitrile to get AMPS. Process development on a laboratory scale has been completed for Na-MAS and AMPS. Based on the data collected, conceptual designs have been prepared and an economic analysis has been carried out.

Copolymers of AMPS with acrylic acid or acrylamide and terpolymers with styrene and styrene sulphonic acid in two different molecular weight ranges have been developed for application in water treatment as scale inhibitors. High molecular weight copolymers of AMPS with acrylamide and a few terpolymers with N-octyl acrylamide and vinyl pyrrolidone have also been developed for use as mobility control agents for enhancing oil recovery.

Pyrene-labelled polyacrylamides and polyacrylic acids are widely employed for polymer conformational studies by fluorescence spectroscopy. A knowledge of molecular characterization in terms of molecular weight and molecular weight distribution assumes great significance in conformational studies. The hydrophobic nature of pyrene groups interferes with the characterization studies employing gel permeation chromatography (GPC). It has been observed that a low concentration of sodium dodecyl sulphate incorporated in the mobile phase eliminates this effect and the GPC analysis yields polydispersity values based on true size exclusion principle.

◆ Surface Modification of Plastics for Biomedical Applications

Polystyrene (PS) is used for making a variety of disposable products used in animal cell culturing, carrying out the ELISA test; such products include diagnostic and other medical kits. However, the surfaces of such products need to be modified to improve their adhesion and binding properties. A method has been developed to modify the surface of PS plasticware used for animal cell culturing and PS ELISA plates and tubes to make them suitable for antigen/protein binding. The surface treated plasticware show good cell adhesion when used for various cell cultures, which is comparable to that of imported PS plasticware. Similarly surface treated ELISA plates and tubes show good antigen binding properties useful for ELISA and diagnostic kit applications.

◆ Living Anionic Polymerization

Room temperature polymerization of methyl methacrylate has been demonstrated successfully with a reasonable control on molecular weight and molecular weight distribution by using a novel metal-free carbanionic initiator. Tertiary-butyl acrylate has also been polymerized at room temperature with near quantitative yields and reasonable control on molecular weight, using the same initiator.

Polyisoprene (M_n : 1500) has been prepared and a hindered amine stabilizer has been attached to it successfully. The polymeric stabilizer thus produced has been evaluated and compared with a commercial stabilizer from Ciba-Geigy and found to be at least equally effective when used in the stabilization of polyolefins.

◆ Polymers with Controlled Architecture

Polystyrene has been successfully grafted to poly(ethylene-vinyl norbornene) copolymer by the living anionic polymerization route. This makes it possible to solubilize the polyolefins in common solvents such as THF. The aromatic ring of polystyrene is available for further chemical modification. The grafted copolymer has hydroxyl end-groups which introduce some hydrophilicity in the polyolefin. The grafting is expected to decrease the difficulties associated with accumulation of static charge in polyolefins and with their dyeability.

Polydiene living ends have been coupled linearly with 1,1,1-(tribromomethyl oxazoline). This is a potential three-arm star polymer when the oxazoline functionality is opened. The water-soluble polyethyleneimine arm makes it an amphiphilic star polymer.

◆ Degradation and Stabilization of Polymers

The mechanism of degradation of heterophasic ethylene-propylene (E-P) copolymers and high-impact polystyrene (HIPS) induced by heat and light has been elucidated. Based on the studies, it has been concluded that in heterophasic E-P copolymers the weak sites, where degradation is initiated, are in the amorphous region of isotactic polypropylene and in HIPS they are in the rubbery polybutadiene phase. E-P copolymers have been stabilized by HALS-based commercial additives. HALS-based additives have also been synthesized and bound to E-P copolymers. The photostabilizing efficiency of the polymeric HALS and polymer-bound HALS was found to be better than that of commercial HALS.

◆ Conducting Polymers

Conducting polymers are materials with important applications in electronic devices. Both intrinsically conducting polymers (ICPs) as well as conducting polymer composites (CPCs) have been synthesized and studied at NCL.

High piezoresistivity has been observed in conducting polymer composites obtained by in situ deposition of conducting polypyrrole in thermoplastic elastomers. These show a large change in their electrical resistance (10^2 ohm-cm to 10^4 ohm-cm) with the application of low pressure (finger touch level, about 140 gm/cm²). Such materials have potential applications in touch-sensitive devices such as switches, computer touch screens, and electronic note pads.

◆ Volume Transitions in Polymeric Gels by Deformation and H-bonding

Stimuli responsive gels are attracting increasing attention in novel applications in biotechnology, separations, robotics, etc. Research on the synthesis of newer stimuli responsive gels is being carried out worldwide. A study of the effect of deformation and hydrogen-bonding interactions on the volume transitions of certain gels carried out at NCL has revealed a potential for developing gels which swell on being sheared and through H-bonding attractions. The first commercial manufacture of smart gels about to commence in the near future and, in this context, NCL's research assumes great importance.

◆ Solid State Processing

Sintering of PTFE: Powder processing of polymers involves compaction of the polymeric powders followed by sintering at a temperature above the melting point of

the polymer. The properties of the finished products are dependent on the process parameters used during compaction and sintering. In the case of semi-crystalline polymers, the properties are governed by the crystalline morphology developed during the sintering cycle. The crystalline morphology that develops during cooling of the sintered product is influenced by the crystallization rate and crystallization temperature which, in turn, depend on the temperature profiles across the cross-section of the product. A computer simulation of temperature profiles during the sintering cycle of PTFE billets, based on heat transfer equations, was carried out. Interesting observations have been made of the effect of sample geometry, materials properties and modification of sintering cycle on the temperature profiles with respect to the physical processes that take place during the sintering cycle.

Sintering is a critical operation since the mechanical properties of the products are determined by the inter-particle coalescence and diffusion of polymer chains across the interface. A study of the strength development during sintering of PTFE and parameters such as powder particle size, compaction pressure, and sintering time and temperature was carried out. The results will be useful for designing the sintering cycle for PTFE.

Structure Development in Polymers : Research on structure development in polymers under different processing and irradiation conditions has been carried out. Study of crystallization of polymers yields valuable information on the processing of polymers, since the structure developed during crystallization influences the mechanical and physical properties of the polymer product. The crystallization of polymers takes place under non-isothermal conditions during processing. Hence, a study of non-isothermal crystallization is essential for optimizing the processing conditions to obtain a product with desired properties.

A simple approach based on an analysis of the non-isothermal crystallization behaviour of a polymer, to characterize the inherent crystallizability of the polymer and its sensitivity to processing conditions has been worked out at NCL. It has shown that a simple graphical analysis of the non-isothermal crystallization parameters can be used to quantify the inherent crystallizability of polymers and the sensitivity of its structure development to changes in the cooling rates.

The effect of high temperature on the chemical as well as physical structure of polyphenylene sulphide (PPS) in the molten state during curing has been studied. It was observed that the thermal parameters such as melting point and heat of fusion are very sensitive to the structural changes and can be effectively used to monitor the curing process. The effect of curing environment was also investigated and it was

found that the rate of change of structure is considerably higher in air than in nitrogen. The results were analyzed on the basis of the different mechanisms for curing of PPS.

Irradiation of polymers brings about significant changes in their structure and, ultimately, in their performance. In many of the applications of PPS, an engineering polymer, it is subjected to high energy radiation. The changes in the melting and crystallization behavior of irradiated PPS were investigated. The retardation in the crystallization of irradiated PPS has been attributed to the possible changes in the chemical structure of the polymer.

◆ Novel Composites Using Crystal Engineering

Composites possessing controlled morphology are materials with hi-tech applications. Nanoparticulate polymeric composites of cadmium sulfide have been synthesized using the polymer-induced crystallization technique. The *in situ* formation of cadmium sulphide in the polyethylene oxide matrix showed the formation of a predominantly beta phase which is difficult to obtain by conventional methods of crystallization.

◆ Synthetic Membranes for Gas Separation

Synthetic membranes with oxygen permeability of $2-5 \times 10^{-5}$ barrers/cm and oxygen/nitrogen selectivity in the range between 3 and 4 are useful for enrichment of oxygen. A novel polyarylate membrane with an oxygen permeability of 2×10^{-5} barrers/cm and an oxygen/nitrogen selectivity between 8 and 10 has been developed. A thin film composite membrane based on polyarylate is also being developed for oxygen enriching.

Polysulfone coated TFC membranes developed at NCL have been used to fabricate a prototype oxygen-enriched air delivery system. The system, suitable for use with a single patient, delivers 7-9 lpm air with 32% O₂ content. Testing of this system on patients in the operation recovery room is in progress at a hospital that has collaborated with NCL in the research. Work on an improved coating method has led to a 50% reduction in the membrane area requirement, with the selectivity remaining the same.

◆ **Commercial Applications of Membranes**

The objectives of this project were to identify membranes and develop processes for manufacturing them, and to develop membranes on a pilot plant scale. Several applications related to dairy, drinking water, and pharmaceuticals are being examined in collaboration with other institutes, companies and universities. A major effort was made on concentration of skim milk with specially designed spiral modules. Reverse osmosis membranes based on thin film composites and modules fabricated at NCL were used for carrying out 3x concentration. The membrane module viability was tested through more than 30 successive concentration/cleaning cycles. The milk concentrate could be transported more easily and used for making products like skim milk powder, *khava*, *basundi*, and ice cream. The viability of processing the concentrate was checked by preparing sweetened condensed milk after re-mixing fat.

◆ **Microfiltration Membranes**

Microfiltration (MF) membranes are widely used in pharmaceutical, analysis, and clarification applications. New formulations to prepare polyvinylidene fluoride and polyamide based MF membranes with a pore size of 0.2 and 0.45 micron have been developed. Scale-up of the fabrication process is in the design stage.

BIOTECHNOLOGY

BIOTECHNOLOGY

Microbial Technology

◆ Microbial Xylanase

(a) *Cellulase-free xylanase* : Fermentation studies were continued to optimize conditions aimed at maximizing enzyme activity and productivity. Studies to optimize downstream processing and recovery were also carried out in the pilot plant facility established with financial support from the Department of Biotechnology. Enzyme samples have been sent to reputed organizations within and outside the country for evaluation of their suitability for paper biotechnology application.

(b) *Alkalo-tolerant Fungal Xylanase* : A *Cephalosporium* strain capable of growing and secreting cellulase-free xylanase under high alkaline conditions was identified and laboratory-scale fermentation parameters were optimized. The report on this success was the first of an alkalo-tolerant and cellulase-free fungal xylanase.

(c) *Screening for Alkaline Xylanase and Cellulase Producing Microbial Strains* : A project for exploring and identifying microbial strains capable of secreting high activity xylanase and cellulase, tolerant to high temperature and high pH, has been undertaken. Innovative techniques to isolate promising strains have been applied in this screening programme, and strains isolated in pure culture are being evaluated in fermentation experiments for their ability to produce the enzymes with the desirable properties.

(d) *Molecular Biological Studies on Microbial Xylanases* : *Chainia* xylanase expression signals were recognized by cloning an *E. coli* strain. Addition of Ca^{++} or PEG raised the tolerance of the xylanase to heat to 60°C and also made it stable under alkaline pH conditions. The alkalothermophilic *Bacillus* xylanase gene was successfully cloned and expressed in *Bacillus subtilis*, a xylanase-proficient strain. Expression of recombinant xylanase was confirmed through cross-reactivity with antibodies raised against purified xylanase from parent strains. Based on xylan hydrolysis patterns, the difference in the mode of action of the two xylanases was established.

◆ Alkaline Proteases for Leather Industry Application

Investigations on an alkalophilic actinomycete (designated BOA-3), isolated from a natural high-alkaline habitat, have led to the identification of an alkaline protease active at a high alkaline pH and high temperature. The enzyme has also shown considerable stability and activity in the presence of chromium ions, a feature of significance to leather industry applications.

Work on alkaline protease from the fungus *Conidiobolus* was continued and the enzyme production from two strains was optimized on a laboratory scale.

A collaborative programme has been initiated with Central Leather Research Institute, Madras, to develop the enzyme-based technologies for leather industry applications, using the strains that have already been identified and those that would be identified in the on-going screening.

