

Regional Research Laboratory, Jorhat

Annual Report 2003-2004

R&D Activities

Effect of outer membrane permeabilizers on the activity of antibiotics and plant extracts against *Pseudomonas aeruginosa*

The muga silkworm *Antheraea assama* are easily susceptible to bacterial infection called 'Flacherie' caused by *Pseudomonas aeruginosa* manifesting the symptoms in the host such as poor appetite, retarded growth, black body fluid and hanging upside down. Several known outer membrane permeabilizers increased susceptibility of a highly resistant pathogenic strain *Pseudomonas aeruginosa* AC-3 to different antibiotics and their plant extracts. Of all the chemicals tested, EDTA, sodium citrate and sodium hexametaphosphate (HMP) were found to be potent permeabilizers as shown by enhanced lysis of the bacteria in the presence of lysozyme. In the presence of EDTA and Sodium citrate susceptibility of the strain to gentamycin and rifampicin increased markedly. The strain was resistant to vancomycin but became susceptible when grown in the presence of increasing amounts of EDTA and sodium citrate. EDTA was found to be most potent permeabilizer in enhancing the activity of the plant extracts. The strain *P. aeruginosa* AC-3 causes heavy economic loss to farmers by damaging entire broods of muga silkworm. As this silkworm is reared outdoor and cannot be domesticated, disease incidences become more severe and unmanageable. The study was expected to lead to an efficient formulation of disease control by combination of antimicrobial compounds and permeabilizers.

Vermi compost from spent citronella grass

The spent up grass from citronella distillation units are waste materials and are usually burnt away. Efforts were made in the branch laboratory at Arunachal Pradesh to utilize the spent grass for making vermi compost using suitable strain of earthworm. Some 700 kg of vermi compost were produced and actions were initiated for evaluation of the field performance of the vermi compost.

Study, exploration and identification of medicinal and aromatic plant sources

Folklore medicinal plants

Folklore medicinal plants used by Mishing, an aboriginal ethnic group of Assam were studied and 62 plant species used for the treatment of as many as 35 ailments were recorded. Of these *Alternanthera philoxeroides* (anaemia), *Equisetum ramosissimum* (fracture of bone), *Ficus hispida* (diabetes), *Smilax acroata* (heart problem), *Lasia spinosa* (Piles) were found to be more prospective.

Identification of chemotypes

Four chemotypes of *Cinnamomum sulphuratum* were identified on the basis of their chemical constituents viz. linalool, citral, geraniol, cinnamaldehyde and methyl cinnamate.

Study of the oil of *Aquilaria malaccensis*

A comparative study of essential oil of diseased and disease free wood of *Aquilaria malaccensis* was investigated. The highest oil content (0.9%) was recorded in the diseased wood and the lowest (0.49%) was recorded in the disease free wood. A total of 41 compounds were identified and the main constituent was

identified to be the hexadecanoid acid. However, the market sample of agar oil was found to contain dioctyl phthalate as the major constituent.

Chemotypes of food plant for Muga silk worm

Three chemotypes of *Litsea cubeba* were identified which were found to be preferred by Muga silk worm as food plant due to low oil content with sabinene and limonene in leaves.

Protocol for multiplication of elite species

An efficient protocol for large scale multiplication of an elite species of *Pogostemon cablin* was standardized.

Agrotechnology and Floral biology

Agrotechnology of *Plumbago zeylenicum* was standardized and floral biology of *Aristolochia india* was studied.

Study of VAM fungi

Occurrence of VAM fungi in soil sample of agarwood plant was studied. Variation in percentage of root colonisation and number of VAM propagules were observed.

Study of the strains of Trichoderma

Three strains of Trichoderma were found to be antagonistic in-vitro against *Fusarium oxysporum* causing storage rot of ginger. *Trichoderma viride* was also found to control the leaf blight of citronella.

Alternative bedding material for mushroom

Dried turmeric leaves could be suitably utilized as alternative bedding material for cultivation of *Oyster mushroom*. Oak tree twigs were successfully utilized for preparation of mushroom spawn shiitake mushroom.

Study of pulp yield

The pulpable biomass, both unbleached and bleached, yield of Keno was 15t/ha, 53% and 48% and that of Roselle and Sesbina were 15.5t/ha, 49% and 19.5t/ha, 49% and 47% respectively. These non-wood fast growing species could be prominent source of raw materials for pulp and paper industry.

Study of feeding deterency

The growth deterrent effect studies of 6 promising fractions extracted from Eupatorium, *Acacia fernisiena* and *Paedaria foetida* were carried out against two target insect species viz. *Helopeltis theivora* and *Tribolium castereum* 75-80% feeding deterency was recorded in both the species.

Bio-active molecules from plant sources

Under the CSIR Network programme on "Discovery, development and commercialization of bio-active molecules from plant sources" a total of 155 plant extracts obtained from 10 CSIR laboratories were screened against *Helopeltis theivara* and *Sitrotoga cereallela* for three types of biological activities viz. mortality, feeding deterency and IGR effect. Three samples were found to be promising.

A new strain of *Cymbopogon flexuosus*

A new strain of *Cymbopogon flexuosus* designated as BLI-1 having high essential oil (1.0-1.12%) and low citral (16.1%) content was developed. The biomass yield of this strain was about 70-75 t/ha. Since the citral content was quite low, effort was underway to increase the citral content by breeding with a high citral content clone of SD-68 strain of *C. flexuosus*. Till the time F5 progeny was raised. Encouring result in respect of oil (0.86%) and citral content was recorded

(88.90%). Comparative trials of parents as well as hybrid were in progress.

Intoduction of *Kaempferia galanga* in Arunachal Pradesh

The strain of *Kaempferia galanga* was introduced in Arunachal Pradesh, acclimatized and multiplied under the climatic conditions of Itanagar. Work on development of agrotechnology was in progress. High-density crop recorded maximum yield of rhizome per unit area in comparison to low-density crop.

