

PROCEEDINGS

HANDS ON TRAINING PROGRAM

on

Nuclear magnetic resonance (NMR), Single crystal X-Ray Diffraction (XRD), Atomic Emission Spectroscopy (AES), High Performance liquid Chromatography (HPLC) & Photoluminescence Instruments

2nd January 2023 to 9th January 2023
at SCS, Goa University



400 MHz NMR



SCXRD



AES



HPLC



Photoluminescence

**Organised by
School of Chemical Sciences (SCS),
Goa University**

in association with

**Sophisticated Analytical Instrumentation Facility (SAIF),
Panjab University,**

**Chandigarh under Synergistic Training Program Utilizing
the Scientific and Technological Infrastructure (STUTI)**



Professor Harilal B. Menon
Vice Chancellor



Goa University
Goa India-403206

MESSAGE

A warm welcome to all the participants of the Training program on “Operation of Nuclear magnetic resonance (NMR), Single crystal X-Ray Diffraction (XRD), Atomic Emission Spectroscopy (AES), High Pressure liquid Chromatography (HPLC) & Photoluminescence Instruments” organized by School of Chemical Sciences (SCS), in association with Sophisticated Analytical Instrumentation Facility (SAIF), Panjab University, Chandigarh under Synergistic Training Program Utilizing the Scientific and Technological Infrastructure (STUTI) initiative of Department of Science and Technology (DST) at Goa University. This is truly a platform which aims to sensitize the young generation about the state-of-the-art equipments through open access Science and Technology infrastructure across the country.

My heartiest Congratulations to the Sophisticated Analytical Facility (SAIF)/Central Instrumentation Laboratory (CIL), Panjab University, Chandigarh for being selected as the Project Management Unit (PMU) and exquisitely executing their role as the hub manager for the STUTI program in the region. The School of Chemical Sciences (SCS), Goa University deserves an applause for taking the initiative of hosting this hands-on training program. It is heartening to see an interdisciplinary consolidation which is the need of the hour.

The facilities of NMR, AES, HPLC and photoluminescence techniques are extensively used by researchers, for academics and industrial applications in Goa and elsewhere. The programme enables hands-on training for separation, identification, and purification of the components.

The endeavor of the organizing committee will certainly result in a fruitful accomplishment. I wish the event a great success.


(Prof. H. B. Menon)



Prof. V. M. S. Verenkar
Dean, School of Chemical Sciences



Goa University
Goa India-403206

MESSAGE

It is a matter of honour for me to extend a very warm welcome to all the dignitaries who are gathered here for the Hands on Training Program on “Operation of Nuclear Magnetic Resonance (NMR), Single Crystal X-Ray Diffraction (XRD), Atomic Emission Spectroscopy (AES), High Pressure liquid Chromatography (HPLC) & Photoluminescence Instruments” being organized by School of Chemical Sciences, Goa University, Goa in association with Sophisticated Analytical Instrumentation Facility (SAIF), Panjab University, Chandigarh under Synergistic Training Program Utilizing the Scientific and Technological Infrastructure (STUTI).

High end research in chemical science is the need of the hour in developing novel drug molecules for the treatments of numerous outrageous diseases. The training aims at creating the significantly needed perspectives and expertise in constantly progressing trends and practices in NMR, X-ray Diffraction (XRD) and Chromatography based techniques. The current hands-on training program could be another milestone in DST’s initiative towards building human resources through STUTI.

These 7 days training program will also encourage the researchers and industry to provide suitable solutions and interface for future scientific ventures. During this training program, attendees will have the opportunity to visit facilities available at the School of Chemical Sciences (SCS). This will also provide tremendous opportunities for all the stakeholders to come together, collaborate and complement each other.

I congratulate the Coordinators and the Organizing Committee for having worked hard to ensure the success of the 7-day training program at the Goa University campus.

I wish you all a fruitful and successful training program.