◆ Germ Plasm Resource Pools for Microbial Biotechnology

(a) *Mycological Taxol Fermentation* : A screening programme to isolate fungal strains, particularly endophytes, and screen them for their ability to produce the anti-cancer compound taxol, or its precursor 10-deacetylbaccatin (10-dab) has been initiated. Pure cultures of over 50 fungal strains are being screened and evaluated for useful leads, in collaboration with the Division of Organic Chemistry Technology.

(b) *2-Ketogulonic Acid Fermentation* : 2-Ketogulonic acid (2-KGA), a key intermediate in the production of ascorbic acid, is currently being produced with genetically engineered bacterial strains. An exploratory survey has been initiated to isolate natural populations of acidogenic bacteria and study them for their ability to synthesize 2-KGA.

(c) *Proteinase Inhibitors for Potential Therapeutic Applications* : Proteinase inhibitors are gaining importance in antiviral and AIDS therapy; aspartyl proteinase inhibitor has been recognized as of particular importance. Exploratory studies have been initiated to screen actinomycetes and bacilli to identify potential strains and develop fermentation processes for their production.

◆ Chitin Degrading Enzymes

Chitinase and chitin synthetase inhibitors are important for their potential as biocontrol and antifungal therapeutic agents. Strains of *Myrothecium verrucaria* secreting chitinase and their application to control mosquito (*Aedes aegypti*) larvae

were investigated. In the case of both I and IV instar larvae, 100% mortality was observed in 24 hours after the application of crude chitinase preparation. The chitinase complex of *Metarrhizium anisopliae*, isolated from a natural infection on *Pyrilla*, is being studied for its biocontrol potential. Basic studies on the rare dimorphic mucoralean fungus, *Benjaminiella poitrasii*, with regard to chitin synthesis and degradation were continued.

◆ **NADP-/NAD-Glutamate Dehydrogenase Ratio in Yeast-Mycelium Transition**

It has been shown for the first time that NADP-/NAD-glutamate dehydrogenase ratio has an evidential cause-and-effect relationship with the yeast-mycelium transition in *B. poitrasii*. In this regard, a high GDH ratio (around 0.15) was associated with the yeast form. This finding was supported by the data on monomorphic (yeast form) mutants.

◆ **Penicillin Acylase**

Bacillus sphaericus fermentation parameters were investigated to optimize intracellular penicillin acylase formation. Purification and characterization of the enzyme have been carried out. Modification of lysine, cysteine or serine residues resulted in loss of enzyme activity indicating that they play an essential role. Enzyme-rich cells were immobilized in porous polyacrylamide beads and tested by both batch and continuous modes.

◆ **Encilium Technology**

Protoplast fusion was employed to transfer the property of producing killer toxin to flocculent Encilium yeast strain. The modified strain retained the flocculence and also showed the presence of the induced property.

The desired effect of silicalite (zeolite) in enhancing fermentation rates and ethanol tolerance has been established by exhaustive investigations. Basic studies on the use of yeast cells entrapped in porous polyacrylamide gels in ethanol production from molasses were also continued.

◆ **Microbial Nucleases**

Fungal single-strand specific nucleases with potential as analytical tools in molecular biology research have been investigated. Of these, the S1 nuclease (a zinc metalloprotein) from *Aspergillus oryzae* is widely used as an analytical tool. This enzyme has been purified to homogeneity and characterized in detail. Chemical

modification studies of the enzyme revealed the involvement of lysine in substrate binding, histidine in catalysis and that of carboxylate groups in metal binding. Affinity labelling studies indicate the probable involvement of lysine 111 in substrate binding.

Purified S1 nuclease has also been immobilized via its carbohydrate moiety to Con A-sepharose and Amino-Bio-Gel P2 with high retention of activity and stability. It has been demonstrated that the bound-enzyme can be used as a reusable analytical tool.

A strain of *Rhizopus stolonifer* showed high activity for extracellular RNAase. Optimization for maximum enzyme production has been partly completed. The enzyme has been purified to homogeneity and the characterization is in progress.

◆ **Marine Biotechnology**

Marine algae are known to produce bioactive compounds. One such seaweed, the chlorophycean *Ulva lactuca* produces an antibacterial agent. Success in growing the species under axenic controlled conditions to produce protoplasts and regenerating them has been achieved. The focus is now on mutation of species and regeneration of thalli. The nature of the antibacterial compound is being studied. Marine bacteria (*Vibrio*) capable of producing a protease (an important industrial enzyme) are under investigation.

◆ **Microbe-enhanced Oil Recovery**

Strains growing at 55°C, 65°C and 75°C have been isolated. The anaerobic strains growing at these temperatures have been found to release 30 - 50% of the trapped residual oil from sand packs. Experiments on core-flooding and core-penetration are in progress.

◆ **Oil Degradation**

Several oil degrading organisms are being studied with a view to improving their degradation abilities.

◆ **Global Carbon Flux**

Coordinated investigations of carbon flux from atmosphere to ocean to sediment are underway in the Arabian Sea.

◆ Protein-Carbohydrate Interactions

Lectins have been isolated from *Xanthomonas campestris*, *Agrobacterium radiobacter* and *Fusarium oxysporum*. These lectins show monosaccharide specificity towards galactose or N-acetylgalactosamine. Both lectins purified from the seeds of *Artocarpus hirsuta* show unusually high binding affinity for their specific monosaccharides.

◆ Rice Biotechnology

The main emphasis is on the development of DNA fingerprinting technology and RFLP analysis of rice genotypes. This work is a part of the International Rice Biotechnology network of Rockefeller Foundation, USA. Linkages have been established with Indian agricultural universities/institutes for DNA marker assisted breeding programmes.

DNA fingerprinting of rice using oligonucleotide probes and hypervariable DNA sequences have been carried out. Since DNA fingerprinting is useful in seed certification and protection of patent rights, it is now proposed to extend it to other crops like cotton, pearl millet and wheat.

◆ Gibberellic Acid

Gibberellic acid is the plant growth regulator widely used for growing seedless varieties of grapes. Around 2,000 Kg of gibberellic acid at a cost of US \$ 2 million is imported every year as it is not produced in India. Production of gibberellic acid, a secondary metabolite, is carried out by fungal fermentation. It is a very skilled fermentation technique and therefore a carefully guarded secret.

Several mutants of a selected strain of *Gibberella fujikuroi* were developed and screened for GA₃ production. Fermentation conditions were optimized in a 20-L fermenter for the production of the selected mutant. Purification of the product yielded above 85% pure gibberellic acid, which was tested on grape vines in the growing season. The product was compared with the imported gibberellic acid and was found to be equal to it in the activity. Detailed cost calculations of the process performed on different production scales indicate the possibility of its commercialization in the near future.

◆ Microbial Xylanases : Scale-up and Downstream Processing

Xylanases, the enzymes which hydrolyze xylan present in the wall of plant cells, have potential application in paper industry for biopulping. *Chainia sp.* NCIM 2980 and an alkalophilic *Bacillus sp.* 87-6-10 strain produce cellulase-free xylanase. Optimization of fermentation conditions for the production of alkaline xylanase was carried out in a one-litre fermenter. Enzyme yields comparable to those in flask experiments (140 IU/mL) were obtained. Scale-up studies in a 10-litre fermenter were also conducted and reproducible activities of approximately 100 IU/mL were obtained.

Downstream processing involving flocculation and microfiltration, followed by ultrafiltration, using cellulose acetate membranes prepared at NCL, were found suitable for achieving 10-fold concentration of the enzyme from fermentation broth. Preliminary adsorption studies were also carried out for the recovery of the enzyme. 100 L scale fermentation trials are planned to be taken up.

◆ Membrane Bioreactors

Fundamental engineering studies in the analysis of membrane bioreactors and transport processes encountered in them, using CGTase production by *Bacillus sp.* as the model, were carried out in a two-stage crossflow cell recycle bioreactor. Growth and CGT production by *Bacillus macerans* in batch mode were optimized.

◆ Use of Membrane and Adsorptive Technology for Improvement of Immunobiological Manufacturing Practices

Adsorptive and membrane technology can be used to improve the basic technology of downstream processing of immunobiologicals. Experiments on recovery of foot and mouth disease (FMD) vaccine and *Clostridium welchii* type C, D vaccine were carried out with very good results. The process conditions were optimized. The aim is to develop improved downstream processing methodologies for vaccine manufacture using membranes and adsorption chromatography techniques. The improvements relate to dose size, time taken for processing of vaccine and improved purity/efficacy of vaccine. The objectives include assessment of vaccine of improved quality with respect to dose/volume, antigen quantification and efficacy in natural hosts. Pilot plant-scale experimentation leading to commercialization of the technology at the Bharatiya Agro Industries Foundation facility at Wagholi, Maharashtra, is in progress.

Plant Tissue Culture

◆ **Biotechnological Approach to Improvement of Legumes/Cereals/Oil Seed Crops/Spices**

(A) **Genetic modification of plants through protoplast technology/plant transformations:** The aim is to improve grain legumes such as chick pea (*Cicer arietinum*,) and pigeon pea (*Cajanus cajan*) through plant transformation techniques is to develop lines with desirable agronomic traits. Development of methods for the generation of transgenic lines and regeneration of explants into plants is the basic prerequisite in the improvement programme.

Various chimeric genes coding for kanamycin/phosphinothricine/gentamicin have been used with chick pea explants. Three plasmid constructs, four antibiotic selection systems and four genotypes of chick pea have been screened for generation of transgenic lines of the legume. The transformation was confirmed by the Southern Hybridization and GUS Staining techniques.

Various explants (mature embryo axes, leaf, immature cotyledonary segments and immature embryos) of chick pea have been regenerated into plants via somatic embryogenesis. The origin and development of somatic embryos formation was determined by histological and scanning electron microscopic studies.

(B) **Plant protoplast technology and morphogenetic studies in Chick pea and Pigeon pea:** The grain legumes (*Cicer arietinum* L. and *Cajanus cajan* L. Mill sp) are being studied with the objective of improving them through transfer of genetically important traits from wild varieties to the cultivated varieties.

Intact seeds, shoot apices and cotyledonary nodes of various genotypes of chick pea and various explants of different pigeon pea genotypes (intact seeds, mature embryos, epicotyls, cotyledons and leaf) were regenerated into plants via direct organogenesis and/or through organogenesis with an intervening callus phase.

The regenerated plants have been rooted, hardened and transferred to field. The regeneration systems thus developed are useful for plant transformation studies involving chimeric genes coding antibiotic resistance and/or genes of desirable traits.

(C) **Development of transgenic cotton varieties improved for resistance to cotton bollworms:** Cotton (*Gossypium* spp.) is an important fibre and vegetable oil crop in India. It needs to be improved for fibre quality, disease and pest tolerance, etc.

Production of cotton hybrids through conventional plant breeding methods is limited by incompatibility barriers, increased costs and yield losses from incomplete fertility restoration in hybrids.

The major prerequisite for development of transgenic cotton lines resistant to bollworm is a reproducible protocol for direct differentiation of explants of different cultivars of cotton through organogenesis and/or embryogenesis, and plant transformation for incorporation of the Bt gene.

A DBT-funded project addressing these problems has recently been initiated.

(D) **Peanut improvement through somatic embryogenesis:** Peanut is a protein-rich leguminous crop cultivated widely for edible oil. The crop is susceptible to diseases and the oil becomes rancid upon exposure to air and heat owing to oxidation. Development of high yielding cultivars with improved agronomical characters is thus necessary to reduce crop loss and increase oil shelf life. Application of recent biotechnological approaches to improvement of crops requires a reliable method for morphogenesis *in vitro*. Reliable protocols have been standardized for regeneration of plantlets *in vitro* via direct embryogenesis and organogenesis. The work was extended to determine the storage lipid in the somatic embryos. The results show that the somatic embryogenesis in peanut can now be exploited for genetic improvement of the crop and for understanding the process of oil synthesis *in vitro*.