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CHEMICAL SCIENCES

Development of a process for production of Testosterone

Testosterone is an androgenic hormone primarily responsible for normal growth and development of male sex and reproductive organs. Declining testosterone level play a major role in a broad range of age-associated symptoms including declining sexuality, cardiovascular disease, osteoporosis and prostate hypertrophy. Hormone replacement therapy with testosterone is rapidly becoming a standard of anti-aging medicine. An improved process for the production of Testosterone from 4-androst-3, 17-dione was developed at the laboratory scale. The process thus developed was a single-step, stereoselective and cost-effective one.

Selective reductive amination

Bayer's yeast catalysed, under non fermenting conditions, the selective reductive amination of non conjugated 1 and conjugated imines to the corresponding secondary amines 2 in the presence of alkali in high yields.

Catalysis of three-component Biginelli condensation

Cupric chloride dihydrate catalysed the three-component Biginelli condensation between an aldehyde, a beta-ketoester and urea or thiurea under microwave irradiation in the absence of solvent to yield various substituted 3,4-dihydropyrimidin -2(1H)-ones. The reaction was also effective when performed at room temperature in acetonitrile or at 100oC in a solvent free approach, whithout any side reaction as observed by Biginelli and others.

Synthesis of a novel class of dihydropyrazo-loindoles

A novel class of dihydropyrazolo [3',4': 3,4] pyrrolo[1,2-a]indoles 5 and spiro [3H-indole-3,3']2 -1,2,4]-triazoline]-2-ones 11 were synthesised via intra and intermolecular 1, 3-dipolar cycloaddition reactions in good yields. The dihydropyrazoloindoles and spiroindoles possessing suitably pliable heterocycles have tremendous potentiality as a precursor to various mitomycin analogues. In addition to this the presence of C-9 methyl group in 5 also enhanced its importance due to possibility of side chain manipulations at C-10 nucleophilic bio-reductive site.

Bioactive Molecules

Under a CSIR Coordinated Programme on Bioactive Molecules, during the period under report, 26 numbers of plants were extracted as per the data furnished below:

Number of plants extracted 26

Number of plant parts extracted 36

Number of extracts made 89

Number of extracts sent for bioevaluation 89

Number of extracts sent for pesticidal activity 86

Number of extracts sent for storage in CLRI 89

Till the time 31 numbers of extracts were found to be active against different activities like antimalarial, antidementia, antidepressant, antiparkinsonism, antianxiety, etc. after repeated testing in in vitro experiments. These extracts were under in vivo testing. A total of 40 extracts were under repeat in vitro testing for reconfirmation of the activities shown by the extracts. One extract was found to be antifungal in both in vitro and in vivo experiments and fractionation of extract was done as per request received from the bioevaluator and sent for further testing. Another extract was found to be highly active as hepatoprotective agent in both in vitro and in vivo experiments. After fractionation, two fractions were found to be highly active. Actions were on at the highest level to find out the active component of the extract.

Synthesis of Steroidal 16-vinyl nitro compounds - a new application

Functionalization of steroidal molecules at different strategic positions is an important area of research in the development of modified steroids possessing diverse biological properties including antitumor activity. In continuation of work on steroid analogue of nitrocyclopentane carboxylic acid (ANCPA), study was made about the reaction of 16-dehydro- 20-oxopregnanes 1-3 with the CAN-NaNO₂-CH₃CN system which furnished exclusively 16-vinyl nitro derivatives viz. 16-dehydro -16- nitro- 20-oxopregnanes 4-6 in good yield with complete suppression of the acetamidation reaction on the double bond. It was for the first time that the reaction was reported in steroid which further demonstrated the build up of positive charge adjacent to a carbonyl group arising from the attack of NO₂⁺ ion generated from CAN-NaNO₂ system on the double bond leading to the formation of the vinyl nitro compounds (Scheme1).

Effective metal catalyst for synthesis of pyridine derivatives

Indium trichloride was found to be an effective metal catalyst for the synthesis of various tetra-substituted pyridine derivatives via Michael addition of β -dicarbonyl compounds to α , β -unsaturated oximes and subsequent ring closure. This new and efficient method for the synthesis of pyridine derivatives without any by-products will make an useful addition to the existing methodologies in heterocyclic synthesis.

Cycloaddition reactions with non-conjugated and conjugated imines

Electron rich uracil amidine undergo (4+2) cycloaddition reactions with various non conjugated imines to give novel pyrimido[4,5-]pyrimidines and with conjugated imines various pyrido[2,3-d]pyrimidines, after elimination of dimethylamine from the (1:1) cycloadducts and oxidative aromatisation. The reaction gives excellent yields when carried out under microwave irradiation in a solvent-free condition.

A novel method for the coupling of α -cyano-imines into α -diketimines

A novel and efficient method for the coupling of α -cyanoimines into α -diketimines employing samarium diiodide in dry THF was achieved. This novel method involved a simple work up and made useful and important addition in the existing methodologies. The α -diketimines thus obtained could be easily hydrolysed in acidic media to the corresponding 1,2-diketones in excellent yields. Of particular interest was that this procedure also provided a novel and convenient method for the synthesis of 1, 2-dicarbonyl functionality, which invited growing attention.

Use of lead bromide as a catalyst for the cleavage of various oximes, hydrazones, etc.

The use of lead bromide as a new, rapid, efficient and selective catalyst for the cleavage of various oximes, hydrazones and semicarbazones to their corresponding carbonyl compounds at ambient pressure under microwave irradiation in a solvent-free condition was achieved. This new procedure for the regeneration of carbonyl compounds had several advantages such as mild reaction conditions, reduced reaction times, minimisation of side products, environment friendly and above all excellent yields.

Reaction of 16-DPA with P4S10 in refluxing benzene

The reaction of 16-DPA with P4S10 in refluxing benzene afforded a novel adduct 16-DPA-P2S5 instead of the expected thione. The adduct undergoes [4+2] cycloaddition with alkyne dienophiles to afford steroidal (17,16-c) pyrans. The novel adduct behaved as an efficient masked conjugated enone and offered a novel strategy for activation of conjugates towards unreactive dienophile.

A novel method for preparation of indolizines

The indolizines constitute the core structure of many naturally occurring alkaloids. A novel method for the preparation of indolizines was developed by employing a three-component reaction under environmentally benign condition. The microwave mediated three-component reaction of acyl bromide, pyrimidine, pyridine and acetylene was found to be catalyzed by basic alumina to give corresponding indolizines in excellent yields in a one-pot reaction.

