ST. JOSEPH'S COLLEGE (AUTONOMOUS)

BENGALURU - 27

DEPARTMENT OF CHEMISTRY

SYLLABUS FOR POSTGRADUATE COURSE
M.Sc. ANALYTICAL / ORGANIC CHEMISTRY
FOURTH SEMESTER - DEPARTMENT ELECTIVE

2021-2024

Re-accredited with 'A++' GRADE and 3.79/4 CGPA by NAAC
Recognised as "College of Excellence" by UGC

FROM 2021-2022 ONWARDS

Semester	IV
Paper code	CHDE 0221
Paper title	Dept. elective: Chemistry of Materials
Number of teaching hours per week	4
Total number of teaching hours persemester	60
Number of credits	4

1. INTRODUCTION **1 h**

Importance of solids in technological applications, solids as materials.

2. MATERIALS CHARACTERISATION TECHNIQUES **(14 + 1) h**

Electron microscopy and related techniques: transmission electron microscopy, scanning electron microscopy, electron diffraction, electron energy loss spectroscopy, energy dispersive X-ray spectroscopy. Atomic force microscopy. Photoelectron spectroscopy and auger spectroscopy. *Particle induced X-ray emission spectroscopy*. Extended X-ray absorption fine structure. Porosity and surface area measurements by sorption- desorption – BET and BJH methods.

3. LAYERED SOLIDS AND POROUS MATERIALS **(8 + 2) h**

Layered solids: general structural features, classification, intercalation and deintercalation. Structure, composition, properties and applications of cationic clays, layered double hydroxides, layered chalcogenides and layered oxides. Polytypism in layered solids.

Microporous and mesoporous materials: structure, composition, synthesis, properties and applications of zeolites and zeotypes, metal organic frameworks. *Macroporous solids: general methods of preparation, properties and applications*.

4. SUPERCONDUCTORS **5 h**

Definition, Meissner effect, type 1 and type 2 superconductors, features of superconductors, Frolich diagram, Cooper pairs, theory of low temperature superconductivity, high Tc superconductors.

5. SOME MATERIALS OF RECENT INTEREST (5 + 1) h

Multiferroics, giant and colossal magneto resistance (GMR, CMR) materials, thermoelectric materials, topological materials, *conducting polymers*.

6. NANOMATERIALS (20 + 3) h

Nanoregime, properties at nanoregime- electronic structure of metals and semiconductors at nanoscale, quantum confinement, superparamagnetism of magnetic solids at nanoscale. Classification of nanomaterials.

Synthesis of nanocrystals: top-down vs bottom-up synthesis, dispersity, La Mer principle, capping agents, simple solution-based synthesis, inverse-micelle synthesis, spray pyrolysis, sol-gel, combustion, solvothermal and electrochemical synthesis.

Synthesis of thin films: physical vapour deposition – pulsed laser deposition and atomic layer deposition, chemical vapour deposition, electrodeposition.

Synthesis of 2D nanomaterials: mechanical, solvent-mediated, and chemical exfoliation.

Use of PXRD, UV-visible and Raman spectroscopy in the characterization of nanomaterials.

Nanocomposites: definition, different types, general methods of synthesis and applications.

Carbon-based nanomaterials: structure, synthesis, properties and applications of fullerenes, carbon onions, carbon nanotubes and graphene.

Applications of nanomaterials: nanomaterials in energy conversion and storage; environmental amelioration applications; electronic and optoelectronic applications; biological and theronastic applications.

Nanotoxicity.

References:

1. C. N. R. Rao and J. Gopalakrishnan, New Directions in Solid State Chemistry, Cambridge Univ. Press, 2ndEdn., 1997.
2. Molecular Sieves, Science and Technology Series, Volume 6, 2008.
3. Kenneth J Klabunde, Nanoscale Materials in Chemistry, John Wiley and Sons (2000).
4. C.N.R Rao, Chemistry of Nanomaterials, Wiley VCH (2007).
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7. The biomolecule-nanoparticle interface, Vincent M Rotello, Nano Today, Vol 2, Number 3, June 2007.
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9. Guozhong Cao, Nanostructures and Nanomaterials: Synthesis, Properties and Applications, Imperial College Press (2004)
10. Hybrid Nanocomposites for Nanotechnology: Electronic, Optical, Magnetic and Biomedical Applications, *Editor* Lhadi Merhari, Springer Publications (2009)
11. Characterization techniques for nanoparticles: comparison and complementarity upon studying nanoparticle properties, Nanoscale, 2018, 10, 12871