◆ **Production of Taxol, An Anticancer Alkaloid**

This is an inter disciplinary project jointly undertaken by the Plant Tissue Culture, Organic Chemistry Technology and Biochemical Sciences Divisions. Production of taxol, or its precursor 10-DAB, from fungal cultures is an attractive alternative proposition to extracting taxol directly from the bark of the tree, an approach that destroys the tree. Callus cultures have been established under aseptic conditions and procedures have been established for quantification and analysis of taxanes. Methods to break dormancy and germination of *Taxus* seeds have been standardized. Sterile cultures have been raised from seed embryos and the *in vitro* seedlings are now being used for raising suspension and callus cultures for transformation studies with agrobacterial strains to induce hairy roots. A screening programme has been undertaken to isolate fungal strains associated with *Taxus* bark, needles, stems and also from litter collected from *Taxus* plantations. The focus has been on identifying basidiomycetous as well as non-sporulating uncommon strains of the fungi. So far 40 such cultures have been brought into the pure culture; preliminary laboratory scale experiments to grow and screen the mycelial biomass for the possible occurrence of

taxol and related compounds are in progress. Techniques for rapid detection of taxol in the fungal biomass are also being worked out.

◆ **C₃/C₄ Photosynthetic Gene(s) Regulation**

Cis- and trans- acting elements/factors play a major role in the regulation of photosynthetic genes. To understand these mechanisms the NADP-ME and PEPC genes have been isolated from sugarcane leaf c-DNA library. Sequencing of the NADP-ME gene of approximately 1.9 kb in size has been carried out. The PEPC gene has been partially sequenced. Simultaneously, a 1.0 kb fragment of the PEPC-PK has been amplified using fully degenerate primers by the polymerase chain reaction technique.

◆ **Molecular Cloning and Expression of Genes for PHB-V**

PHB-V is a biodegradable polymer accumulated by certain bacteria. It is currently being synthesized by bacterial fermentation. The genes responsible for the synthesis can be made to express in higher plants by genetic engineering. Towards this end, the genomic DNA from *Alcaligenes eutrophus* was digested with appropriate restriction enzymes and then a genomic library was built. The library was screened with oligo probes for conserved regions; positive clones have been isolated and are being characterized.

◆ **Standardization of Protocols for Multiplication of Highly Sought Commercial Ornamentals/Orchids**

The project was initiated keeping in view the growing interest in the commercialization of tissue culture methodologies for large scale production of ornamentals and orchids. Six plant species with a potential for export, namely, saintpaulias, carnations, *Dendrobiums*, lilies, gerberas and chrysanthemums were chosen and tissue culture protocols were standardized for their large scale propagation. The documented protocols for saintpaulias, *Dendrobiums* and lilies are available to parties with commercial interest. They can demonstrate the protocols at a production level of 1,000 plants.

◆ **Technology Transfer, Biotechnological Evaluation and Clonal Multiplication of Eucalyptus, Bamboo and Salvadoria**

Under Phase-I of the NCL-NABARD project, protocols have been standardized for micropropagation of *Eucalyptus camaludensis*, *Eucalyptus tereticornis*, *Dendrocalamus*

strictus and *Salvadora persica*. A method has been standardized for *ex vitro* rooting of microcuttings, resulting in considerable cost savings. Under the on-going Phase-II of the project, the technology has been successfully transferred to three regional laboratories viz. GRASIM Forest Research Institute (GFRI), Harihar, Karnataka; Central Salt and Marine Chemicals Research Institute (CSMCRI), Bhavnagar, Gujarat; and Tamil Nadu Forest Plantation Corporation, Pudukkottai, Tiruchirappalli Dist., Tamil Nadu.

At present, NCL is monitoring the plant production in these laboratories. All three laboratories are now producing plants through tissue culture technologies developed at NCL, for field plantation and trials. A genetic garden of eucalyptus species has been established at GFRI, Harihar, with 13,000 tissue culture-raised plants from different genotypes. The field trials of species of *Eucalyptus* and *Salvadora* are being conducted in a 10-hectare plot each. Data on field performance of tissue culture raised plants is being collected for ascertaining the feasibility of using the tissue culture technology in afforestation programmes. Demonstration programmes have been conducted in lands belonging to private farmers, aimed at involving the farmers and employing the technology for the upliftment of the rural people.

◆ **Tissue Culture of Pinus Species**

Success was achieved in bud-break and plant formation in three species of pinus. This work is being continued in collaboration with the Swedish Agricultural University, Uppsala. The main objectives of this project are to study the morphogenetic response of *pinus* species, and to improve them through biotechnological methods.

◆ **Clonal Multiplication of the Alphonso Mango**

Alphonso is one of the most sought after mango varieties in the domestic and foreign markets. Developing true-to-type planting stocks of this variety for large scale plantations has been identified as an urgent necessity. Tissue culture work on the alphonso mango has been initiated under a DBT sponsored project. An embryogenic line has already been established.

◆ **Flowering in Bamboo**

In vitro induction of flowering in bamboos leads to increased production and improved species. A better understanding of bamboo flowering is being gained through detailed studies of the floral biology and breeding behaviour in many bamboo species. This is necessary for understanding their reproductive biology, comparing *in*

vitro and *in vivo* flowering patterns, and modifying *in vitro* flowering protocol. So far, the floral biology and the breeding behaviour of three species, *Bambusa arundinacea*, *Dendrocalamus strictus* and *Melocanna bambusoides*, have been investigated.

A novel method of dividing bamboo genera into two categories based on maturation of reproductive structures is useful in understanding the interrelationships between bamboo species and genera. The potential of selection as a method for improvement in bamboos has been reported. A combination of selection and long vegetative growth phases can increase bamboo production considerably. Work on identifying the factors responsible for precocious flowering is in progress.

ORGANIC CHEMISTRY AND TECHNOLOGY

ORGANIC CHEMISTRY AND TECHNOLOGY

Bio-organic Chemistry

◆ DNA Molecular Recognition

Understanding the physical basis of recognition of nucleic acids (DNA, RNA) by small molecules (drugs) and macromolecules (proteins) is an important area of current interest. In this context rational modification of the structural components of nucleic acid bases and the sugar-phosphate backbone has emerged as a powerful technique for investigating molecular recognition. Towards this end the effect of 5-amino-pyrimidines, 8-amino purines and N⁴-spermine on the biophysical properties of nucleic acids has been studied. It was found that while 8-amino-dA forms a reverse Hoogsteen base pairing, 5-amino-dU has no effect on DNA duplex formation. Interestingly, oligonucleotides in which N⁴-spermine-dC is incorporated exhibited favourable triple helical properties, including stability at pH 7.0, making them suitable for identifying them as lead compounds in drug development. 5-Amino pyrimidines and their fluorescent conjugates with fluoresceinyl fluorophores, have been synthesized and their utility in PCR-based nucleic acid diagnostics has been demonstrated. By employing a novel fluorescent assay, sugar-DNA interaction in the minor groove has been discovered.

◆ Nuclease Mimics

Non-enzymatic hydrolysis of biological phosphates is currently one of the most pursued topics owing to its challenging chemistry and potential applications in biotechnology. A novel dinucleotide-histamine conjugate was designed, synthesized and shown to undergo accelerated self-hydrolysis in presence of zinc chloride. This is a mechanistic mimic of ribonuclease and has a potential for elaboration to a custom-designed nuclease. An approach towards developing models for the DNA photo repair enzyme, photolyase, has resulted in the synthesis and structure elucidation of a new trans-syn T-T photodimer. The mechanistic aspect of "chemical nuclease" activity of the metal chelator drug, desferal, has been elucidated.

◆ Modified Nucleosides

The following modified nucleosides with potential antiviral activity were synthesized from various 3'/3',5'-O-mesyl substituted pyrimidine nucleosides :

- ** A series of 2,3'-O-anhydro-5'-deoxy-5'-alkylamino-thymidines.
- ** A new class of iso-cytidine derivatives, prepared from 2,2'-O-anhydro-3'-O-mesyluridine.
- ** Enaminonucleosides and nucleoside aminoalcohols.

A highly reactive analogue of AZT, 3'-deoxy-3',5'-(vinyl sulphonyl) thymidine has been synthesized and its reactivity with various nucleophiles studied. It has a potential for development as an enzyme inhibitor and for building dephosphono nucleic acids.

◆ Chemistry of Inositols/Neighbouring Group Effects

Myo-inositol phosphates and their lipid derivatives have attracted considerable attention in recent years owing to their involvement in cellular signal transduction pathways. A simple method has been devised for the preparation of 2,4-di-O-benzoyl-myo-inositol-1,3,5-ortho-formate in gram quantities. This has been used for the preparation of a number of protected myo-inositol derivatives which are useful intermediates in the synthesis of myo-inositol phosphates. During this investigation, an unusual hydroxy group assisted alkylation of 2,4-di-O-benzoyl-myo-inositol orthoformate was discovered.

The reaction of 2-formylbenzenesulphonyl chloride with amines gave N-substituted 2-formylbenzenesulphonamides which have interesting tautomeric properties. These investigations throw light on the nature of the interaction between a carbonyl group and a sulphonyl group in close proximity. Some of these derivatives were tested for antiviral activity.

Organometallics

◆ Investigation of Organozinc Compounds

Mixed dialkylzinc compounds, obtained in good yields from methylzinc iodide and Grignard reagents, were found to react smoothly with aldehydes with preferential transfer of alkyl/aryl group; the methyl group remains the non-migrating moiety. Use of C₂-symmetric diols and diamines as potential auxiliaries for chiral organozinc reagents, is being studied.

◆ Organotransition Metal Chemistry

Fischer carbene complexes are important intermediates in organic synthesis. A convenient, biphasic alkylation protocol has been developed for making them. An unprecedented metal-aldol reaction has been observed which leads to cyclic carbene complexes with highly shielded carbene carbon. Stereoselective annulation was achieved on selected arene-chromium templates, which may provide usefully functionalized, and advanced intermediates for biologically relevant target molecules. Conformation of molybdenum *pi*-allyl complexes was shown to be guided by a weak but tunable CH/*pi* interaction in solution.

◆ Organomolybdenum Complexes

Stable binuclear dioxomolybdenum (VI) complexes were prepared from flexibly-bridged hexadentate tetra-anionic ligands derived from methylene-ordithio-bis (salicylaldehyde) with benzoyl hydrazide or *p*-nitrobenzoyl hydrazide and dioxo-bis (acetyl acetonato) molybdenum. Spectral and other studies reveal that these complexes achieve a pseudo-octahedral structure via Mo-O--Mo bridging, which is irreversible to quasi-reversible reduction of Mo(VI) to Mo(IV).

◆ Organotungsten Complexes

Mononuclear dioxotungsten(IV) complexes of the type [WO₂L(MeOH)] have been synthesized using the precursor [WO₂(acac)₂] and tridentate Schiff base ligands. The structure of the complex [WO₂(CO)₆H₄CH=NH=C(O)C₆H₅(MeOH)] has been found to be formed by an octahedral coordination for tungsten (VI), and the butylammonium fascile W(VI) to W(VI) EPC value was found to be 1.48 V.

Synthetic Methodologies

◆ Photochemical Organic Synthesis

The carbon-selenium bond in organic compounds has been activated by photoinduced electron transfer (PET) processes as carbocation equivalent. This finding has been applied to one pot sequential selenylation and deselenylation reactions to get functionalized cyclic compounds.

Cross coupling reaction between organoselenium and organosilicon compounds has also been achieved as a new approach to C-C bond formation reactions.

Electrophilic selenium species generated *in situ* by the PET from activated PhSeSePh has been used for carbocyclization of enynes.

A novel photosystem consisting of light-harvesting electron acceptor and sacrificial electron donor has been developed to harvest electrons from sunlight trigger one electron redox reactions in organic synthesis.

New Approaches to Synthesis

◆ Regioselective Formylation of Activated Aromatics

A study of the factors affecting the regioselectivity of arene formylation was initiated, with the objective of developing novel routes to key acyl aromatic intermediates in pharmaceutical manufacture. It was observed that an increase in the steric requirements of the reagent and of substituents on the substrate is beneficial for directing formylation away from the ring substituent.