A facile and fast preparation of oxetene

The oxetanes are four membered oxygen heterocycles which constitute the core structure of biologically active compounds such as anticancer Taxol and cholesterol lowering agent Lipastatin. A process for facile and fast preparation of oxetene was developed by a microwave mediated [2+2] cycloaddition reaction of steroidal formyl enamides and alkynes in a solventless condition.

Work on cellulose science

With the growing shortage of conventional raw materials, attention has been focussed on identification and generation of alternative fibre crops, which could serve as a source of raw materials. Thus fibres extracted from perennial herbs (plants) have acquired considerable importance as economic source of cellulosic raw materials. It was that quality of fibres varied not only with the species but also with the stage of maturity of the plants during fibre extraction. The age maturity of the plants has also an influence on the mechanical properties of the fibres and hence the quality of production of ropes, papers, etc.

Alpinia allughas (Zingiberaceae) locally known as Talong Tapi in Arunachal Pradesh is a perennial tuberous plant with stout leafy stem, 1-2 m height, grows in swamps, and marshy plots. The stem fibre of the plant is used by the local people as substitute of cane or for making ordinary ropes and rhizomes and seeds have also medicinal use. This study was undertaken to determine the effects of maturity of the plants on various properties of fibres like crystallinity, crystalline sizes, morphology, etc. to determine the optimum harvesting cycle of the plant in order to get acceptable formation of economic growth of fibres for its different industrial uses.

The cellulose samples were extracted from three different age groups a) 4 month old (A), b) 8 month old (B) and c) 12 month old (C). The degree of crystallinity of cellulose were found to be 39%, 44% and 40% for the samples A B and C respectively. The crystalline sizes were calculated for quantitative comparison along (002) and (040) plane directions using *Scherrer* method (Cauchy and Gaussian corrections). The crystalline sizes were found to be 133 Å (corresponding to 17 cellulose unit cells) for A, 160 Å (corresponding to 21 cellulose unit cells) for B and 114 Å (corresponding to 15 cellulose unit cells) for C in (002) plane direction and in (040) plane direction these values were found to be 206 Å (corresponding to 20 cellulose unit cells) for A, 275 Å (corresponding to 27 cellulose unit cells) for b and 164 Å (corresponding to 16 cellulose unit cells) for C using Gaussian correction in both cases. The morphological properties were also

studied. From the results it could be concluded that the maximum harvesting cycle of the plant was 8 months old. Also, the *Alpinia* cellulose fibres were found to be suitable for use as raw material for industrial utilization.

Analytical method development

A promising LCMS method of analysis was established to investigate various groups of polyphenols in Green Tea. Under this LCMS method, altogether 15 different chemical constituents were evaluated, of which major six polyphenols were identified; viz. Epicatechin (MW290), Catechinhydrate (MW290), Gallocatechin (MW306), Epicatechin gallate (MW442), Epigallocatechingallate (MW458) and Epigallocatechin (MW306).

Atom transfer radical polymerization of n-alkyl (Meth)acrylates

The commercially viable production of novel polymers and advanced materials through controlled synthesis has been the goal of industry. Academic areas as well as the industry have recognized the versatility of atom transfer radical polymerization (ATRP) for the development of many commercial polymers. Recent advances on the controlled/living polymerization techniques made it possible to produce well defined complex polymeric structures. The interest in the synthesis of such complex polymers containing (meth)acrylate segments was increased enormously. The large demand for (co)polymers of n-alkyl (meth)acrylates with narrow polydispersity prompted research towards improving the processes for the polymerization of n-alkyl (meth)acrylates. Work was initiated for the development of petroleum additives based on (co) polymerization of n-alkyl (meth)acrylates by controlled synthesis was aimed at generating efficient technologies for commercial exploitation.

Radical copolymerization of behenyl (systematic IUPAC nomenclature : n-docosyl) acrylate and behenyl fumarate was carried out in toluene at 70°C using benzoyl peroxide as initiator. Gel permeation chromatography was used to determine molecular weights (MW) and molecular weight distribution (MWD) of behenyl acrylate-behenyl fumarate (BA-BF) copolymers. ¹H NMR and carbon analysis was used to determine the composition of BA-BF copolymers. Monomer reactivity ratios for high conversion polymerization were calculated by conversion-extended Kelen-Tudos plot. Differential scanning calorimetric (DSC) measurements show sharp melting peaks at about 64°C. Thermal stability studies were performed with thermogravimetric analyzer (TGA). By using these DSC and TGA data in several non-isothermal methods, the activation energies were calculated. X-ray diffraction studies showed the linearity of the copolymers.

The development of controlled/living radical polymerization (CRP) methods has been long standing goal in polymer chemistry. As radical process it is tolerant of functional groups and impurities, and is the leading industrial method to produce specialty polymers. CRP provided access to materials with complex polymeric architectures, some of which had never been synthesized before by conventional free radical polymerization. Atom transfer radical polymerization (ATRP), which is the most promising synthetic method, was applied to the (co)polymerization of various (meth)acrylate monomers having pendant alkyl side chains. ATRP produced well defined polymers with controlled topology and functionality.

Development of specialty polymers for use in petroleum and allied industries

Polymeric additives enhance/impart certain desirable properties of crude oil. Polymers of n-alkyl(meth) acrylates with long pendant alkyl side chains are generally used as pour point depressant/flow improvers for transportation of waxy crude oils. The power of controlled living radical polymerization as synthetic tools in polymer science could be better appreciated by considering the potential advantage when compared to traditional polymerization techniques. R&D activities were undertaken in the laboratory for the development of tailor made multifunctional additives to act as pour point depressants/flow improvers for the transportation of waxy Indian crude oils.

Use of gel-polymer as water shut off reagent

Late in the life of a water driven mechanism, as the level of water in reservoir rises, more and more water begins to enter the well-bore increasing the water oil ratio. At times only traces of oil and gas can be lifted along with large amount of water. It is desirable to reduce permeability of the water channels so as to render the formation more uniformly permeable and alternatively to shut off the water intrusion. Injection of gelled polymer/in-situ gel-formation is used to

control the permeability variation by blocking the water channels in the reservoir. Polymer gel can immobilize some water flooding swept new paths and yield more than traditional water flooding. It is also used to reduce the detrimental effect of reservoir heterogeneity by treating fractures.