(Prof. V. M. S. Verenkar)



Dr. Prajesh Volvoikar
Workshop Convenor
School of Chemical Sciences,



Goa University
Goa India- 403206

MESSAGE

On behalf of the organizing Committee, it is an honour and pleasure to officially welcome you to the Training Program. The Training program encompasses key aspects NMR spectroscopy, SC-XRD, HPLC, and Photoluminescence techniques that are employed extensively in modern day new molecules discovery, research and development and diagnostics. This training program will primarily help the participants who are seeking basic and advanced level training in these techniques to get insight into their research and other industrial based technical operations. The training program enhances the working horizon among faculty, post-doc fellow, PhD Fellow. The scope of the training program is also extended to the industrial persons who are extensively involved in new molecules production and their detailed qualitative and quantitative descriptions. This training program will play a vital role in understanding formations, actions, and regulations of various novel molecules that can be used to efficiently target new drugs.

This puts light on the increasing importance of on the hands-on training on NMR spectroscopy, SC-XRD, HPLC, Photoluminescence, etc. techniques for the qualitative and quantitative analysis. The programme enables participants to acquire the basic NMR techniques required for success in scientific research in chemical sciences. During this training program, attendees will have the opportunity to visit facilities available at SCS and provide basic introduction of other instrumental facilities like BET, AFM, VSM TG-DTA etc. Additionally, the attendees will have an opportunity to closely interact with eminent scientists of Goa University.

We hope that this training program will stimulate new ideas and approaches for promoting chemistry and enhance skills in the field. We wish all delegates a fruitful attendance and involvement in the excellent event and hope that this training program is productive. Thank you for your participation and we wish you a very enjoyable stay in Goa.

(Dr. Prajesh Volvoikar)

GOA UNIVERSITY, GOA

Goa University was established under the Goa University Act of 1984 (Act No. 7 of 1984) and commenced operations on 1 June 1985. The university provides higher education in the Indian state of Goa. It is located on Taleigao Plateau overlooking Zuari estuary on a picturesque campus spread over 402 acres with state-of-the-art infrastructure such as faculty blocks, administrative building, library, sports facilities, student hostels, bank, post-office, staff quarters, etc. Campus-Wide Internet connectivity with strong bandwidth is available for all 24 hours a day.

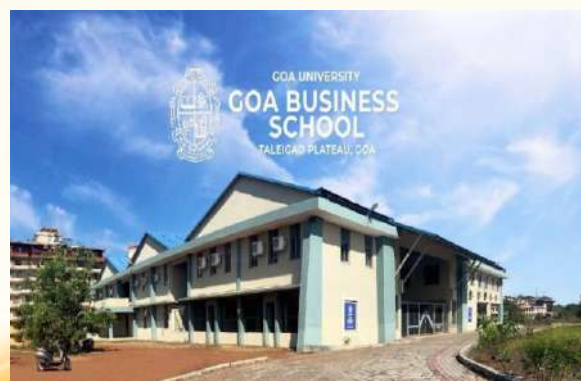


The University took over the enhanced role of Centre of Post-Graduate Instruction and Research (CPIR) which was set after the liberation of Goa by India in December 1961, by the University of Bombay (now Mumbai), in June 1962. Since 1985 Goa University offers graduate and post-graduate studies and research programmes. It is currently (2014-19) accredited to the National Assessment and Accreditation Council (NAAC) in India with A Grade. The National Institutional Ranking Framework (NIRF) (an organ of Ministry of Human Resources and Development, Govt. of India) has currently (2022) ranked the University at Rank-band 101-150. Among the QS World University Rankings for 'BRICS countries 2019', Goa University is among the group of 241-250 universities. There are about 9000 universities in BRICS countries. QS University rankings - a World University rankings agency - has ranked Goa University in the range of 61-65 among Indian Universities for the year 2022 and among the QS World University Rankings for 'QS Asia University Rankings 2022', Goa University is among the group of 501-550 universities. Over the past 35 years, the University has steadily expanded its reach, both in terms of the number

of affiliated colleges - professional and general education numbering to 61, as well as the diversity of courses offered. These colleges offer various courses leading to a degree at graduate, post-graduate level. 7 of them are also recognized as research centres to offer Ph.D. programmes. The University, on its campus, has 10 schools (Table 1). The formation of schools has been done at the start of the academic year 2019-20 with amalgamation of traditional departments to allow organic evolution of new courses. They offer programmes leading to Undergraduate degree (3), Master's degree (35) and Ph.D. degree (25) in various disciplines. In addition, 7 recognised institutions in various disciplines situated in the state of Goa are also recognised for research programmes leading to Ph.D. degree by the University.