◆ Stereodivergent Synthesis of α -Hydroxy Acids

A new approach to enantiomerically enriched α -hydroxy acids, which are important intermediates in the synthesis of natural products and biologically active molecules, was developed. The procedure involves a reagent-based stereodivergent reduction of chiral α -keto amides derived from amino alcohols. Substrate directed reduction, involving a hydroxyl group in the substrate, generates one of the diastereomers, whereas reduction with conventional borohydride reducing agents generates the other. The reductions proceed with good stereoselectivity at ambient temperature and offer certain advantages over existing reductive processes for α -hydroxy acid synthesis.

◆ Diastereoselective Synthesis of β -lactams

Homochiral β -lactams are important intermediates for biologically active compounds, such as β -lactam antibiotics, alkaloids, aminosugars, and α -hydroxy- β -amino acids. A diastereoselective synthesis of β -lactams has been achieved using homochiral imines derived from (+)-(3)-carene in very good yield. Appropriately substituted β -lactam has been converted into β -amino substituted- α -hydroxyacid, which is an important intermediate for taxol side chain. This approach provides a practical synthesis for α -hydroxy- β -amino- β -phenyl propionic acid, a taxol side chain starting from an easily available natural product.

◆ **Permanganate-based New Reagent**

A new reagent derived from KMnO_4 , tetradecyl trimethyl ammonium permanganate (TDTAP), has been prepared, characterized and used for *cis*-vicinal dihydroxylation of alkenes. It has been used in combination with Me_3SiCl or Me_3SiBr , in the stereoselective and chemoselective *trans*-vicinal dichlorination or dibromination of olefins with excellent yield.

◆ **New Route to Aromatic Amino Acids**

Titanium tetrachloride-mediated chlorocynoethylation of anisole furnished α -chloro- β -(4-methoxy phenyl) propionitrile which could be converted into a tyrosine derivative by a simple reaction sequence.

◆ **Oxazaborolidine-catalyzed Asymmetric Reduction**

Oxazaborolidine-catalyzed reduction of functionalized ketones provided a highly enantioselective route to 1,2-diarylethanedioles, a very useful class of chiral auxiliaries.

◆ **Nitro-olefin Synthon**

A novel conversion of conjugated nitro-olefins to functionalized or non-functionalized hydroximoyl chlorides (nitrile oxide precursors) has been achieved with TiCl_4 -based reagents. The product distribution in the reaction of conjugated nitro-olefins with sulphur ylides depends on the substituent in the α -position: if there is none, nitrocyclopropane is the major product; if an alkyl group is present, cyclic nitronic ester is the major product.

◆ **Phosphacumulene Ylides for Biologically Useful Skeletons**

Phosphacumulene ylides are building blocks of carbocyclic and heterocyclic compounds. *N*-phenyl (triphenylphosphoranylidene) ethanimine mediated one-pot conversion of *N*-substituted anthranilic acid into a variety of antibacterial quinolones via intramolecular Wittig cyclization has been achieved. The synthesis of more biologically useful skeletons, such as pyrrolizidine and indolizidine alkaloids and taxol, is in progress.

◆ **Formation of Tetracyclo [10.2.n.0^{2,11}.0^{4,9}] pentadeca-2,4,5,7,13-pentaene-3,10-diones and Related Systems Induced by Samarium Diiodide**

With a view to synthesizing the carbocyclic frame of taxol with C-ring as the aromatic, the reaction of endo/exo tetracyclo [10.2.n.0^{2,11}.0^{4,9}] pentadeca-2,4,5,7,13-tetraene-3,10-diones with SmI_2 was investigated. Contrary to expectations, however, SmI_2 led to an efficient formation of bicyclic quinones. The generality of SmI_2 -induced efficient oxidation to title bicyclic quinones either from endo or exo tetracyclo [10.2.n.0^{2,11}.0^{4,9}] pentadeca-4,5,7,13-tetraene-3,10-diones has been established. The final products formed are known to be useful for photochemical solar energy storage.

◆ **Polymer Supported Nitrobenzaldehyde as a Selective, Efficient and Convenient Reagent for Deprotection of Oxathioacetals**

In continuation of development of efficient methodologies for functional group transformations, nitrobenzaldehyde supported on a polymer was found to deprotect oxathioacetals efficiently at room temperature to the corresponding carbonyl compounds in the presence of TMSOTf as the catalyst. The salient features of this protocol are the mild conditions of operation, catalytic nature, and the selective deprotection of oxathioacetals of ketones in the presence of aldehyde-derived thioacetals.

The *p*-nitrobenzaldehyde reagent mentioned above, in the presence of TMSOTf, has also been found to be very effective in the selective transformation of thioketones to the corresponding ketones in the presence of thioamides; however, the reagent was not supported on the polymer in this class of reactions.

◆ **Interconversion of Oxathiolanes and the Corresponding Carbonyl Compounds**

It has been observed that TMSOTf is a mild, efficient catalyst in effecting facile interconversion of carbonyl compounds to oxathiolanes and vice-versa under essentially identical conditions.

An interesting point that emerges from this study is that the oxathiolane derived from benzophenone can be converted to benzophenone essentially under identical conditions; this conversion is mediated by TMSOTf catalyst, in a "merry-go-round" fashion.

◆ **Solid Superacids as Efficient Catalysts in trans-Esterification of Ketoesters and Hydrolysis of Allyl Esters**

Solid superacids, being heterogeneous, possess two specific advantages: they can be easily worked up and they can be retrieved with ease. The catalytic activity of solid superacids has been demonstrated in the efficient trans-esterification of ketoesters with primary, secondary and allyl alcohols; use of excess t-butyl alcohol brings about the trans-esterification which yields t-butyl esters which are normally not accessible with conventional reagents.

Selective deprotection of allyl esters in the presence of normal esters has also been achieved by the use of solid superacids.

◆ **Mg/MeOH Mediated Intramolecular Reductive Cyclization of Activated Dienes**

Reductive cyclization of activated dienes can be brought about employing electrochemistry, photochemistry or metals (SmI_2 , Bu_3SnH) but these methods involve the use of expensive and/or toxic reagents and are highly inconvenient. Activated dienes, when treated with Mg in MeOH at room temperature underwent smooth cyclization. A three-membered and five-membered framework could be easily generated by following this methodology. Similarly, a five-membered heterocycle could also be prepared.

Because of the easy availability of magnesium and the mild conditions of operation this strategy has an edge over other methods.

◆ **Ultrasound-irradiated Bromination of Activated Aromatics**

Several activated aromatics have been brominated with high regio-selectivity and yield using N-bromosuccinimide under ultrasound irradiation (50 kHz) in the absence of any catalyst.

◆ **Paclobutrazol**

Paclobutrazol is a well-known plant growth regulator, known specially for its use on fruit plants. Besides enhancing the yield of mango crop, it is also known to induce an annual cycle of flowering in the crop instead of the usual biennial cycle. The preparation of a key intermediate for paclobutrazol, viz. 1,2,4-triazole and of paclobutrazol itself has been standardized. Field trials are underway at Konkan Krishi

Vidyapeeth with paclobutrazol samples supplied by NCL. 1,2,4-triazole is also an important drug intermediate.

◆ **Tenidap Sodium**

Tenidap sodium is a futuristic anti-inflammatory drug with an immense market potential. A laboratory scale process for making it has been successfully developed and is being optimized.

◆ **Roxatidine Acetate**

A laboratory scale process has been developed for making this anti-ulcer drug; process optimization is underway.

◆ **Cetirizine Hydrochloride**

Cetirizine hydrochloride is an antihistamine with high specificity and potency for H-1 receptor and used as an anti-allergic drug. A highly cost effective process for making it was developed and demonstrated to the sponsor.

◆ **Triacontanol**

The preparation of triacontanol, a plant growth regulator, was undertaken as a sponsored project and the product was supplied to the sponsor.

◆ **Optically Active Caronaldehyde**

Optically active caronaldehyde, required for the resolution of dl-4-hydroxy-2-cyclopentenone, an intermediate for the synthesis of prostaglandins, from DV-acid, was prepared under sponsorship and the product was supplied to the sponsor.

◆ **Misoprostol**

Development of a process for misoprostol, an anti-ulcer drug, was undertaken under sponsorship. The methodology had been developed earlier at NCL. Further optimization of the modified three component coupling methodology has been completed.

◆ **D(+) Biotin**

D(+) Biotin, a member of the B-complex group of vitamins, is widely used in poultry feed for rapid growth of chicks and healthy hatching of eggs. The current demand for it is met through imports. A lab-scale process, based on a bench-scale method previously evolved at NCL, has been developed.

◆ **Synthesis and Process Development of Insect Sex-Pheromones**

A useful role can be found for pheromones in integrated pest management. By controlling the mating behaviour of harmful insects they protect plants against the pests. A laboratory scale process for (z)-11-hexadecenal and (z)-9-hexadecenol, both useful pheromones, has been developed.

Zeolite-Mediated Organic Synthesis

◆ **Method for Making EMME**

Ethoxymethylene malonic acid ester (EMME) is an important starting material for the synthesis of several quinolone drugs. An improved method has been developed for the preparation of EMME using clay catalysts.

◆ **Efficient Synthesis of Diacetates from Aldehydes**

Beta zeolite has been found to catalyze the reaction of a variety of aldehydes with acetic anhydride affording the corresponding 1,1-diacetates in very good yields. The generality of the reaction has been established.

◆ **Titanium Silicate Molecular Sieve-Induced Epoxidation**

The epoxidation of styrene was studied using the titanium-silicate molecular sieve, TS-1, as the catalyst and dilute (25%) H₂O₂ as the oxidizing agent. Mainly, two types of reactions were observed: the major reaction (75-85%) was the epoxidation of styrene and its further isomerization into phenylacetaldehyde; the other reaction was the oxidative cleavage into benzaldehyde (10-20%). Polar and non-polar solvents have a marked influence on product distribution in the reaction.

◆ **Epoxidation of Alkenes Using Beta Zeolites**

Zeolites doped with various transition metals such as Ti, V and Cr, in the framework have been successfully applied to the epoxidation of alkenes and to the oxidation of various amines to azoxy, hydroxylamino and nitro-compounds.

◆ **Synthesis of Bromoaromatics Using Zeolites**

HZSM-5 has been found to catalyze regioselective bromination of activated aromatics with N-bromosuccinimide in excellent yields. The interesting observation made was that, under zeolite mediated conditions, xylene underwent side-chain bromination in significant yields in the absence of any radical initiator.

Synthesis Of Natural Products and Analogs

◆ **Brassinolides and analogs**

Brassinolide and a few analog compounds have been synthesized from readily available 16-dehydropregnenolone acetate. They are potent plant growth promoters and have been used in horticulture and agriculture.

◆ **RU-486**

A laboratory scale process for mifepristone (RU-486), a progesterone antagonist used for terminating early pregnancies, has been completed. The synthesis starts from d(+)-estrone and involves 11 steps.

◆ **Chaetomelic acid A**

Chaetomelic acid A anhydride, a potent inhibitor of ras farnesyl-protein transferase, has been synthesized by a simple, three-step, efficient route. The imidazopyridinium bromide obtained from the reaction of 2-bromopalmitoyl chloride and 2-aminopyridine was reacted with maleic anhydride /NaOAc/AcOH to form chaetomelic acid A anhydride in 62% overall yield.

◆ **Epibatidine**

X-Azabicyclo (m.n.1) alkane ring systems were constructed by (3+2)-cycloaddition of corresponding non-stabilized azomethine ylides. The latter were generated by sequential double desilylation of N-alkyl- α,α' -di(trimethylsilyl)cyclic amines using Ag(I)F

as one electron oxidant, with a variety of dipolarophiles. The strategy has been extended to the synthesis of epibatidine, an analgesic that is 200-500 times more potent than morphine.

◆ **Retronecene**

(+)-Retronecene possessing heptotoxicity and antitumour activity has also been synthesized by a similar strategy.

◆ **(±)Laevigatin**

Laevigatin, a naturally occurring terpene with an unusual skeleton, was isolated from *Eupatorium laevigatum*.

In view of NCL's interest in the synthesis of heritol, heritonin and related compounds, two convenient and efficient methodologies were developed to generate butenolides skeletons (i) via osmylation of beta, gamma-unsaturated esters, and (ii) by direct oxidative conversion of beta, gamma-ester to butenolide by CAN at room temperature.