Anionic cross-linked polymers of acrylamide is frequently used as water shut off agent in the oil recovery processes. But this polymer gets degraded at high temperature in presence of multivalent brine. Work was continued to develop high temperature stable cross-linked polymer. It was observed that cross-linked polymer of N, N-dimethyl acrylamide-sodium salt of acrylamido methyl propane sulphonate was a thermally stable brine compatible. The novelty of the gel polymer was that the copolymer before gelation was also thermally stable and brine compatible. Efforts were on to replace inorganic cross linker by environmentally acceptable organic cross linker.

Study on fractionation of high waxy distillate

The objective of the work was to (i) optimize the distillation parameters to obtain maximum yield of distillates (ii) to obtain distillates free from asphaltenes which could be easily blended with PWD feedstock processed in Solvent Dewaxing Unit of the refinery. PWD feedstock was distilled by applying SPD Unit using evaporated temperature in the range 190-230°C, pressure 0.001 mbar and flow rate 0.9-1.6 L/h. Altogether 24 fractions were obtained by varying the evaporated temperature and flow rates. It was observed that (i) distillates fractions were free from asphaltenes (<200 ppm) (ii) maximum yield was 76%, distilled at 230°C using 1.2 L/h and 0.001 mbar pressure (iii) on increasing the evaporated temperature, the yield of distillates increased (iv) on increasing the flow rate of the feedstock the yield of distillates decreased.

Scale-up study for making high strength proppants

Oil & Natural Gas producing organizations use high strength proppant, a speciality product for hydraulic fracturing operations in order to enhance oil/gas productivity. The product has been an imported item. Based on the laboratory scale study for making high strength proppants, scale-up study was carried out upto a level of 50 kg/batch. Different parameters were optimized for preparing sintered bauxite proppants. The products conformed to the specifications of American Petroleum Institute (API) for high strength proppants. The work was sponsored by the Oil Industry Development Board, Ministry of Petroleum and Natural Gas, New Delhi.

Upgradation and utilization of ores, minerals and waste materials

(i) Demineralization of Assam coal

Various parameters of the process developed for demineralization of Assam coal were optimized in laboratory scale. Results showed that the ash content of the coal could be reduced by around 50%.

(ii) Liberation and beneficiation of kaolin from Meleih deposit, Meghalaya, Assam

Various routes developed for liberation and upgradation of the clay were already optimized. Preliminary cost economics for setting up a washery based on the process was worked out.

(iii) Characterization and beneficiation of limestone from Umrangshu deposit, North Cachar Hill, Assam

Limestone collected from the deposit had the assay CaO: 52.26, MgO: 1.03, Al₂O₃: 1.04, Fe₂O₃: 1.24 and LOI: 42.97%. Preliminary results revealed that the quality might be improved by selective grinding. the CaO content of the -40+25 mm fraction improved to 53.15% and Al₂O₃ content decreased to 0.76%. The CaO content of the - 6.3 mm fraction dropped to 51.96% and Al₂O₃ content increased to 1.56%. XRD pattern revealed a very very strong (100) and medium strong (70) peaks for calcite in the -40+25 mm - 6.3 mm fractions respectively. The work was funded by Ministry of Coal and Mines, Govt. of India. Synthesis of novel rhodium (I) and ruthenium (II) carbonyl complexes of P-O, P-S and P-Se donors ligands and their applications as catalysts

(a) A few new rhodium (I) carbonyl complexes were synthesized and characterized by IR, NMR and X-ray structure and evaluated as catalysts. Results indicated that one of the ligands i.e. 2-(diphenylphosphino) methyl benzoate formed electron rich rhodium complex through "secondary interaction". Such complex showed high catalytic activity (TON) in carbonylation of methanol to acetic acid and its ester.

(b) Reaction of $[\text{Ru}(\text{CO})_2\text{Cl}_2]_n$ with ligands $\text{Ph}_2\text{P}(\text{CH}_2)_n\text{P}(\text{S})\text{Ph}_2$ [$n=1(\text{a}), 2(\text{b}), 3(\text{c}), 4(\text{d})$] in 1:1 molar ratio produced complex $\text{cis}-[\text{Ru}(\text{CO})_2\text{Cl}_2(\text{P}\sim\text{S})]$ (1a) ($\text{P}\sim\text{S} = (\text{P}, \text{S})$ coordinated) and $\text{cis}-[\text{Ru}(\text{CO})_2\text{Cl}_2(\text{P}\sim\text{S})]$ (1b-d) ($\text{P}\sim\text{S} = -(\text{P})$ coordinated), while 1:2 molar ratio yields complex of the type $\text{cis}-[\text{Ru}(\text{CO})_2\text{Cl}_2(\text{P}\sim\text{S})_2]$ (2a-d). The complex 2a underwent partial decarbonylation reaction in CH_2Cl_2 -hexane solution to give a new chelated complex $[\text{Ru}(\text{CO})\text{Cl}(\text{P}\sim\text{S})_2]\text{Cl}$ (2a'). Abstraction of halide with AgClO_4 from the nonchelated complexes 1b-d and 2a-d afforded corresponding chelated complexes $[\text{Ru}(\text{CO})_2\text{Cl}(\text{P}\sim\text{S})](\text{ClO}_4)$ (3b-d) and $[\text{Ru}(\text{CO})_2(\text{P}\sim\text{S})_2](\text{ClO}_4)_2$ (4a-d). The molecular structure of the complex 2a' was determined by single crystal X-ray diffraction. The ruthenium atom was at the centre of slightly distorted octahedral structure having the two phosphorus atoms of the two chelated P, S coordinated ligands at trans to each other, one CO group and Cl atom completing the coordination sphere. Other complexes were characterized by elemental analysis, IR, ^1H and $^{31}\text{P}\{-\text{H}\}$ NMR spectroscopy.