Over 30000 youth from all talukas of Goa are studying in affiliated colleges and over 2000 are enrolled for post-graduate programmes at the University campus. The percentage of women (over 60%) outnumber men (about 40%).

The University has made a significant impact at the national level in various areas of specialization and draws students in select disciplines from across the country. Geographically, Goa is located in an ecologically sensitive region along the Western Ghats and the Arabian Sea. Goa University has appropriately emerged as an important resource centre for research in the field of flora and fauna endemic to this region, as well as the marine environment. The Ministry of Earth Sciences has recognized Goa University's significant contribution in this domain, as a consequence of which a Centre of Excellence was established in Marine Microbiology. The Departments at the University have developed excellent research facilities. Large funding for research is received from Central Government agencies such as University Grants Commission, Department of Science & Technology, Ministry of Earth Sciences, Ministry of Environmental Sciences and Climate Change, Department of Biotechnology, etc. For a relatively small university, Goa University attracts generous funding of research projects from national funding agencies which reflects upon the high quality of research undertaken at the University. The research outcomes (over 6500) in the form of papers, theses, etc., have been made available to the public over the university website in its publication's repository. The Web of Science® reports about 1400 items on their database accruing over 16000 citations. The University publications have an h-index of 54.





In addition to the conventional graduate and postgraduate programmes, Goa University has also taken initiatives to provide innovative programmes. Some noteworthy initiatives are the National Resource Centre in Marine Science under the Ministry of Human Resource Development (MHRD) for the professional development of higher education using the MOOCs platform SWAYAM, the State Resource Centre for Women funded by Ministry of Women and Child Development, Govt. of India as well as Dept. of Women and Child, Govt. of Goa, the Study India Programme with Nihon University of Japan, etc. Goa University launched the Visiting Research Professors Programme (VRPP) from the year 2013-14 to bring luminaries in the field of liberal arts & literature, social and natural sciences, and other fields. The visiting professors interact with students, deliver lectures, offer courses, and work on collaborative research projects, or stimulate the creation of art installations and music performances with faculty and students. These visiting professors are found to generate a creative environment in learning and contemporary knowledge production practices with their intellectual and aesthetic endeavours. The programme is being supported by Directorate of Art and Culture, Government of Goa through generous grants and open to the general public too.

School of Chemical Sciences

The establishment of the School of Chemical Sciences took place by transforming one of the existing and oldest (established in 1965 as a part of Centre for Post-Graduate Instruction and Research of the then Bombay University) department (of Chemistry) into a School. The School is currently located in the faculty block E since April 2013, with modern infrastructure, conducive for quality education and research in Chemistry. The School (formerly Department of Chemistry) earlier had Organic, Inorganic, Physical and Analytical Chemistry as distinct streams and since AY 2019-20, the Biochemistry has been added as its fifth stream. The establishment of this School is in line with the reorganization of the institutional architecture that allows us to rebrand and showcase our strengths in research and teaching in various Chemistry streams. The research at this School has been recognized by the award of projects to the individual faculty members and also receiving handsome grants from the University Grants Commission (UGC) and the

Department of Science & Technology (DST), Govt. of India in their prestigious programs like UGC-SAP and DST-FIST. The School of Chemical Sciences has various modern sophisticated instruments required for state-of-the-arts research viz., single crystal X-Ray diffractometer, 400 MHz NMR spectrometer, AFM, VSM, Thermal analysers, Ball mill, Microwave reactor, LCMS, HPLC, Surface area analyser, UV-Visible, PL and Infra-Red spectrophotometers, CHN analyser, Atomic emission spectrometer, Gas chromatography etc. which has attracted many DST-Inspire Fellows and Kothari Fellowship holders to our school. The school has a rich tradition of excellent research output and the culture of performing better which has resulted, over the years, in the contribution of 30-40 percent of research publications of Goa University, annually.