A short and efficient synthesis of (±) laevigatin has been achieved by the above protocol.

◆ **Utilization of Non-edible Oilseeds for Insect Control and Industrial Purposes**

Isolation of biologically active compounds from three non-edible oil seeds, namely, mahua (*Madhuca latifolia*), undi (*Calophyllum inophyllum*) and nahor (*Mesua ferrea*), has been undertaken for their utilization as botanical insect control agents. The oil and the cake are also upgraded by the removal of biologically active non-lipid compounds.

Fractions prepared from the seeds were formulated and tested for larvicidal activity against *Spodoptera litura* and *Helicoverpa armigera*. One fraction from mahua (MC) and two from undi (UA & UE) showed good larvicidal activity under laboratory conditions; they are now being subjected to field trials on bengal gram. The fractions are also being tested for their insect control activity against pests affecting different economically important crops and some vectors.

Further field trials with Neemrich-I (on cabbage) and Neemrich-II (on bengal gram) have been conducted with consistently satisfactory results.

◆ **Development of Botanical Pesticides**

Bioevaluation Under Laboratory & Field Conditions: Many plant species are reported to possess biological activity, particularly insect control activity. NCL has undertaken development of processes for the preparation of products with insect control activity from five plants: *Annona squamosa* (seeds, leaves), *Acorus calamus* (rhizomes), *Vitex negundo* (twigs), *Tephrosia purpurea* (aerial parts), *Derris* (roots) and *Melia azedarach* (seeds). Extracts and fractions from these plants were prepared and tested for their activity against agricultural pests such as *Spodoptera litura*, *Diacrisia obliqua*, *Helicoverpa armigera* and aphids. Further fractionation of these fractions/extracts to obtain more active principles is underway.

Two products AC₂ & TP₅ (from *A. Calamus* rhizomes and *T. purpurea* aerial parts which showed promising results under laboratory conditions have been field-tested on two crops with good results.

◆ **Utilization of Indian Medicinal Plants**

A laboratory-scale process was developed for isolating camptothecin, an anticancer drug from heartwood of the plants *Natopodytes foetida* syn. and *Mappia foetida*; the yield was 0.075%.

◆ **Taxol and Its Derivatives : Anti-Cancer Drug**

Taxol, taxotere and other taxoid compounds have found therapeutic use as anticancer drugs. A lab-scale process to isolate the taxol intermediate, 10-DAB, from *Taxus baccata* needles has been standardized. Resolution of the side chain of the intermediate has been carried out. Simultaneously, using asymmetric method/catalyst, the chiral diol intermediate has been obtained. Synthesis of the side chain in the required optical form and its coupling to 10-DAB to produce taxol/taxotere is in progress.

◆ **Microbial Transformations of Steroids**

Microbial transformations offer the advantage of regio- and stereo- selective functionalization, especially in steroid systems, leading to key intermediates for the synthesis of pharmacologically important steroidal hormones. Among the cultures screened for the hydroxy derivative of progesterone, *Cunninghamella blakesteena* showed the promise of yielding essentially the 11β-, 14α hydroxy derivative.

Similarly, microbial side chain degradation of the abundantly available cholesterol and β -sitosterol can lead to important organic synthetic intermediates. In this context, an initial exploratory study of the side chain degradation of these steroids led to mixtures of mono and dienic 17-keto compounds; the yields are being optimized.

◆ **Tartaric Acid from Tamarind Pulp**

The process developed for the isolation of L-tartaric acid from tamarind pulp was scaled up to 1 kg tartaric acid per batch.

◆ **Isolation of Food Grade Pectin from Tamarind Pulp**

One of the by-products of tartaric acid from tamarind pulp process is pectin. The crude pectin was further purified and upgraded to obtain a value-added product, making the process more economical.

Others

◆ **Screening of Compounds for Possible Pesticidal Activity**

NCL generates a number of new compounds incorporating a variety of structural features in its extensive research activities. A collaborative programme has been initiated with agrochemical companies to screen these new compounds for possible pesticidal activity. This screening has opened up the possibility of identifying new compounds with pesticidal properties.

◆ **Thiourea**

Thiourea is an important chemical which finds application in the manufacture of pharmaceuticals and pesticides. It is not manufactured in India currently and the entire requirement of the country is met through imports. Its estimated annual demand, for various applications, is about 1000 tonnes. NCL has developed a process for its manufacture from calcium cyanamide and hydrogen sulfide. The process has been demonstrated on a 10 Kg/batch scale to Straw Products Ltd., New Delhi.

MATERIALS CHEMISTRY

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◆ Ceramic Humidity and Gas Sensors

Sensitivity and temperature coefficient values, and the optimum composition were determined for making a device circuit incorporating the new humidity sensing material, lithium stannate, were determined and found to be satisfactory. However, about 4% piece-to-piece variation in resistance was initially found at a constant RH. By improving the preparation procedure and using a critical binder composition the variation was reduced to 1%. Attempts are being made to reduce the variation level to 0.5%.

Novel humidity sensors identified in this connection include sulphated zirconium oxide and boron phosphate; investigations are being carried out to understand the mechanism of proton transport in these materials.

◆ Electrocatalysis using Zeolite Modified Electrodes

Although the catalytic properties of zeolites have been extensively investigated in the last few decades, their electrocatalytic properties in a variety of electrochemical reactions of technological significance remain unexplored. Moreover, recent trends in functionalized or modified electrodes emphasize that the inherent catalytic properties of the substrate electrode and zeolites are particularly useful for this purpose. Different vanadium zeolites have been studied using electrochemical techniques; their electrocatalytic behaviour shows promise of being useful in electrooxidation reactions.

◆ Langmuir Blodgett Films

1. *Spontaneous reorganization of Langmuir Blodgett films:* An interesting aspect of the Langmuir Blodgett (LB) deposition technique is that the superlattice structures can easily be grown. However, it was found that considerable reorganization of alternating layers of fatty acid films with different cations and different chain length molecules was occurring. This has thrown up many fundamental questions regarding the processes occurring at the air-water interface as well as in the aqueous medium, which are being looked into.

2. *Spontaneous self-organization and ion exchange in LB films:* Vacuum-deposited films of fatty acids or long chain amines, when dipped into suitable electrolyte solutions, display ion exchange and molecular reorganization phenomena.

The reorganization leads to a lamellar structure similar to that obtained by the classical LB deposition technique. The incorporation of anions in the amine films has shown the promise of their being useful as precursors for obtaining ultrathin oxide films. Ultra-thin TiO_2 and ZrO_2 films have been obtained by this route. Thermal evaporation enables patterning of the films through suitable masks. Patterned ultrathin oxide films have been obtained using an electron microscope grid as a mask and this will be extended to smaller structures. The results obtained so far reveal a potential for industrial application. The work is being extended to a wide range of anions.

◆ Amorphous Si-C Alloys

The tetrahedrally coordinated and hydrogenated amorphous Si-C alloys offer thin film semiconductors with widely-ranging band gaps (from 1.7 to 2.8 eV), merely by variation their compositions. This quality makes these alloys attractive for use in the manufacture of optoelectronic devices. However, the mid-gap defect density in these materials increases with an increase in the carbon content which adversely affects the electronic quality of the alloy film. These defects originate from dangling bonds left on Si by migration of H to C atoms, and consequently enhance the disorder in the alloy. Our investigations on Raman and optical properties of the fluorine-incorporated Si-C alloys, prepared by RF plasma CVD, have shown that the fluorine not only helps in tying the dangling bonds, but, interestingly, suppresses the disorder effects of the carbon in these alloys. This advantageously improves the electronic quality of the material by an order of magnitude. Similar enhancements in structural and optoelectronic properties have been observed in fluorinated a-Si:H alloy films.

Clean amorphous silicon-carbon alloy films grown by a simpler technique, known as hot wire-assisted chemical vapour deposition under "starving" hot-wire conditions, have been shown to have the characteristic properties of stoichiometric (carbon-free) a-SiC material. Such synthesis is seldom possible by other techniques.

◆ Solid-State NMR

Development of multi-nuclear and multi-dimensional solid-state NMR techniques and their application to the elucidation of the structure and dynamical properties of polymers, zeolites and other solid-state materials was pursued. The swelling and dissolution phenomena in polymeric systems have been investigated by *in situ* ^1H NMR. A suitable model was developed to explain the appearance of the swelling and dissolution plateau in NMR data of a rubbery material undergoing Fickian diffusion. The role of a hydronamic field under stagnant and stirring conditions has also been studied.

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A heteronuclear ^1H - ^{13}C wide-line separation technique has been developed to separate NMR response from polymer and water components, and hydration details in swollen starch-grafted-hydrolyzed poly(acrylonitrile) and LCST polymer poly(acrylamide) have been revealed. Using the MAS NMR technique, both homonuclear (^1H - ^1H) and heteronuclear (^1H - ^{13}C) two-dimensional nuclear Overhauser enhancement techniques were shown to be powerful tools to study chemical exchange and cross-relaxation phenomena in water-swollen polymers.

A combination of the rate data for water-centred 17O relaxation and cross-relaxation were shown to lead to the location of water in the polymer adjacent to ^{13}C site that was probed. Novel techniques using ^{29}Si were shown to establish the lattice connections among the tetrahedral T sites in high-silica zeolites. The utility of ^{51}V NMR to identify vanadium environments in vanado silicates was demonstrated. A solid-state 2-D NMR technique was developed to separate quadrupolar and paramagnetic dipolar interactions in layered oxides such as $\text{La}_2\text{Li}_{0.5}\text{Ni}_{0.5}\text{O}_4$. Programs for solid-state NMR spectral simulation were developed for a variety of situations.

FUNDAMENTAL RESEARCH

FUNDAMENTAL RESEARCH

Chemical Engineering

◆ Artificial Neural Networks

Artificial neural networks (ANNs) are mathematical models which mimic the human brain's phenomenal abilities such as learning and generalization. ANNs comprise interconnected layers of elements called neurons which individually carry out simple mathematical functions. Like the human brain, ANNs learn and generalize the hidden information contained in the examples that are shown to them. ANNs have been found very useful in pattern recognition and pattern classification, tasks which even modern-day supercomputers find difficult to handle. ANNs are being widely employed in physical, chemical, and engineering sciences. The Reaction Engineering Group of NCL has successfully employed the ANNs to solve complex process identification modelling, and nonlinear control problems in chemical engineering. ANNs have also been used to model systems in molecular biology. The highlights of this research are given below.

◆ Robust nonlinear control

A new method for robust nonlinear control of single-input single-output systems has been proposed. The control law utilizes the universal approximation characteristic of neural networks augmented with the ability of adaptation. The presence of neural networks obviates the need for a mechanistic model for control law computations and the difficulties associated with model-based approaches become irrelevant. The new control law called N-RNCL incorporates the ability to adapt itself through an adjustment of bias neurons and ensures offset-free performance in the presence of load and unmeasured disturbances. The N-RNCL has been implemented successfully on two chemical engineering systems, namely, strong-acid strong base pH control and nonlinear heat exchanger.

◆ Identification of MSF desalination plant

Fully connected, multilayer feed-forward artificial neural networks trained using the error-back-propagation algorithm have been employed to identify the nonlinear multivariable multistage flash (MSF) desalination plant. Both, multiple input-single output (MISO) and multiple input-multiple output (MIMO) networks have been used for the purpose of identification. Correlation coefficient values greater than 0.99 were

obtained, suggesting that the neural network can serve as a good alternative to model MSF desalination plant.

◆ Estimating diffusion coefficients of a micellar system

A three-layer feed-forward ANN, using the error-back-propagation algorithm, has been developed to compute the diffusion coefficient of the sodium dodecyl sulphate (SDS) micellar system over a wide range of operating parameters such as temperature and concentrations of SDS and NaCl. The network model validates the experimentally observed qualitative and quantitative trends. The optimal model parameters in terms of network weights have been estimated and can be used for computing diffusion coefficients over wide-ranging experimental conditions.