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Code number and Title of the paper: CHDE 0221**:** Chemistry of Materials

Chapter Number	**Title**	**Number of teaching hours (As mentioned in the syllabus)**	**Maximum marks for which questions are to be framed from this chapter (including bonus questions)**
1.	**Introduction**	1	2
2.	**Materials characterisation techniques**	15	26
3.	**Layered solids and porous materials**	10	17
4.	**Super conductors**	5	8
5.	**Some materials of recent interest**	6	10
6.	**Nanomaterials**	23	40
Total marks excluding bonus questions			70
Total marks including bonus questions			103

Semester	IV
Paper Code	CHDE 0321
Paper title	Dept. elective: GREEN CHEMISTRY AND DIVERSITY OF ITS APPLICATIONS
Number of teaching hours per week	4
Total number of teaching hours per semester	60
Number of credits	4

1. PRINCIPLES OF GREEN CHEMISTRY 3 h

Twelve Principles of green chemistry: prevention of waste, less hazardous chemical synthesis, safer solvents and auxillaries, use of renewable feed stock, catalysis, real time analysis for pollution prevention, atom efficiency, designing safer chemicals, design for energy efficiency, reduced derivatives, design for degradation, inherently safer chemistry for accident prevention.

2. USE OF ULTRASOUND AND MICROWAVE IN ORGANIC SYNTHESIS (4 + 1) h

Use of ultrasound: instrumentation and the phenomenon of cavitation. Sonochemical esterification, oxidation and reduction. Use of microwave: introduction, reaction vessel and medium, specific effects, atom efficiency, advantages and limitations, N-alkylation and alkylation of active methylene compounds with aldehydes and amines.

Diels-Alder reaction and oxidation of alcohols.

3. MECHANOCHEMISTRY 5 h

Definition of mechanochemistry. Mortar and pestle for organic synthesis. Ball milling as reactors for organic synthesis; effect of operating frequency, milling time and reaction temperature. Energy efficiency; comparison of $KMnO_4$ mediated oxidation of p-toluidine to other methods (classic heating, microwave and ultrasound).

4. POLYMER SUPPORTED REAGENTS IN ORGANIC SYNTHESIS (5 + 1) h

Introduction- structure of polymer supports, properties of polymer support, advantages of polymer supported reagents and choice of polymers.

Applications: substrate covalently bound to the support- synthesis of oligosaccharides, Dieckmann cyclisation. Use of Merrifield resin in peptide synthesis.

Linkers and advantages, reagent linked to a polymeric material - synthesis of polymer bound per acid and its applications.

Polymer supported catalytic reactions: preparation of polymer supported $AlCl_3$, and application in acetal formation reaction.

5. PHASE TRANSFER CATALYSIS (PTC) AND CROWN ETHERS (6 + 1) h

Definition, mechanism of PTC, types of PTC reactions and advantages. Preparation of catalysts and their application in alkylation, oxidation, and reduction reactions.

Crown ethers: general structure, nomenclature, features, and nature of donor site. General synthesis of crown ethers. Synthetic applications: aromatic substitutions.

Generation of carbenes and alkylation.

6. MULTICOMPONENT ONE-POT REACTIONS (4 + 1) h

Meaning of one pot synthesis (mention of synonyms domino/cascade/ tandem reactions).

Effective reactions for one-pot synthesis; reaction in which the intermediate compound is unstable, reaction in which the intermediate compound is hazardous, reactions in which there is equilibrium between intermediate compounds, reaction in which the starting compound is in equilibrium with the intermediate, *reaction in which same reagents are employed in subsequent reactions; an example each.*

Restriction for one-pot reactions; reaction, solvent, amount of reagent. Ex: Passerini, Ugi, Biginelli and Mannich reactions.

7. ORGANOCATALYSIS (12 + 1) h

Introduction- types of organocatalysts, advantages, reusability.

Enamine catalysis: Aldol and Mannich type reactions, α-heteroatom functionalization, direct conjugate additions via enamine activation.