The school offers two-year M.Sc. (64 credits) program in Organic, Physical, Inorganic, Analytical Chemistry as well as in Biochemistry. The research work leading to Ph.D. degree in the above subjects encompasses various domains of chemical research with emphasis on synthesis of natural products, developments of reagents for organic synthesis, synthesis and application of polymers and nanocomposites, synthesis of pure and mixed metal oxide nanoparticles and their applications as gas sensors/supercapacitors etc. The research extends further into computational and theoretical chemistry, synthesis of coordination compounds as models for biological systems, development of catalysts/ electrocatalysts and their use in kinetics evaluation for various fundamental processes related to energy and environment.

HIGHLIGHTS PROGRAM OF THE TRAINING

The aim of this 7-day training is to provide skills to participants with the basic knowledge required to handle sophisticated instruments like Nuclear magnetic resonance (NMR), Single crystal X-Ray Diffraction (XRD), Atomic Emission Spectroscopy (AES), High Performance liquid Chromatography (HPLC) & Photoluminescence Instruments. This hands-on training program is intended for participants who are seeking basic and advanced-level insight and hand on training on these instruments. This includes Faculty/Scientists/Post-Doc Fellows/Ph.D. Fellows who are actively involved in Research and Development (R&D) and require knowledge of instrumentation technique.

It will enable participants acquire the basic NMR techniques required for success in scientific research in chemical sciences. Participants will experience hands-on training on, NMR, Single crystal XRD, HPLC, AES, Photoluminescence etc. During this training program, attendees will have the opportunity to visit facilities available at School of Chemical Sciences (SCS) facility and provide basic introduction of other instrumental facilities like BET, AFM, VSM TG-DTA etc. Additionally, the attendees will have an opportunity to closely interact with eminent scientists of Goa University.

LEARNING OUTCOMES OF THE PROGRAM

At the end of the training, participants will be aware and will have practical knowledge on using and operation of following:

- Basic and advanced information on Nuclear magnetic resonance (NMR) technique
- Handling of samples and spectra interpretation of ^1H NMR and ^{13}C NMR
- Basic, advanced information, handling of samples and spectra interpretation of single crystal X-ray technique
- HPLC techniques
- Atomic emission spectroscopic (AES) technique
- Photoluminescence Instrument
- Data interpretations of these techniques

RESOURCE PERSONS



Prof. Vishnu S. Nadkarni
Registrar and
Senior Professor in Organic Chemistry
Goa University



Prof. Ganga Ram Chaudhary,
Director, SAIF,
Panjab University



Prof. Vidyadatta M. S. Verenkar
Dean, SCS
Professor in Inorganic Chemistry
Goa University



Dr. Pratishtha Pandey
Head, DST Infrastructure Division,
Government of India



Prof. Sundar N. Dhuri
Vice Dean, Research, SCS
Professor in Inorganic Chemistry
Goa University



Dr. Yogesh Nagpal
Managing Director
Winsom IP

RESOURCE PERSONS



Dr. Rupesh E. Patre
Associate Professor in
Analytical Chemistry
Goa University



Dr. Sandesh T. Bugde
Assistant Professor in
Organic Chemistry
Goa University



Dr. Venkatesha R. Hathwar
UGC - Assistant Professor of Physics
Goa University



Dr. Hari K. Kadam
Assistant Professor in
Analytical Chemistry
Goa University



Dr. Prajesh S. Volvoikar
Assistant Professor in
Analytical Chemistry
Goa University

Day 1. 02.01.2023

Dr. Prachi Torney, Assistant Professor School of Chemical Sciences welcomed the Chief guests and guest of honour. She further gave a formal welcome to dignitaries and participants. She also highlighted the diversity of participants in the STUTI program ranging from Haryana to Tamil Nadu.



Guest of honour Prof. Ganga Ram Chaudhary Director SAIF/CIL and Punjab University and Coordinator of the STUTI-PMU, PU, Chandigarh focused on integrating technology for transforming waste into valuables. He also shared the details of the state-of-the-art equipment's and facilities that SAIF/CIL offers. Along with this, he highlighted the contributions made by the department by innovating and donating air-purifiers and UV currency sanitizers to hospitals

and offices in and the previously held STUTI training and awareness programs.