◆ ANN applications to problems in molecular biology

ANNs form an important tool to deal with systems which cannot be described by phenomenological modelling approaches. Biological systems with their inherent complexity fall into this category. For the two biology problems that have been addressed, ANNs have provided very valuable insights. In one case, an ANN model has been built to identify terminator sequences. A new coding strategy for coding nucleic acids has been suggested and is based on the electron ion interaction potential (EIIP) associated with each nucleotide base. A caliper randomization approach has also been developed to arrive at the consensus of the sequence responsible for a particular biological function.

The second problem concerns gene expression. An ANN model has been built to capture the transcription control signals contained in the upstream region of a protein coding gene. The ANN model predicts the rate of mRNA synthesis based on the pattern present in the upstream region. The specific system under study was the β -globin gene. This gene is of particular importance since the mutations associated with it are known to cause genetic disorders like thalassaemia and sickle cell anaemia. These are primarily due to the decrease in the synthesis of the globin gene chains caused by mutations. The ANN model developed can be used, (i) to probe conditions which may be genotypically defective but phenotypically normal and, (ii) in designing mutation experiments.

The work on ANN applications to chemical engineering problems has been funded by the Department of Science and Technology (DST), Govt. of India, New Delhi.

Simulation and Modelling

◆ **Modelling of Multiphase Catalytic Reactions for Hydrogenation of Organic Compounds**

Multiphase reactors are important for a variety of hydrogenation processes in fine chemicals, pharmaceuticals and petroleum processing. Extensive work on understanding the reaction engineering aspects of these processes has been carried out as part of a joint project between NCL and Unite Mixte, CNRS-Rhone Poulenc, Lyon, France, under the sponsorship of the Commission of European Communities. This research, in particular, involves the kinetics of catalytic hydrogenation of 2,4 dinitrotoluene, an important step in the manufacture of toluene diisocyanate (TDI). Mathematical models have been developed for predicting the performance of catalytic hydrogenation reactors under non-isothermal conditions and the results compared with experimental data on hydrogenation of 2,4 dinitrotoluene and ketones (acetophenone). This work is very important for understanding the design and scale-up of slurry and trickle bed reactors specially for complex catalytic reactions with large heat effects.

A catalyst system has also been developed for hydrogenation of dicarboxylic acids to value added products. This project has been sponsored by DuPont, USA with the aim of understanding the catalytic and reaction engineering aspects.

◆ **Hydrodynamic Modelling of Multiphase Reactors**

A general purpose computer code with an easy to operate user interface has been developed for simulating turbulent flows in multiphase reactors. This code incorporates the recent advances in numerical algorithms as well as physics of multiphase flows. The code called SPARE, can be used as an effective and economical design tool to screen various design options for a given process.

◆ **Dynamics of Gas-liquid Flows**

Multiphase flows are inherently unsteady. It is indeed necessary to characterize and model these unsteady, large scale flow structures in industrial multiphase equipment. A computational snapshot approach has been developed to capture these flow structures in bubble column reactors. Systematic experimental investigations have been initiated using the recent advances in analyzing chaotic time series. The results are expected to lead to a general purpose diagnostic toolkit based on pressure probes and computational models to characterize the behaviour of industrial multiphase equipment.

◆ **Industrial Flow Modelling**

The Fluid Dynamics and Transport Phenomena Group at the Chemical Engineering Division is engaged in providing flow modelling solutions to industry. The group also organizes training courses on computational fluid dynamics for engineers from industry. It has also established tie-ups with Flow Solve Ltd., U.D. and Fluent Inc., USA, through which an access to commercial SFD codes PHOENICS and FLUENT, has been obtained. Industrial problems that are currently being studied are simulation of vortex flow meters, regeneration of fixed bed reactors, demisting of vehicles, etc.

Theoretical Chemistry

◆ **Correlated Molecular Properties**

A new stationary development to calculate molecular properties using the biorthogonal functional has been undertaken. This has the advantage of taking care of some earlier difficulties encountered in implementing the stationary approach for non-linear properties. Numerical results using this have shown that this approach is reliable. Further work is expected to make the approach suitable for larger systems.

◆ **Chemical Hardness for Open Shell States**

Significant advances have been made in the quantitative elucidation of complex ideas in the area of hardness-softness. Using highly correlated coupled cluster methods, the principle of maximum hardness has been shown to be valid for asymmetric distortions in molecules. It has also been shown that the principle cannot be used for symmetric distortions. General equations relating to the change of chemical hardness in the course of binding were developed, and these allow hardness to be used as an index in a reaction profile. A Δ SCF procedure has been used to obtain fully theoretical values of the open shell chemical species.

◆ **Electron-molecule Interaction for Low-energy Scattering**

The corrected static-exchange (CSE) potential of molecules for low energy electron-molecule scattering has been studied in detail using accurate techniques. Dominant-exchange effects in the CSE potential, observed in the studies, clearly showed the treatment of the exchange effects in the previous studies to be incorrect. It has also been shown that the hole-hole and particle-particle block of density matrix contribute to the CSE and that the hole-particle block contributes to the static part of CSE.

◆ **Solvation Models of Hypermodified Components of tRNAs**

Transfer ribonucleic acids (tRNAs) play a key role in protein biosynthesis. These molecules are also involved in several other biochemical reactions, such as chlorophyll biosynthesis and retrovirus replication. A unique feature of tRNA molecules is the presence of a large variety of modified components at several locations throughout the molecule. Extensive chemical modifications of nucleic acid bases occur naturally at crucial positions in the anticodon loop of tRNA. The hydrophobic- and the hydrophilic- substituted adenines occur at anticodon 3 - adjacent in tRNAs. The first systematic theoretical investigation of these hypermodified components has revealed that these substituents may probe the molecular environment in the vicinity of the anticodon. The differences in the conformational adaptability of the various substituents as well as the significance of the partial and the complete modification for these has been brought out. It has been shown that the changes in the protonation status as well as the site of protonation can induce conformational transitions. The results obtained indicate that N(7) protonation may cause ambiguity in the defining of the reading frame for codon-anticodon interactions.

◆ **Hydrophobic Interactions in Organic Reactions and Nucleic Acids**

Some recent experimental observations regarding the dramatic rate enhancement in some organic reactions were analyzed in terms of hydrophobic interactions. A suitable explanation was proposed in terms of volume of activation and internal pressure of the reacting system for the observed rate enhancement in Diels-Alder reactions when carried out in pure solvents and in their solutions with ionic solutes. These two factors can easily account for the fact that when such solvents are used ultrahigh pressure conditions, normally required for carrying out the reactions, can be eliminated.

Similarly, thermal stability of nucleic acids, like DNA, has been interpreted in terms of hydrophobicity. Variations in thermal stability of the DNA duplex with ionic concentrations have been explained using the condensation and screening effects built into the correlations developed in the study. Thermal stabilities of duplexes with different base-pairings have been found to exhibit a linear relation to the hydrostatic pressure.

◆ **Understanding Protein Structure and Function**

Ion-bonding is important for both the function and the stability of proteins. The location of ion-binding sites in various proteins, the geometry of binding, the sequence

involved and the effect of the binding on the side-chain conformations have been investigated using the known protein structures in the data base. The results can be used for the *de novo* design or the modification of proteins, so as to impart ion-binding properties to them.

◆ **X-ray Diffraction Study of Organic Structures**

*X-ray diffraction studies of tetrabutyl ammonium hydrogen bisbenzoate (bioxanion), a useful nucleophile, soluble in organic solvents and capable of catalyzing the silylketene acetal that initiates group transfer polymerization of alkylacrylate monomers, as well as a carbonate interchange reaction of aromatic biphenols with diphenyl, were carried out. These studies have thrown light on the nature of hydrogen bonding in the molecules and have revealed the packing of cations and anions in the crystal lattice.

*A new pregnane steroid, villosterol, isolated from the aerial parts of *Turraea villosa*, was characterized. This is the first report on the isolate of "cis" A,B rings fused pregnane steroid from a plant.

*The structure of binuclear organotin (IV) complex of Malonobis (salicylidenehydrazide), a compound active against *M. tuberculosis*, has been solved.

*X-ray structures of isostructural molybdenum (II) π -allyl complexes provided definite evidence to conclude that, other factors remaining equal, a CH/ π interaction dictates conformational preferences in these molecules.

*Studies on several mixed metal, mixed chalcogenide carbonyl clusters have been carried out. Analysis of $i(\text{CO})_6\text{Fe}_2 \text{ U-Se-CC(H)C(CH}_3\text{) SeCSE}$ revealed an unusual cyclization of the diacetylene with incorporation of an Se atom to form a cyclic C₄Se ligand attached to the $\text{Fe}_2(\text{CO})_6\text{Se}_2$ core.

*X-ray structure of an intermediate in the synthetic scheme for taxol showed that the position of the hydroxy group is crucial in determining further synthesis strategies and that it points away from the protecting group and is below the plane of the benzene ring.

APPENDICES

LIST OF PUBLICATIONS

(** Papers just communicated and those still in press have not been included. Only the papers for which the volume, issue and page numbers are known have been included.

** Authors who find that some of their papers are not included in their particular area should look for them in the areas in which the co-authors of the papers work.

** Papers presented at conferences and contributed to books and monographs are also included.)

BIOCHEMICAL SCIENCES

1993-94

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PATENTS GRANTED

1993-94

Patents Granted in India

No.	Patent No.	Date of Grant	Title	Inventors
1.	170448	25/6/93	Synthesis of alpha(RS) -cyano-3-phenoxy benzyl(+)cis-(2,2,-dichlorovinyl) cyclopropane carboxylate, a highly potent insecticide belonging to the synthetic pyrethroids group.	R.B.Mitra, G.H.Kulkarni P.N.Khanna B.M.Bhawal A.R.A.S.Deshmukh
2.	171363	26/11/93	Process for the preparation of a catalyst composite material.	P.Ratnasamy S.Sivasanker
3.	171984	11/3/94	An improved process for the preparation of elastomers having random distribution of functional groups from olefinic polymers.	K.S.Balaraman S.Gopichand S.Gundiah R.A.Mashelkar S.H.Vaidya A.J.Varma G.R.Venkita-krishnan
4.	170829	1/10/93	An improved process for the preparation of a high silica zeolite catalyst composite material.	Rajiv Kumar
5.	170837	30/7/93	An improved process for the conversion of natural gas into middle distillates.	P.Ratnasamy S.Sivasanker

6.	170903	1/10/93	A process for the production of kerosene and diesel from FCC naphtha.	C.Gopinathan J.Kuruvilla S.Gopinathan A.M.Hundekar S.K.Pandit I.R.Unny S.S.Deshpande S.A.Pardhy
7.	171794	4/2/94	An improved process for the preparation of high temperature superconductors.	I.S.Mulla A.P.B.Sinha M.R.Chandrachud
8.	169816	22/5/93	An improved process for the preparation of 1,1,1-trichloro-4-methyl-pent-3-ene-2YL diazoacetate.	R.B.Mitra G.H.Kulkarni P.N.Khanna B.M.Bhawal A.R.A.S.Deshmukh
9.	170907	1/10/93	An improved process for the preparation of alkyl carbamates.	R.V.Chaudhari S.P.Gupte A.A.Kelkar D.S.Kolhe
10.	170908	1/10/93	An improved process for the preparation of aryl-N-alkyl carbamates.	G.H.Kulkarni R.H.Naik S.Rajappa
11.		29/3/94	A process for the preparation of trifunctional amino acid derivatives of dicarboxylic acid chlorides.	M.G.Kulkarni A.U.Nadgauda

1994-95

1.	172135	13/04/94	An improved process for the preparation of 4-phenyl-5-dichloroacetamido-1,3-dioxane.	B.G.Hazra V.S.Pore S.P.Maybhatte M.V.Natekar
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2.	172333	27/05/94	Process for the preparation of a novel crystalline aluminosilicate.	A.N.Kotasthane A.J.Chandwadkar P.Ratnasamy
3.	172361	27/05/94	An improved naphtha reforming process.	P.Ratnasamy S.Sivasanker
4.	172287	20/05/94	Improved process for carbonylation of alcohols to carboxylic acid.	A.A.Kelkar R.V.Chaudhari
5.	172653	29/07/94	A process for the preparation of high silica large port modernite.	A.J.Chandwadkar P.Ratnasamy
6.	172604	22/07/94	An improved process for preparation of 1-substituted amino-1-substituted thio-2-nitro alkenes.	A.R.A.S.Deshmukh B.M.Bhawal V.P.Shiralkar S.Rajappa