Iminium catalysis: cycloaddition reactions, 1,4-addition reactions, transfer hydrogen reactions, cascade reactions- total synthesis of natural products- tetrahydroquinoline alkaloids.

N-Heterocyclic Carbenes (NHC): Conjugate umpolung of α,β-unsaturated aldehydes for the synthesis of gamma-butyrolactone.

Hydrogen bonding networks - epoxidation of olefins and Baeyer–Villiger oxidation of ketones. Supported organocatalyst and Ionic liquid organocatalyst.

Precursors and generation of NHC.

8. GREEN CHEMISTRY PRACTICES IN PHARMACEUTICAL INDUSTRY (10+1) h

Solvent categories in pharmaceutical process development and greenness factor.

Supercritical fluids and applications.

Water as solvent: under pressure enabling reactions at high temperature, in ring closure reactions under PTC conditions, dehydrohalogenation under PTC conditions.

Solvent free reactions: ex; Biginelli reaction.

Case studies: (i) Convergent synthesis of Sildenafil citrate (ii) Comparison of old and new commercial synthesis of sertraline HCl (use of green solvent) (iii) Use of biocatalyst to replace Cr based catalyst in the synthesis of LY 300164 (iv) Improved ecological footprint in the synthesis of Celecoxib (v) Quinaprin synthesis avoiding the use of potentially explosive hydroxybenzotriazole

Green technologies in generic pharmaceutical industry: Current vs greener method, ex; bromination (Reddy's lab).

9. FLOW CHEMISTRY (3 + 2) h

Introduction: Batch vs flow operations, flow reactor, types of reactors. Meaning of residence time and molar flow rate.

Mass transfer: mixing rate vs reaction rate, Damkohler number, manipulation of Damkohler number: e.g. synthesis of Verubecestat.

Advantages of flow chemistry: Outpacing intramolecular reactions, e.g. Fries rearrangement. Practical applications: Fischer esterification using in-line GC analysis.

Swern-Moffatt oxidation.

Handling hazardous reagents, ex; diazomethane, phosgene.

Limitations of flow chemistry

References:

1. Green Chemistry Twelve Principles, K R Desai, Bhuvanaben D Mistry, Tarulata N Chhowala. Himalaya publishing House, 2018
2. Organic synthesis Special techniques, V K Ahluwalia, Renu Aggarwal. Norosa publishing house, New Delhi, 2006.
3. Green Techniques for Organic Synthesis, Wei Zhang, Berkeley W. Cue Jr. Wiley, 2012.

4. Green Chemistry in Pharmaceutical Industry, Peter J. Dunn, Andrew S. Wells and Michael T. Williams. WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim, 2010.
5. Supercritical Carbon Dioxide in Polymer Reaction Engineering, Kemmere, M. F., Meyer, T. (eds.) 2005, ISBN: 978-3-527-31092-0
6. Green synthesis interventions of pharmaceutical industries for sustainable development, Mohit Mishra, Mansi Sharma, Ragini Dubey, Pooja Kumari, Vikas Ranjan, Jaya Pandey. Current Research in sustainable Chemistry. 4 (2021) 100174.
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11. Burstein C, Glorius F. Organocatalyzed conjugate umpolung of α, β-unsaturated aldehydes for the synthesis of γ-butyrolactones. Angew Chem Int Ed 2004, 43, 6205–6208.
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17. Advanced Organic Chemistry, Part-B, F. A. Carey and R. J. Sundberg, 4th edn, Springer international edn, 2001. 7. Green chemistry: Environmentally friendly alternatives, R. Sanghi and M. M Srivastava, Norosa, New Delhi, 2003. 8. Green Chemistry-an introduction text, The Royal Society of Chemistry.