Dr. Yogesh Nagpal, Managing Director, Winsom IP delivered lecture on Importance of Patents in Designing and Conducting Scientific Research focusing on types of Intellectual Property Rights. Patents, Copyright, Trade Marks, Registered designs, and trade secrets were discussed in detail. He further briefed about IP rights, Protects and How to acquire these intellectual property rights. Sir mentioned Stages of Designing Research which includes defining the topic, narrow, gather, creative, develop, find, design experimental, compile and write. Evening session included visit to the laboratories of School of Chemical Sciences where instruments like NMR, SCXRD, VSM, TG-DTA, PL, HPLC, AES etc facilities were available and a brief overview of each instrument was given to the participants.



Day 2. 03.01.2023

Prof. V. M. S. Verenkar, Dean of Goa University, School of chemical sciences briefed the participants about the campus of Goa University, the projects being carried out, credentials of faculty and the overall history of the Goa University. Further he added about the various DST sponsored schemes. After this address by the dean of the school there was introduction of the participants. Dr. Hari kadam delivered a talk on various DST sponsored scheme regarding

R&D infrastructure. He also encouraged the participants and the faculty members to avail the opportunities through the STUTI programme to build human resources and its knowledge capacity.



In the second half of the morning session began with Dr. Hari kadam delivered a lecture on NMR briefing the instrument and its parts, basic principle of NMR, interpretation of data, the standard, solvents used, and he further concluded his lecture mentioning the various applications of NMR. This was followed by Dr. Rupesh Patre who discussed an overview of HPLC, he use of HPLC and principle behind it. He further spoke on various points like why separation is needed, two types of elution, normal and reverse phase, stated the difference between GC and HPLC. A detailed schematic diagram was also shown stating the working principle. Later Sundar Dhuri briefed about single crystal XRD and differentiated the powder XRD and single crystal XRD. SCXRD talk was followed by talk on photoluminescence which was delivered by Dr. Sandesh Budge. His lecture began with definition of spectroscopy and also explained on how PI can be used to differentiate real and artificial diamonds. Jablonski diagram, different types of luminescence etc were also discussed

Afternoon session began with talk by Sundar Dhuri who gave an overview of single crystal XRD, its importance in structure determination and also showed various crystals synthesized by him and his PhD students in his laboratory. Sir mentioned that if crystal structure changes, then the phase changes and he also added that the simplest method of formation of crystal is by simple evaporation.



Venkatesh Hatwar explained in detailed the working of single crystal XRD instrument and also a comparison between powder XRD thus briefing their importance.

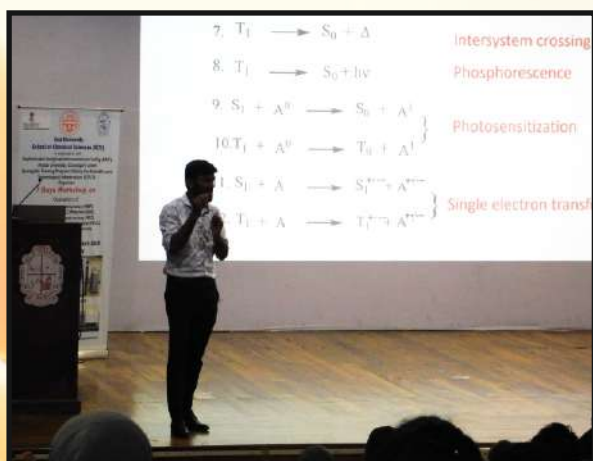


Day 3. 04.01.2023

Morning lecture was regarding NMR, which was delivered by Dr. Vishnu Nadkarni. This lecture included the principle, instrumentation and the working of NMR. Procellsonal frequency, chemical shift value, gyromagnetic ratio, different functional group signal.

Second lecture was delivered by Dr. Rupesh Patre explaining working and principle of HPLC And detail with a schematic block diagram. He further different names of HPLC, types of detectors, mobile phase, problems involved in general elution, reason for band broadening, pump, precolumn. Sir further explained about the column temperature where heaters are used to control the temperature, elution trends of solute, retention time, peak width, resolution etc

During afternoon session Dr. Sandesh Bugde discussed photo luminescence. He explained different types of electronic transition processes including excitation, vibrational relaxation, fluorescence, internal conversion, intersystem crossing, phosphorescence and photosensitization. Sir further briefed about the light source, monochromators, light detectors used in the instruments. excitation, emission, fluorophores was explained in the session.