Synthesis and application of adsorbent and catalyst/reagent based on intercalated metal layered hydroxides, clay supported metal/metal complex

Generation of metal nanoparticles on montmorillonite K 10 and their characterization :

In recent times, supported metal nanoparticles have gained much importance because of their novelty in various applications such as solid acid catalysis, oxidation of hydrocarbons, dehydrogenation of hydrocarbons, etc. The present work carried out for in situ synthesis of Ni-Montmorillonite K10 (Composite I) and Co-Montmorillonite K 10 (Composite II) by polyol reduction of nickel acetate and cobalt acetate impregnated Montmorillonite K 10. The TEM study revealed that the size of metal nanoparticles in Composite I were upto 26 nm and were comparatively smaller than that of the Composite II where maximum particle size of metal limits to 40 nm. The XRD study showed face centered cubic structure in both of nickel and cobalt nanoparticles in Composite I and II. These composites were also characterized by thermal and IR studies. Such nanometal-clay composites may find application as bifunctional catalysts (metal+acid functions).

Adsorption of hematite ion and allied organic anion on the surface of oxides of iron, aluminium, silicon and their mixtures

Adsorption of salicylate on α -alumina was studied in aqueous medium over the pH range 4 to 9. The kinetics of adsorption of salicylate on α -alumina was studied at pH 5. The increase in adsorption density was due to the increase in surface loading. Adsorption of salicylate decreased as the pH of the medium increased and was more reflected at the isoelectric point of the α -alumina. The shifting of the characteristic peak frequencies of $-\text{COO}-$ and appearance of a new band due to $>\text{C}=\text{O}$ indicated that the salicylate was coordinated monodentally to the α -alumina with respect to the carboxyl group.

Kinetics of adsorption of benzoate and salicylate on hematite at different temperatures were studied. Isoelectric point of hematite at 25°C was estimated from the zeta potential measurement at different ionic strength and it was found to be pH 5.3. The adsorption of benzoate on hematite increased with the increase of benzoate concentration then became constant up to pH 8. But the adsorption of benzoate on hematite became linear beyond pH 9. The work was funded by DST, New Delhi.

Studies on trace metal distribution in high sulphur Makum coals of Assam

The high sulphur Assam coals from Makum Coal Field was studied for evaluation of its trace metal distribution pattern. The trace metals like Au, Ag, Cd, Pb, Cr, Mn, Fe, Co, Ni, Cu, Zn, Na, K, Ca and Mg were considered for the present study. For the first time the occurrence of Au and Ag were reported from the coals of NE India. The occurrence of gold observed as 22.8 to 32.0 ppm. The silver and lead contents varied from 25.2 to 31.4 and 43.2 to 50.4 ppm respectively. Na, Mg and Zn were observed as ion exchangeable form and all other metals under study were associated with the organic matter contents as non-exchangeable form.

EARTH SCIENCES

Study for opening up new avenues of thought regarding earthquakes in NE India

The study pertained to (i) Development of signal duration calibration empirical relations for each of analog seismic stations of different gains, in order to assign duration magnitudes to the earthquakes in the North-East India and its vicinity conformable to local Richter magnitudes, (ii) determination of over - and under - estimations of station magnitudes with respect to Local Richter Magnitudes, (iii) evaluation of station factors and iv) application of over - and under - estimations of station magnitudes and station factors as corrections to station magnitudes so as to obtain homogenized network duration magnitudes [MD(A)] conformable to local Richter magnitudes with + 0.2 units since the present order of scatter of [MD(A)] with respect to local Richter magnitudes was 0.8 units.

The study of signal durations and local Richter Magnitudes for the earthquakes in an around North - East India appeared to be unique since no such calibrated empirical relations were worked out even for most of the stations in North - East India apart from the determination of over - and under - estimations of station magnitudes and station factors. Because of the application of this study, the local area seismic station networks envisaged the reporting of network duration magnitudes conformable to Local Richter Magnitudes with + 0.2 units which was significantly better compared to the present order of scatter of [MD(A)] with respect to local Richter magnitudes i.e. 0.8 units.

The study on signal durations and local Richter Magnitudes for the earthquakes in and around North - East India provided signal duration empirical relations for each of 24 previously operated analog stations in North-East India and also provided the method to obtain consistent station magnitudes conformable to local Richter Magnitudes with + 0.2 units which however, facilitates to derive consistent energy estimates that were useful to local seismicity, lower magnitude seismicity and earthquake prediction studies.

A Study on attenuation of coda waves in the Northeastern region of India

The values of the quality factor Q_c of coda waves were computed for about 45 shallow earthquakes occurred during the periods 2000, 2001 and 2003 as recorded by Tezpur (TZR), Dokmok (DMK) and Seijusa (SJA) and Manikganj Seismic Station (MND) at the central frequencies 1, 1.5, 2, 3, 4, 6, 8, 12, 16 and 18 Hz falling in the lapse time window of 20, 30 and 40 Sec. Contour plots were obtained which showed the variation of quality factor Q_c of coda wave in the different regions of NE, India. Q_c values increased with frequency and lapse time as revealed from the distribution of coda Q_c with respect to frequency and lapse time.

Seismological data base was prepared for about 532 earthquakes in and around Chedrang Fault, East Garo Hills, Meghalaya during the period 1897 - 1999. the data base was useful for the assessment of seismic hazard in the Chedrang valley region in Meghalaya.

Study of signal durations and local Richter Magnitudes for the earthquakes in and around North-East India