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Code number and Title of the paper: CHDE 0321:

Green Chemistry and diversity of its applications

Chapter Number	Title	Number of teaching hours (As mentioned in the syllabus)	Maximum marks for which questions are to be framed from this chapter (including bonus questions)
1.	Principles of green chemistry	3	5
2.	Use of ultrasound and microwaves in organic synthesis	5	9
3.	Mechanochemistry	5	9
4.	Polymer supported reagents in organic synthesis	6	10
5.	Phase transfer catalysis (ptc) and crown ethers	7	12
6.	Multicomponent one-pot reactions	5	8
7.	Organocatalysis	13	22
8.	Green chemistry practices in pharmaceutical industry	11	19
9.	Flow chemistry	5	9
Total marks excluding bonus questions			70
Total marks including bonus questions			103

Semester	IV
Paper code	CHDE 0421
Paper title	Dept. elective: FORENSIC CHEMISTRY
Number of teaching hours per week	4
Total number of teaching hours per semester	60
Number of credits	4

1. INTRODUCTION TO FORENSIC SCIENCE 11+2 hrs

Definition, historical aspects, scope, code of conduct of forensic science. Crime Scene-types-indoor and outdoor. Securing and isolating the crime scene. Crime scene search methods.

Case study - Amanda Knox: A Flawed Case of Murder

Legal aspects of crime- Role of Investigator.

Case study - Dr. Coppolino's Deadly House Calls

Classification of crime scene evidence – physical and trace evidence. Collection, labeling, sealing of evidence.

Case study - Bruce McArthur: A Mountain of Physical Evidence.

Criminal Profiling -Profile of victim and culprit, its role in crime investigation, Lie detection (Polygraphy), Narco analysis, Brain mapping.

2. FINGERPRINTS 9 hrs

Introduction- Basics of fingerprinting, Types of fingerprints. Fingerprint patterns. Development of Fingerprints- Latent prints. Latent fingerprints' detection by physical and chemical techniques.

Case study - Killer Twin: Ronald and Donald Smith

Case study - The Mayfield Affair

3. FORESNIC TOXICOLOGY 8 hrs

Significance of toxicological findings. Techniques used in toxicology. Toxicological analysis – detection alcohol in blood sample, chemical intoxication tests - breath testing for alcohol. Human performance toxicology.

Case study-Accidental overdose: The Tragedy of Michael Jackson and Mac Miller.

4. ANALYTICAL METHODS IN FORENSIC CHEMISTRY 19+1 hrs

Sample preparation for chromatographic and spectroscopic techniques. Chromatographic methods - forensic applications of thin layer chromatography, gas chromatography and liquid chromatography. Spectroscopic methods - forensic applications of ultraviolet-visible spectroscopy, infrared spectroscopy, atomic absorption spectroscopy, atomic emission spectroscopy. Mass spectrometry. X-ray diffraction. Colorimetric analysis of narcotics. Electrophoresis –forensic applications. Forensic photography- Basic principles and applications of photography in forensic science. 3D photography- Infrared and ultraviolet photography. *Digital photography. Videography.*

5. NANOTECHNOLOGY IN FORENSIC CHEMISTRY 10 hrs

Nanomaterials-Classification. Synthesis of nanomaterials-top-down and bottom-up synthesis - CVD. Application of nanotechnology in forensic evidence analysis- Collection and analysis of evidence of different types of crime scenes including explosive, drugs, DNA analysis, latent finger-marks.

References

1. W.J. Tilstone, M.L. Hastrup and C. Hald, Fisher's, *Techniques of Crime Scene Investigation*, CRC Press, Boca Raton (2013).
2. R. Saferstein, *Criminalistics: An Introduction to Forensic Science*, 13th Edition, Pearson Education, (2021).
3. M. Byrd, *Crime Scene Evidence: A Guide to the Recovery and Collection of Physical Evidence*, CRC Press, Boca Raton (2001).
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12. Fundamentals of Molecular Spectroscopy by Colin N Banwell and Leaine McCash, Fourth Edition-2017, McGraw Hill Education Pvt. Ltd.

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Code number and Title of the paper: CHDE 0421: Forensic Chemistry

Chapter Number	Title	Number of teaching hours (As mentioned in the syllabus)	Maximum marks for which questions are to be framed from this chapter (including bonus questions)
1.	Introduction to forensic science	13	24
2.	Fingerprints	9	15
3.	Forensic toxicology	8	12
4.	Analytical methods in forensic chemistry	20	34
5.	Nanotechnology in forensic chemistry	10	18
Total marks excluding bonus questions			70
Total marks including bonus questions			103

Semester	IV
Paper code	CHDE 0521
Paper title	Dept. elective: SUPRAMOLECULAR CHEMISTRY
Number of teaching hours per week	4
Total number of teaching hours per semester	60
Number of credits	4

1. INTRODUCTION TO SUPRAMOLECULAR CHEMISTRY (6 + 2) h

Definition and development of supramolecular chemistry- lock and key analogy**,** cooperativity-pre-organisation-complementarity-thermodynamic, kinetic selectivity-nature of supramolecular interactions- solvation effects, supramolecular concepts and design. Host-guest chemistry. Synthesis: The template effect and high dilution.