Day 4. 05.01.2023

The session started with the demonstration of the sample analysis by research scholar Mr. Dinesh Nadimetla. He gave a brief introduction to all the participants about the spectroscopy, EMR spectrum and explained the basic principles of spectroscopic techniques such as ^1H NMR and ^{13}C NMR. He also explained the identification of different peaks and its interpretation in the spectra obtained using the software. Later he helped the participants with the injection of the sample and also the precautions and safety measures to be followed while handling the instrument.



In the second session, the demonstration and hands on training of the Single Crystal X-Ray Diffractometer (SCXRD) was given by a research scholar Ms. Nikita Harmalkar. She gave a detailed overview of the instrument including its basic principle and the working of the instrument. She also demonstrated all the participants with the selection of the best crystal appropriate for the SCXRD using a microscope. she even showed different types of tools used

to separate a crystal from the bulk sample. Later on, she gave a proper demo of the software to analyse the crystal structure data obtained from the instrument. She mentioned the safety measures to be followed during handling of the instrument.



Day 5. 06.01.2023

The morning session included a detailed demonstration of atomic emission spectroscopy (AES) by Dr. Prajesh Volvoikar. After briefing the participants about the important applications of AES in research studies. He explained the working of the AES instrument and covered up all the theory portion of the technique. Later on, the demonstration and hands on training of the AES instrument was given by the research scholar Ms. Luen Dsouza, wherein she introduced the AES instrument and its working to all the participants. She guided the participants regarding

all the parts of the instrument and its functions, later on how to upload the sample and software operation.



The afternoon session started with the demonstration and hands on training of the high-performance liquid chromatography (HPLC) instrument which was handled by the expert Ms. Siddhi Salgaonkar, a research scholar. She demonstrated the sample analysis to all the participants and explained the working of each component of the instrument. The data interpretation was explained by her wherein she covered the stepwise operation of software.





Day 6. 07.01.2023

The morning session started with the lecture by Dr. Hari K. Kadam on interpretation of data and solved the doubts on NMR spectroscopy. He gave the detailed explanation on spectra solving. He even provided the participants with some unsolved spectra for practice.

The other half of the session started with the demonstration and hands on training of photoluminescence (PL) instrument by Dr. Sandesh T. Bugde. The trainees were acquainted with the importance of the instrument and its application in research related work. He gave a detailed explanation and demonstration starting from sample loading till data interpretation on the software





KEYNOTE SPEAKER

Day 6 we had a talk by Dr. Pratishtha T. Pandey, Head (R & D Infrastructure Division) DST, GOI on various DST scheme to participants and faculty members of university. It was a interactive session where participants and faculty members has good discussion with keynote speaker.





Day 7. 09.01.2023

Participants analysed their samples on AES, HPLC, Single crystal XRD and NMR with the help of research scholar of host institution. Participant could have actual feel of instrument as they operated these DST funded instrument to add to their knowledge.