An attempt was made to establish signal duration calibration empirical relations for each of 24 previously operated analog seismic stations while intending to determine duration magnitudes (MD) of the earthquakes in and around North-East India during the period 1985-1999. The relations were evaluated by calibrating signal durations with known Local Richter Magnitudes (ML) for each analog station based on models I & II since the stations were operated at different gains. The models were of the form : Model - 1 : $MD = CO + C1 \text{ Log } (S.D) + C2 D + C3h$; Model II : $MD = CO + C1 \text{ Log } (S.D) + C2 D + C3h + C4[\text{Log } (S D)]^2$, where S D is the signal duration in seconds, delta, epicentral distance and h, focal depth are in kilometers. In order to compensate the curvature

observed in the relation of Log (S D) versus ML, a fourth variable, [Log (S D)]² is introduced in Model II. Using these relations and signal durations, duration magnitudes were computed. The models yield magnitudes having standard deviations as low as 0.07 units. Further, network duration magnitudes MD(A) were determined as the arithmetic mean of station magnitudes for each of several earthquakes. Estimates of MD(A) were found to scatter with respect to ML within 0.8 units. In order to attain local magnitudes conformable to ML, scatter of MD (A) needs to be minimized. In this connection, an effort was made to quantify over - and under - estimations of station magnitudes with respect to ML. MD versus ML plots are obtained for each of 24 stations along with 1: 1 lines. With respect to 1 : 1 line, over - and under - estimations of station magnitudes were computed and applied as corrections to station magnitudes. Using corrected station magnitudes, network duration magnitudes were determined for about 825 earthquakes in and around North - East India during the period 1985-1999. These corrected MD(A) estimates were found to scatter within 0.5 units. That is, over- and under -estimations of station magnitudes account for 0.3 units out of 0.8 units of scatter of MD(A) versus ML observations. Still for further minimization of scatter, an attempt was made to determine station factors for each of 24 stations. Duration magnitude residuals were determined by comparing station magnitudes with MD(A) for each station. Station factors were obtained as the mean of deviations of MD(A) from MD for each station. These station factors were applied to station magnitudes yielding the scatter of MD(A) versus ML reduced by about 0.1 unit. In this way homogenized station magnitudes were obtained conformable to ML with +0.2 units.

Suitability of a model for the determination of consistent station magnitudes conformable to Local Richter Magnitudes is also discussed in view of over - and under - estimations of station magnitudes. Model -I appeared to be suitable because the scatter of MD(A) versus ML observation points was reduced by 0.3 units which was significant compared to that of Model - II. About 825 each - observations were used in obtaining the plot showing the distribution of MD(A) estimates based on Models - I&II with respect to ML and the empirical relations representing the line of fits were derived with the regression coefficients 0.97 and 0.97 respectively. The following relations facilitated the conversion of one magnitude into another :

Model - I :

$$MD(A) = (1.05+0.01) ML + (-0.17+ 0.05)$$

and

Model - II :

$$MD(A) = (1.01+ 0.01)ML + (-0.02 + 0.05)$$

Similarly, relations between MD(A) based on Models I & II and mb were also obtained and the corresponding relations were

$$MD(A) = (0.82 +0.03) mb (0.77 + 0.14)$$

and

$$MD(A) = (0.98 +0.03) mb (0.10 + 0.16)$$

in which mb is body - wave magnitude, obtained from the Earthquake Data Reports of United States Geological Survey. Similarly, relations between ML with respect to mb was also obtained and the respective relation was

$$ML = (0.99 - 0.03)mb + (-0.02 - 0.16)$$

Similar relations for each of about 24 stations, were also derived for obtaining local Richter magnitudes from duration magnitudes as per the models I & II.

Evaluation of an empirical relation between Network Duration Magnitude MD(A) and Energy (E)

In connection with the derivation of an empirical relation between Network Duration Magnitude, MD(A) and Energy (E), initially, corresponding to ML estimates in the interval 2.5 - 6.0, the values of Log₁₀E were calculated as per the Richter (1958) relation Log₁₀ E = 9.9 + 1.9 ML. Next, following the relations Model I :

MD (A) $(0.92 + 0.01)ML = (0.31-0.04)$; Model II: MD (A) $(0.94 + 0.01)ML + (0.21-0.04)$, the values of MD(A) were calculated corresponding to ML values. In order to derive the relation between MD(A) and Log₁₀E for ML during the interval 2. - 6.0. The relations obtained for both the models are : Model I : Log₁₀ E = $(9.23 +- 0.02) + (2.01 + 0.01) MD(A) (0.030 +- 0.001) [MD (A)]^2$ and Model II : Log₁₀ E = $(9.48 +- 0.02) + (2.03 +- 0.008) MD (A) -- (0.027 +- 0.001) [MD (A)]^2$. These relations facilitated to obtain energies released during the earthquakes of duration magnitudes 2.5 - 6.0.

Modelling of Earthquake source and ground motion in Chedrang Fault and its neighbourhood through integrated seismographic network of broadband and strong motion instrumentation : An approach towards the estimation of earthquake hazard in NER, India

During a reconnaissance survey made in the East Garo Hills, Meghalaya, suitable site was selected at Manikgunj under Mendipather subdivision based on exposed bed rocks, signal - to - noise ratio, availability of electrical power supply, proximity of distance with the Chedrang and Dudhnoi faults and proper sky view clearance with GPS antenna. The site is 11 km south east of Chedrang fault and 14 km west of Dudhnoi fault. The exposed bedrock was granite gneiss. Existence of crystalline rocks of younger deposits were also observed adjacent to the site. A RCC pier of 2X2ft was constructed coupling the bed rock at about 3ft depth and surrounded by a 6 inch gap filled with granular dry sand. A shed of 9X8X9 ft was also constructed to house the pier and seismic equipments.

CMG-3T Broadband seismometer was installed over the pier. REFTEK 72 A-08 Digitizer was connected to the external SCSI disc of 4GB memory which was also connected with GPS antenna in order to have synchronised time. The sensor and digitizer got power supply from 12V DC, 25 Plate external lead acid battery which was charged by Aplab power supply unit. A Desktop IBM computer was used not only to control the digital acquisition system but also to retrieve the digital signal spectrums. The power supply to the seismic station was obtained from the electric power supply of Meghalaya State Electricity Board (MSEB). The GPS time based three component broadband digital seismograph came into operation at Manikgunj, East Garo Hills, Meghalaya for monitoring of earthquake in the Chedrang valley fault area and its vicinity.

Seismic Phase data bulletin was published for about 119 earthquake events in the North East India and its vicinity as recorded by Manikgunj Seismic station during the period July to August, 2003 and the Epicentral plot was included. The Bulletin (Volume - I) was submitted to the DST New Delhi, the funding organisation of the work, and also conveyed to Central Seismological Observatory, Shillong operated by IMD, New Delhi, GSI, Kolkata, NGRI, Hyderabad and Tezpur Project Office of Manipur & Guwahati universities.