Lariat ethers, podands, cyclodextrins cyclophanes, cryptophanes, carcerands, hemicarcerands. Anion binding: Concepts in anion host design, different types of anion hosts. Simultaneous cation and anion binding. Cation-binding: crown ethers, cryptands, spherands, calixarenes (review-recall),

2. NATURE OF SUPRAMOLECULAR INTERACTION 3 h

Ion-ion interactions, ion-dipole interaction, dipole-dipole interaction, hydrogen bonds, hydrophobic interactions.

3. CRYSTAL ENGINEERING 10 h

Self-assembling capsules, molecular containers, metal directed capsules, hydrogen bonded capsules, concepts in crystal engineering, The Cambridge structural database, crystal engineering with hydrogen bonds, pi interactions - halogen bonding and other weak interactions, co-crystal, salts, polymorphs and their physico-chemical properties, coordination polymers. Solid state reactivity: metal-organic frameworks, guest properties of metal-organic frameworks.

4. SOLID STATE SUPRAMOLECULAR CHEMISTRY 8 h

Zeolites: structure, composition and catalysis. Clathrates: urea/thiourea clathrates, trimesic acid clathrates, clathrate hydrates (structure and function of the above species), uses. Inclusion compounds, intercalation compounds.

5. SELF-ASSEMBLY 12 h

Self-assembly in synthetic systems: pi-electron donor-acceptor systems, transition metal directed assemblies, hydrogen bond assemblies, anion directed assemblies, catenanes, rotaxanes, helicates, helical assemblies and molecular knots.

Guest binding by cavitands - calixarenes, resorcarenes, glycourils, cyclodexdrins; molecular clefts, tweezers, cyclophanes, cryptophanes, carcerends and hemicarcerends.

Molecular devices: Photo-switchable devices. Applications of supramolecular chemistry in sensors, switches and molecular machinery and molecular biology.

6. BIOLOGICAL MIMICS AND SUPRAMOLECULAR CATALYSIS 3 h

Characteristics of biological models. Supramolecular catalysis: cyclodexdrin as enzyme mimics.

7. SURFACTANTS AND INTERFACIAL ORDERING 3 h

Micelles and vesicles, surface self-assembled monolayers**.** Application to medicinal chemistry**.** Soft lithography, microlens arrays, transfer printing.

8. DENDRIMERS 5 h

Synthesis - divergent and convergent methods, host-guest chemistry of dendrimers. Supramolecular dendrimer assemblies. Applications of dendrimer for drug delivery.

9. NANOMATERIALS WITH SUPRAMOLECULAR STRUCTURE 8 h

Nanorod, nanowire self-Assembly: metal templating nanowires. Self-assembling nanorods. nanorod devices – nanotubes from nano porous templates. VLS synthesis of nanowires, nanowire quantum size effects. Manipulating nanowires, nanowire sensors.

Nanocluster self-assembly: synthesis of metal capped semiconductor nanoclusters, electrons and holes in nanocluster boxes, nanocrystal semiconductor alloys, nanocluster phase transition water soluble nanoclusters. Polymer nanocomposites.

REFERENCES:

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Code number and Title of the paper: **CH DE 0521:** Supramolecular Chemistry

Chapter Number	**Title**	**Number of teaching hours (As mentioned in the syllabus)**	**Maximum marks for which questions are to be framed from this chapter (including bonus questions)**
1.	**Introduction to supramolecular chemistry**	8	13
2.	**Nature of Supramolecular Interaction**	3	6
3.	**Crystal Engineering**	10	17
4.	**Solid State Supramolecular Chemistry**	8	12
5.	**Self-Assembly**	12	20
6.	**Biological Mimics and Supramolecular Catalysis**	3	6
7.	**Surfactants and Interfacial Ordering**	3	6
8.	**Dendrimers**	5	10
9.	**Nanomaterials with supramolecular structure**	8	12
Total marks excluding bonus questions			70
Total marks including bonus questions			103