Patents Granted in Other Countries

1993-94

No.	Patent No.	Date of Grant	Title	Inventors
1.	89304707.6 (Europe)	27/10/93	An improved process for the preparation of 1-substituted amino-1-substituted thio-2-nitro alkenes.	A.R.A.S.Deshmukh B.M.Bhawal V.P.Shiralkar S.Rajappa
2.	5262045 (USA)	16/11/93	A process for reforming of pyrolysis naphtha.	K.S.Prasada Rao S.Sivasanker P.Ratnasamy
3.	5288838 (USA)	22/02/94	A process for the preparation of novel bioxyanion catalysts useful	S.Sivaram J.C.Sehra V.S.Iyer

4.	5266702 (USA)	30/11/93	A process for the preparation of polycarbonates and a process for the preparation of polycarbonates using the said catalysts.	D.Baskaran P.K.Dhal S.P.Kashikar R.S.Khisti B.M.Shinde S.Sivaram
5.	5219813 (USA)	15/06/93	A process for the preparation of novel crystalline molecular sieves.	R.Kumar K.R.Reddy P.Ratnasamy
6.	5266659 (USA)	30/11/93	An improved process for the preparation of high molecular weight poly (arylcarbonate)s.	S.Sivaram J.C.Sehra V.S.Iyer K.Ravindranath
7.	5274107 (USA)	28/12/93	An improved process for the synthesis of D(+) biotin.	S.P.Chavan R.B.Tejuani T.Ravindranathan

1994-95

1.	2025449 (Canada)	14/03/95	A process for reforming of pyrolysis naphtha.	K.S.Prasada Rao S.Sivasanker P.Ratnasamy
2.	22474 (Mexico)	04/08/94	A process for reforming of pyrolysis naphtha.	K.S.Prasada Rao S.Sivasanker P.Ratnasamy
3.	5306854 (USA)	26/04/94	A two step process for the production of liquid hydrocarbons from natural gas.	V.R.Choudhary S.D.Sansare A.M.Rajput

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|----|------------------|----------|--|---|
| 4. | 5336825
(USA) | 09/08/94 | An improved integrated two step process for for the conversion of methane to liquid hydrocarbons of gasoline range. | V.R.Choudhary
S.D.Sansare
S.T.Choudhari |
| 5. | 5338488
(USA) | 16/08/94 | An improved process for the production of synthesis gas by oxidative conversion of methane using composite catalyst containing transitional and alkaline earth metal oxides. | V.R.Choudhary
A.M.Rajput
S.D.Sansare
B.Prabhakar
A.S.Mamman |
| 6. | 5368835
(USA) | 29/11/94 | Process for the production of synthesis gas by oxidative conversion of methane methane (or natural gas) using composite catalyst. | V.R.Choudhary
V.H.Rane
A.M.Rajput |
| 7. | 5340908
(USA) | 23/08/94 | An improved process for the preparation of aromatic polyester(s). | B.B.Idage
S.B.Chavan
S.S.Mahajan
S.Sivaram |
| 8. | 5401343
(USA) | 28/03/95 | An improved process for the preparation of caprolactam from cyclohexanone oxime using micellar solutions, macro-emulsion and micro-emulsion systems. | B.K.Jha
A.S.Chhatre
B.D.Kulkarni |

HONOURS AND AWARDS

1993-94

01. Dr.R.A.Mashelkar, Director, NCL, was awarded an honorary doctorate degree in science by the University of Salford, U.K., in recognition of his outstanding contributions to science.
02. He was elected fellow of Third World Academy of Sciences, Trieste, Italy, for his creative contributions to fundamental research in polymer science.
03. He received the G.D.Birla Award for Scientific Research for 1993, in recognition of his outstanding contributions in polymer science and engineering.
04. Dr.P.Ratnasamy, Deputy Director, NCL, and Head, Catalysis Division, was selected for the Vishwakarma Medal by the Indian National Science Academy, for his achievements leading to starting of a new industry and to improvements in processes.
05. Dr.P.Ganguly, Head, Physical Chemistry Division, was elected fellow of the Indian Academy of Sciences, Bangalore, in recognition of his major contributions to the spheres of solid state chemistry and surface chemistry.
06. Dr.K.N.Ganesh, Head, Organic Chemistry Synthesis Division, was selected by the Homi Bhabha Fellowship Council, Bombay, for the Homi Bhabha Fellowship for a period of two years for successfully employing the NMR technique to study molecular structure and dynamics.
07. Dr.S.P.Chavan and Dr.Murali Sastry were selected for the CSIR Young Scientist Award for 1993 in the Chemical Sciences and Physical Sciences, respectively. Dr.Chavan has made innovative contributions in the development of new methodologies, elucidation of reaction mechanisms and the synthesis of natural and non-natural products of biological importance. Dr.Sastry's contributions have been in quantitative surface science using electron spectroscopy. His unique work on Langmuir-Blodgett films, a frontline materials science area, has won international recognition.
08. Dr.S.Sivasanker (Catalysis Division), Dr.(Ms.)R.S.Nadgauda (Plant Tissue Culture Division), and Dr.R.V.Choudhari (Chemical Engineering Division) were chosen for the 1991 Award in Chemical Sciences and Technology, Smt. Chandaben Mohanbhai Patel industrial Research Award for Women Scientists, and 1989 Award in Chemical Sciences and Technology, respectively, by the Vasvik Foundation.

Dr.Sivasanker has done significant work on the development of zeolite catalysts for the petroleum refining and petrochemical industries in the past two decades. Many catalysts developed by him have been commercialized. Dr.Nadgauda's work on the early flowering of bamboo has won worldwide recognition. Dr.Choudhari has been involved in the development of several innovative processes based on homogeneous catalysts,

such as butenediol, butanediol, propionic acid, acetic acid via methanol carbonylation, and non-phosgene, non-MIC route for carbamate pesticides.

09. Dr.Ganesh Pandey, Scientist, Div. of Org. Chem.: Synthesis, was elected Fellow of The National Academy of Sciences, India, based at Allahabad.
10. Mr.R.R.Hirwani, Head, Project Planning and Development Unit, has been selected for the Hubert H. Humphrey Fellowship Program by the United States Information Agency, in recognition of his initiatives in marketing NCL's knowledge base and other R&D management activities. This is the first time that a scientist from India's S&T establishments has been selected for this fellowship. He was the only one to be selected from any country in 1994 in the area of *Technology Policy and Management*.
11. Dr.P.Ratnasamy, Deputy Director, NCL, was invited to become a member of the Editorial Board of *Applied Catalysis* for a period of three years.

1994-95

01. Dr.R.A.Mashelkar, Director, NCL, was invited to deliver the P.V.Danckwerts Memorial Lecture at Glaciers Hall, London, in June 1994. He talked on *Seamless Chemical Engineering Science: The Emerging Paradigm*. The lecture has been published in the first issue of the 50th volume of *Chemical Engineering Science*.
02. Dr.Mashelkar was invited to attend the symposium *Frontiers of Chemical Engineering Science*, held at the Massachusetts Institute of Technology (MIT), USA, in which he was given the honour of delivering the closing speech about his views and vision about the future of chemical engineering science.
03. Dr.Mashelkar received the *G.D.Birla Award* for his outstanding contributions in polymer science and engineering.
04. Dr.Mashelkar delivered the prestigious *Lala Karamchand Thapar Centenary Memorial Lecture* in New Delhi, on *India's Emergence as a Global R&D Platform: New Challenges and Opportunities*, which was presided over by the Hon'ble Finance Minister, Dr.Manmohan Singh.
05. Dr.Mashelkar was awarded the Raj Kristo Dutt Memorial Award by the Indian Science Congress Association, Calcutta. He delivered the Memorial Lecture at the Annual Session of the Association.
06. Dr.Mashelkar was elected Vice President of the Indian Academy of Sciences, Bangalore.
07. The Catalysis Society of India chose Dr.P.Ratnasamy, Deputy Director, NCL, for the *Eminent Scientist Award*, and Dr.Rajiv Kumar, Scientist, Catalysis Division, for the *Sistla Kameshwari Siddhanti's Young Scientist Award*.

08. Dr.S.Sivaram, Head, Polymer Chemistry Division, and Dr.T.Ravindranathan, Head, Division of Organic Chemistry: Technology were chosen for the K.G.Naik Gold Medal.
09. Dr.M.C.Srinivasan, Head, Division of Biochemical Sciences, was elected Fellow of the Indian National Science Academy, New Delhi. He was also elected Life Member of the Maharashtra Association for the Cultivation of Science.
10. Dr.S.Sivasanker, Scientist, Catalysis Division, and Dr.V.S.Patwardhan, Scientist, Chemical Engineering Division, were elected Fellows by the Indian Academy of Sciences, Bangalore.
11. The University of Salford, U.K., honoured Dr.S.Devotta, Head, Engineering Services, by appointing him Visiting Fellow in its Overseas Education Development Office for the year 1994-95.
12. Dr.R.N.Sharma, Scientist, Division of Organic Chemistry: Synthesis, was elected Fellow of the National Academy of Agricultural Sciences, New Delhi.
13. Dr.V.V.Ranade, Scientist, Chemical Engineering Division, was chosen as an Associate by the Indian Academy of Sciences, Bangalore.
14. Mr.P.M.Suryawanshi, Scientist, Division of Organic Chemistry: Synthesis, was selected by Bruker India Scientific Pvt. Ltd. for the *Bruker Award for Instrument Engineers*.
15. Dr.M.V.Deshpande, Scientist, Biochemical Sciences Division, was conferred the D.Sc. degree of the University of Pune.
16. Mr.H.P.Chakraborty, Technical Officer, Glass Blowing Section, received the third prize in the All India Glass Blowing Competition.

IMPORTANT EVENTS

NCL FOUNDATION DAY-94 (3 January 1994)

Dr.R.A.Mashelkar, Director, NCL, introduced the function and said that the NCL Research Foundation had been set up three years earlier, with the help of funds made generously available by the industry, to create an environment in which appreciation and recognition of excellence in performance was shown by conferring awards on deserving members of staff. These awards were for excellence in science, and applied research leading to commercialization; highest cash flow; new initiatives in the R&D support system; and for unusual merit in individual staff members and groups.

Dr.Mashelkar introduced Prof. Peter Day, the Foundation Day Speaker for 1994, as the Director of the Royal Institution of Great Britain, the oldest continuously-functioning research laboratory in the world, which was founded in the Age of Enlightenment following the French and American revolutions. It is associated with such legendary names as Humphrey Davy, Michael Faraday, John Tyndall and James Dewar.

Prof. Day then delivered the NCL Foundation Day Lecture on *An Organization for Science: The Royal Institution of Great Britain*. The theme of Prof. Day's talk was the seminal role played by the development of organizations like the Royal Institution in the progress of science.

After the lecture Prof.Day gave away the Foundation Day prizes. Dr.(Ms.)Aditi Pant compered the awards ceremony. A list of the award winners is given below.

ANNUAL AWARDS - 1993

Name of Award	Award	Award Winner
Shirinbai & Manekji Neterwala Scientist of the Year Award	Rs.20,000/- & Citation	Dr.P.Ratnasamy (Catalysis Division)

For original contributions in the synthesis of novel molecular sieves and for opening up new frontiers in zeolite catalysis.

ICICI Technology of the Year Award

Rs.30,000/- & Citation

Formaldehyde Catalyst (Catalysis Division)
Dr.P.Ratnasamy
Dr.A.V.Ramaswamy
Dr.V.P.Shiralkar
Dr.A.N.Kotasthane
Dr.P.A.Awasarkar
Mr.A.Y.Sonsale
Dr.S. Sivasanker
Mr.V.R.Chumbale
Dr.M.J.Eapen
Dr.(Ms.)Veda Ramaswamy
Ms.S.V.Awate
Ms.N.E.Jacob
Dr.H.S.Soni
Mr.P.N.Joshi
Ms.A.A.Belhekar
Mr.K.Ramakrishnan

(For successful development and commercialization of a superior metal-oxide catalyst for formaldehyde production.)