SAMPLE ANALYSIS BY THE PARTICIPANTS



VALEDICTION & MOMENTO AND CERTIFICATE DISTRIBUTION





PARTICIPATION SUMMARY

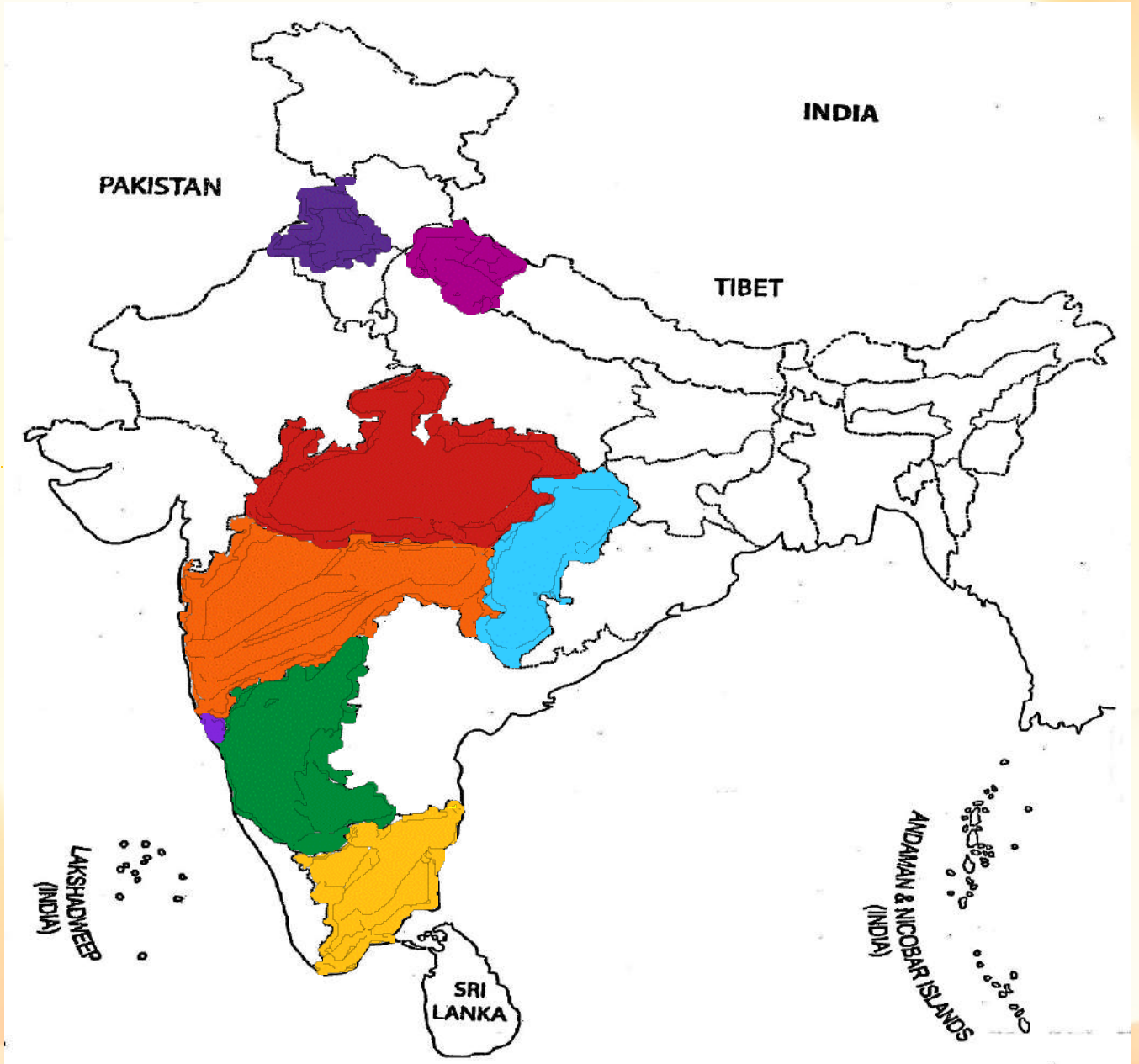
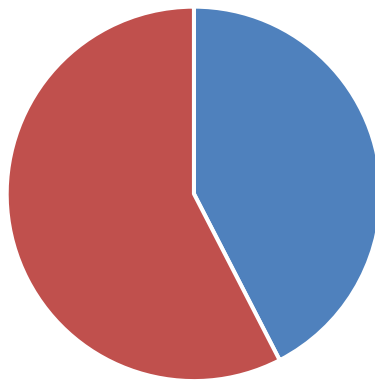
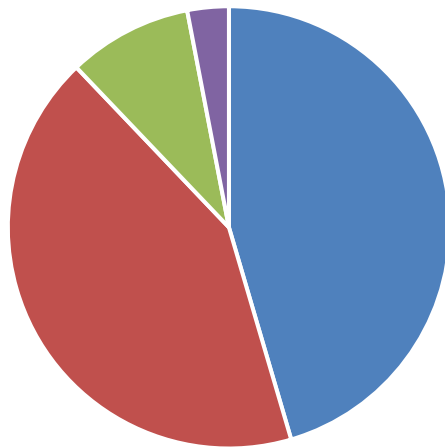


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Prepared By:

Dr. Prajesh S. Volvoikar & Dr. Diptesh G. Naik

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