Source zone characteristics and site amplification behavious under seismic microzonation of greater Guwahati

The data for hypocentral paramters such as origin time, epicentral co-ordinates, focal depth and duration magnitudes for the earthquakes in lower Assam occured during the period 1982 to 1990 & 1991 were computed to obtain epicentral map for the region bounded by 23o-28oN Latitude and 89o-93oE Longitude. The work was carried out with the financial grants from the Department of Science & Technology, Govt. of India, New Delhi.

Broadband seismic network in North-East India

The laboratory in collaboration with the National Geophysical Research Institute, Hyderabad initiated the project for establishment of a three component digital broadband seismograph stations at Yaongyimsen, Mokokchung district of Nagaland and Khonsa, Tirap district of Arunachal Pradesh. One such seismograph station was already established and operated at Yaongyimsen, Nagaland.

ENGINEERING SCIENCES

Effect of molecular structure on enantio-selective permeation of amino acids in membranes from condensation products L/D amino-acids

A substantial experimental and theoretical contributions were made towards understading the effect of molecular structure on enantio-selective permeation of amino acids in membranes from condensation products L/D amino-acids with glutaraldehyde embedded in polysulfone matrix. Aiming at the development of amino-acids recemate resolution membrane, the study made so far revealed that a unique combination of single amino-acid isomer self association and diffusion across the membrane could be exploited to achieve the desired resolution. The permeation behaviour expressed in terms of diffusion and partition co-efficients of amino-acids exhibited excellent correlation with their hydrophobicity parameters. The correlations so obtained were qualitatively identical but quantitatively different from the two isomers which indicated enantio-selective permeation and leading to a scientific /technological knowledge-base on development of enantio-selective membrane for amino-acids. For recemic resolution of other therapeutically important compounds i.e. propanolol (beta-blocker), the principle of reactive extraction with a chiral selector namely cholesterol-L-glutamate was exploited using a hollow fibre membrane device. Appreciable enantioselectivity for D-propanolol in a single step was obtained.

Lipase catalysis

The kinetics and mechanism of Pseudomonas cepacia (Lipase) catalyzed transesterification of 2-O-benzylglycerol (pro-chiral) to 2-s-bezylglycerol acetate (chiral) was elucidated from both experimental and modelling study and suitable kinetic models including lipase deactivation was formulated. The works had the advantages over the conventional S&T and field practices with respect to efficiency, cost, ease of operation, etc.

These contributions opened up avenues for new and efficient technology development particularly in the field of separation and purification technology to be applied in various fields such as drug and pharmaceuticals, waste water treatment, refining of vegetable oils, etc. The studies on membrane and lipase catalyzed reaction led to generation of technological knowledge-base for (1) membrane-based process for recemic resolution of therapeutically important drug/drug intermediates (2) an environmentally benign process for a chiral drug intermediate of high therapeutical value and market demand.

Extraction and purification of green tea polyphenols (GTP)

Work on the extraction and purification of Green Tea Polyphenols (GTP) from fresh leaves of Assam was undertaken for developing technology. Different routes were investigated for extraction and purification. Process development works using various solvents and solvent system alongwith related analytical works were carried out and further work was in progress.

Enzymatic reaction

Enzyme(Lipase) reactivity in organic solvent was exhaustively studied for conversion of pro-chiral 2-o-benzylglycerol for chiral 2-o-s benzylglycerol acetate through transesterification with vinyl acetate. Intrinsic activity of the lipase was correlated with various solvent properties i.e. hydrophobicity, water solubility, electron-pair donor and acceptance index, etc. and profound correlations were obtained indicating solvent effect on the reactivity.

Enantio-selective Membrane

Enantio-selective permeation of D-Propanolol from a reactive mixture was demonstrated in a Hollow Fiber Membrane using Cholesterol-L-Glutamate (CLG) as the chiral selector. The permeation mechanism relied on reversible complexation of L-propanolol with boronic complex of CLG through which the principle of reactive extraction from aqueous to an organic medium was realized.

Geotechnical study and design of foundation with particular reference to North Eastern Region

A large number of soil exploration work were conducted at different locations and the profiles of the soil were made from the borelogs and different soil paramters based on field and laboratory analysis. The geotechnical investigations were carried out at different places of Golaghat, Sivasagar and Jorhat district

of Assam. The various engineering properties were evaluated and tabulated from the places which were identified in the district map for reference.

Study of landslide in a part of NH-52(A) from Banderdewa to Itanagar in Arunachal Pradesh

The field and laboratory work to determine sub-soil parameters and also piezometers reading for the project were completed. Further topographical survey and other field work continued. The work was funded by Department of Science & Technology, Govt. of India, New Delhi.

ECOLOGY & ENVIRONMENTAL SCIENCES

Field trial for preparing common masonry bricks utilizing Lakwa ETP sludge, a hazardous waste

Arrangements were made in consultation with ONGCL, Nazira and State Pollution Department, Sivasagar for undertaking the field trial in three brick fields involving 50,000 brick in each brick field. The raw materials were characterized, their mix proportions were characterized out for implementation in each of the fields. The trials were in progress. The work was undertaken with the financial support from Ministry of Environment & Forest, Govt. of India, New Delhi.

An appraisal of the bank erosion and flood problem of the Brahmaputra river channel from Majuli to Kaziranga

The Brahmaputra river basin ranking fourth in terms of average discharge at its mouth with a flow rate of 19.830 m³/sec and second in terms of sediment transport per unit drainage area in the world. The migration of channel with time towards south is a characteristic feature. Moreover, the location of the system in highly seismic prone area needs immediate evaluation of its geomorphologic behaviour keeping in view of its socio-economic implications. Devastation by flood and other related consequences are historically known natural hazards. However, the control of flood with numerous turbulent tributaries is not an easy task. The study has significant ecosocietal implication and created a strong data base for any future comprehensive models.