Highest Industrial Earning Award

Rotating Shield & Trophy

Polymer Chemistry Division

For highest per capita (net) industrial earning during 1992-93.

International Patent Award (for US Patents granted during 1992 and 1993)

Silver Medallion

Mr.S.Malik
Dr.S.N.Shintre
Dr.R.A.Mashelkar
[Polymeric Drag Reducer]

Mr.D.Baskaran
Dr.P.K.Dhal
Dr.S.P.Kashikar
Dr.(Ms.)R.S.Khisti
Dr.B.M.Shinde
Dr.S.Sivaram
[Poly(acrylonitrile) Oligomers]

Dr.V.R.Choudhary
Dr.M.Y.Pandit
Dr.S.T.Choudhari
[Oxidative Coupling
of Methane to
Ethylene]

Dr.S.Sivasanker
Dr.K.S.Prasada Rao
Dr.K.M.Reddy
Dr.P.Ratnasamy
[New Catalyst Composite
Material]

Dr.Rajiv Kumar
Mr.K.R.Reddy
Dr.P.Ratnasamy
[Novel Molecular Sieves]

Dr.S.Sivaram
Dr.J.C.Sehra
Dr.K.Ravindranath
Mr.V.S.Iyer
[HMW Poly (aryl
carbonates)]

Mr.R.R.Hirwani
Mr.P.Venugopal
Mr.G.Prabhakaran

Award for
New Initiatives
Taken by the R&D Support
System

Rs.20,000/-
& Citation

(For expanding the scope of consultancy and technical services and generating substantial external cash flow from information, knowledge based and database services.)

Individual Merit
Award

Rs.1,000 (each)
& Certificate of
Merit

Mr.D.B.Pradhan
Mr.Y.B.Lotekar
(Workshop)

(Designing, fabricating and successfully commissioning a laboratory-scale fermenter resulting in substantial saving.)

Given below are the texts of the citations that accompanied the NCL Foundation Day Awards for 1993.

Shirinbai and Maneckji Neterwala Scientist of the Year Award

Dr.P.Ratnasamy has done pioneering work on the synthesis of novel molecular sieves. Majority of the novel ferrisilicate molecular sieves known internationally today have been synthesized by Dr.Ratnasamy. His synthesis of molecular sieves containing some specific transition elements in lattice positions has widened the scope of zeolite catalysis to include oxidation reactions, thus extending the frontiers of zeolite catalysis. He has combined high science and high technology in a remarkable way to place India among the leaders in the area of zeolite catalysis.

ICICI Technology of the Year Award

(Given to Dr.P.Ratnasamy jointly with Catalysis Division team members)

This award is given for the development of technology of an improved iron-molybdenum catalyst for formaldehyde production by methanol oxidation. The NCL catalyst, manufactured by M/s. International Catalyst Ltd., is being used in a commercial plant by Cibatul since January 1992. This catalyst is superior to the state-of-the-art catalysts in terms of activity and stability and has already brought significant economic benefits to Cibatul. With the commercialization of this catalyst technology, NCL has joined the company of three multinationals, who possessed this closely held technology.

R&D Support System - New Initiatives Award

(Given to Mr.R.R.Hirwani, jointly with Mr.P.Venugopal and Mr.G.Prabhakaran)

For creating awareness about the potential of information and knowledge based services amongst scientists and conceptualizing new schemes to market NCL's knowledge and skill base. This has expanded the scope of consultancy and technical services resulting in generation of substantial external cash flow from information, knowledge and database services.

Certificate of Merit

(Awarded to Mr.Y.B.Lotekar, jointly with Mr.D.B.Pradhan)

For designing, fabricating and successfully commissioning a laboratory scale fermenter system. The workmanship of this set-up is equivalent to the state-of-the-art imported fermenters. The fabrication of this fermenter in-house has resulted in considerable saving.

NCL FOUNDATION DAY-95 (3 January 1995)

Prof.J.F.Davidson, FRS, University of Cambridge, UK, delivered the Foundation Day Lecture. Dr.R.A.Mashelkar, Director, NCL, and Chairman of the NCL Research Foundation, welcomed the guests.

Dr.Mashelkar announced two new schemes to be undertaken by the NCL Research Foundation. In the first, the Foundation would be paying the fees of children of lower income group staff members until they complete the school and also provide them with uniforms, text books and note-books. Also, the McBain Memorial Lecture in Chemical Sciences and the Venkataraman Memorial Lecture in Technology have been introduced to honour two of NCL's former directors, who are no longer amongst us.

In the Foundation Day Lecture, Prof.Davidson, by citing his own research experience, showed how best research arises in an accidental, unplanned way. Putting the entire question of research planning in perspective he said that too rigid an adherence to plans leads to mediocrity and stifles creativity. He showed how research and teaching are intimately linked and are interdependent.

Prof. Davidson, a pioneer in the area of fluidization, briefly traced the history of the early work in the area, in particular the research on the existence of bubbles, their rise velocity, and its dependence on the diameters of the bubbles as well as the fluidized bed.

After the lecture Prof.Davidson gave away awards for the year 1994 to individuals and groups of NCL for new initiatives taken and for offbeat and original contributions. A list of the award winners is given below.

ANNUAL AWARDS - 1994

Name of Award	Award	Winner/s
Shirinbai & Manekji Neterwala Scientist of the Year Award	Rs.20,000/- & citation	Dr.S.Sivaram (Poly. Chem. Division)
<i>Novel and creative contributions to polymer synthesis.)</i>		
Highest Industrial Earning Award	Rotating Shield & Trophy	Catalysis Division

International Patent Award (for US patents granted during 1993 and 1994)

Silver Medallion

Mr.D.Baskaran
Dr.P.K.Dhal
Dr.S.P.Kashikar
Dr.(Ms.)R.S.Khisti
B.M.Shinde
Dr.S.Sivaram

New reactive anionic initiators]

Dr.S.P.Chavan
Dr.R.B.Tejuwani
Dr.T.Ravindra-nathan

Synthesis of D(+)-Biotin)

Dr.V.S.Iyer
Dr.J.C.Sehra
Dr.S.Sivaram

Novel bioxyanion catalysts]

Dr.V.R.Choudhary
Dr.S.D.Sansare
Mr.A.M.Rajput

Liquid hydrocarbons from natural gas]

Dr.B.B.Idage
Dr.N.N.Chavan
Dr.S.S.Mahajan
Dr.S.Sivaram

Process for aromatic polyesters]

Dr.V.R.Choudhary
Mr.A.M.Rajput
Dr.S.D.Sansare
Dr.B.Prabhakar
Mr.A.S.Mamman

Synthesis gas oxidative conversion of methane]

Individual Merit Award

Rs.1,000/- & Certificate of Merit

Mr.David Soloman

(For designing and fabricating a sophisticated high pressure CSTR of international class.)

Award for the Best
Support Service Group
of the Year

Shield

Library,
SMIS &
NICHEM

(For best services rendered to the NCL scientific community, based on an opinion poll.)

Special commendation
and award for success-
ful efforts made to
renovate the auditorium
and giving it a modern and pleasing look

Civil & Electrical
Sections

The Citations

Shirinbai and Manekji Neterwala Scientist of the Year Award (Dr.S.Sivaram, Head, Polymer Chemistry Division)

Dr.S.Sivaram has made highly original and creative contributions to polymer synthesis. He has contributed to the generation of novel functional anionic initiators, and through them, the first controlled polymerization of acrylonitrile and alkyl acrylates. He has also developed a solid state post polymerization chemistry for polycarbonates. He has been instrumental in the development of new functional polyolefins. Dr.Sivaram has combined high science and high technology most admirably. It is this combination that has attracted the world's leading multinationals to seek partnerships with his group in frontline areas of technology.

Award for Outstanding Individual Merit (Mr.David Soloman)

For making significant contributions towards development of a sophisticated high pressure continuous stirred tank reactor system, most essential for carrying out scale up work on multiphase catalytic processes. The unit has subsequently been used in major externally funded projects which are currently getting commercialized. The performance of the CSTR system was found to be at par with similar units fabricated abroad. This is the first time such a facility has been developed at NCL and it has resulted in considerable savings to the organization.

Special Award to the Civil and Electrical Groups at NCL

This commendation is for conceptualizing, designing and successfully completing a major renovation of the NCL auditorium in a very short time. This is the first refurbishment of this auditorium since its construction in 1950. The quality of the work carried out is self-evident. This is the result of the imagination, sense of responsibility and hard work of these two groups.

CSIR FOUNDATION DAY 1993 (26 September 1993)

The laboratory was kept open to visitors on 25 September 1993.

Dr.Paul Ratnasamy, Acting Director, introduced the function held in the auditorium on the occasion. He said that the CSIR, after more than half a century of its existence, could be proud of its highly productive efforts to establish a strong base of scientific research and the contributions it has made to improve the lot of the common man in the country. The impact of its efforts on the life of the common man may not be readily discernible, but it has been very significant.

Referring to the troubled future that lay ahead, he said in view of the Government's shift in its industrial and fiscal policies, and the constraints on its support of science, the CSIR's role had undergone a change. The awareness that research, like everything else, has to be utilitarian has penetrated to the core of research planning and management. He concluded by stressing the fact that commercial technologies have to meet three criteria if they have to succeed: competitiveness, stringent quality and cost effectiveness.

The Chief Guest, Dr.V.P.Bhatkar, Director, C-DAC, Pune, delivered the Foundation Day Lecture on *Perspectives in Advanced Computing*. He described the chemical industry as the fastest growing industry in the country, and one that interfaced with all the facets of the manufacturing sector. In the field of computers, India had started with the early versions of the analog computer; the advent of the numerous new technologies in the 80's had led to the replacement of the analog computer by the digital computer. The electronics and computer industries owed a lot to the chemical industry, which provided the means of this transition, namely, the new materials. The chemical industry, in turn, benefitted greatly by the developments in the electronic control instrumentation and computerized control systems.

The following awards/prizes/mementoes were given away by the Chief Guest: a memento from NCL staff to Prof.B.D.Tilak, former Director, NCL, whose 75th birthday fell on the same day; prizes to SC/ST students who had stood first in science subjects in the S.S.C examinations held by the Boards in the state in 1992; prizes to the winners of the Science Quiz contest held in connection with the Foundation Day; and mementoes to members of staff who had retired in 1992, and to those who had completed 30 years of CSIR service that year.

Referring to the appropriateness of felicitating Prof.Tilak, Dr.S.H.Iqbal, Head, Division of Technical Services, drew attention to the fact that Prof.Tilak had been NCL's Director during some critical years, and it was his inspired leadership and missionary zeal that had paved the way to the successes that NCL has had in technology development.

The Chief Guest then gave away the prizes and mementoes. The wards of CSIR staff members selected for excellent performance in a group of three science subjects at the higher secondary examination also received their prizes from the Chief Guest.

CSIR Foundation Day 1994 (26 September 1994)

Dr. Paul Ratnasamy, Acting Director, presided over the function. He spoke about the new market and economic forces to which NCL was exposed, and how the laboratory could adapt itself to them and carry out world-class research leading to competitive technologies and products. He listed three elements that play a vital role in the conduct of research and development, viz. professionalism, capital and management. He said that the new pressures on chemical industries resulting from the globalization measures initiated by Government's new policies, would in turn reflect on the R&D laboratories. NCL should therefore become market-oriented, come out with new products and earn more and more capital. To achieve this, it was essential for it to inculcate a totally new culture made up of new organizational skills and management strategies. It also had to augment its income from sources other than Government agencies, he added.

After his speech, Dr. Ratnasamy officially released the first issue of *In and Around NCL*, which had been started as a new communication channel within the NCL community.

Dr. Ratnasamy then presented mementoes to members of staff who had retired during 1993-94, and to those who had completed 30 years of CSIR service in that year. The wards of CSIR staff selected for excellence in their academic performance in a group of three science subjects then received their prizes from him. The function ended with the presentation of prizes to the winners of the Science Quiz Competition.

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