The Brahmaputra river channel from Majuli to Kaziranga was studied to evaluate its geomorphologic development. Hydrological characteristics as observed from the data spanning the period from 1971 to 1997, indicated highest volume of discharge in July (34724.01 m³s⁻¹) and the minimum in February (2344.03m³s⁻¹). Most devastating flood occur between June to October, however, June recorded the maximum fluctuation. On an average 0.57x10¹¹m³ of water and 1732.64haM of suspended sediment load are added to the river every year in the studied stretch. Majuli, the largest river island of the world within the two arms of the mighty river Brahmaputra faces an acute erosion problem and the present study on the basis of IRS satellite imagery of 1998 indicated total land area as 577.65 km², which was earlier 124 km². The available data indicated an erosion rate of 1.9 km²/yr for the period of 1920-1998. If it continues unattended, one day it would be definitely engulfed by the Brahmaputra river. The formation and tranformation of nodal points along the channel also played important roles in erosion/deposition of channel within the reach under study. The geomorphic implication of swamp developed within the reach under study at different time periods were studied and their importance in flood management and wet land development suggested.

Study of toxic heavy metals in the soil/water systems in and around Lakwa oil field

The North Eastern region of India occupies a prominent position in the history of natural resources and it contributes a huge amount towards the growth of national economy. However, active exploitation and exploration of crude oil causes a large-scale disposal of polluted waters, oil contaminated waste waters and also spillage of crude oil. This complex nature of mechanisms of anthropogenic substances in the environment and its effect on human system viz. the atmosphere or soil during occupational (work place) or recreational activities assumes a great dimension in the environmental toxicology.

Since 1968, Lakwa became the major oil producing centre of the region with more than 200 oil producing wells. The unattended environmental degradation caused by the oil field developmental activities became a subject of concern. The basic objective of the study was to ascertain the concentration of total heavy

metals in soil/water systems both vertically and laterally around Lakwa oil field region (27.3oN : 94.7oE) and to establish the background/threshold levels and their impact on existing ecology of the region.

The soil/water systems around Group Gathering Stations (GGSs) I and III within the Lakwa oil field region were studied both laterally as well as vertically. Texturally sandy-clay-loam (80%) and sand loam (20%) represented the soil of the area. The mean soil background values for Fe, Cu, Cr, Zn, Pb, Ni and Co were found at the higher side within a broad range of fluctuation. The background values of water samples were within the stipulated guide line values of IS (1982) and WHO(1984). The major and trace metals within pond and stagnated water bodies showed marked seasonal variations. The higher values of Na, K, Ca and Mg over the background values could be attributed to the entry of oil contaminated waste water from the GGSs, which imparted hardness to the water bodies.

The heavy metal contents were found to be associated with the clay/silt fractions. The chemical partitioning of metal contents indicated higher association with the residual and Fe-Mn oxide fractions and possessed less environmental risks. The study indicated ample evidences to implement a continuous geochemical monitoring in the area. The local mean values of metals when compared with the accepted values of normal abundance and geochemical background, indicated two to four fold increased in this area. However, the values were within the range of normal abundance as well as from natural soil geochemical behaviour, anthropogenic influence might have a close bearing on the association of metals with the soil system in the studied area.

Preparation and optimization of novel Hydrotalcite based ceramic monolith for decomposition of environmentally harmful N₂O gas

Prepared a set of hydrotalcites like Mg-Al, Ni-Al, Palladium doped Co-Al, Zn-Cr etc. for use as a component for coatings over honey-comb ceramic monoliths. Carried out shear rate - shear stress flow behaviour study for aqueous montmorillonite - hydrotalcites using Brookfield viscometer. Fabricated a laboratory scale dip-coating equipment and completed design for an improved version. The work was funded by Ministry of Environment and Forest, New Delhi.

Study on mitigation and erosion of Subansiri river

The part of the Subansiri River basin, which is within the plains of Assam is one of the most thickly populated area and faces serious perennial problems of heavy flood. The catchment area of the river is about 37,000 km², out of which 10,000 km² lies in Assam and remaining part lies in Arunachal Pradesh and Tibet Himalayas. The length of the river is 520 km. The river has been causing severe flood and bank erosion after the Great Assam Earthquake of 1950. The significant change in its river course and fluviosedimentologic behaviour after the earthquake needed an in-depth investigation. The study attributed a significant impact on the migration of river system and its erosion behaviour. After the 1950 Great Assam Earthquake, the river migrated westward at some places upto a distance of 5 km.

SOCIETAL ACTIVITIES

Awareness programme for cultivation of medicinal plants in Arunachal Pradesh

With the financial support extended by the Department of Biotechnology, Govt. of India, 15 awareness programmes-cum-exhibitions on medicinal plants cultivation and processing were conducted at various places of Arunachal Pradesh viz. Lumla, Along, Roing, Anini, Ruksin, Seppa, Pedung, Iduli, Kongkong, Boling, Meka, Bhima, etc. Under this programme, two nursery-cum-demonstration centres were also established at Roing (tropical climate) and New Bomdila (Temperate climate). Medicinal plants appropriate to the said climatic zone were introduced for multiplication and demonstration.

RRL Branch laboratory at Itanagar conducted an awareness programme on medicinal and aromatic plant at Namsu village in West Kameng district of Arunachal Pradesh. A total of 45 villagers took part in the programme.

Assistance to Self Help Group

A total of 95 hectares of land were covered under aromatic plants cultivation engaging 38 families, producing 250 tons of oil annually. 525 village level farmers and women under Self Help Group were trained in cultivation of various mushroom species viz. Pleurotus djamore, P. florida & P. cornucopiae. These SH groups belonged to Bahona, Kenduguri, Teok, Titabar of Jorhat district, Bokakhat and Golaghat of Golaghat district, Sariahjan, Bokajan, Silanijan in Karbi Anglong District, Jogeshpur, Gopinathpur, Koirara & Gopal Bazar of Nalbari District of Assam.

Mission oriented rural development activities

The cultivation and processing technologies of citronella and lemongrass were transferred to 62 beneficiaries of Nalbari district under the societal mission oriented programme of RRL-Jorhat with the financial assistance of Department of Biotechnology, Govt. of India, New Delhi. More than 70 hectares of land was brought under the cultivation. Two distillation plants of 700 kg per batch capacity were installed at Kairara and Kshudramakhibaha of Nalbari district. Scientists from RRL imparted training and demonstrated the processes and distributed the basic planting materials free of charge to beneficiaries. The fallow high lands were utilized for cultivation of these crops which paid handsome returns to the farmers. Installation of distillation units added advantage to the growers.