MANONMANIAM SUNDARANAR UNIVERSITY, TIRUNELVELI-12
DEPARTMENT OF CHEMISTRY
M. Phil CHEMISTRY (CBCS) COURSE (1 Year)

For the academic year 2018-2019

Preamble

M.Phil is a research oriented program. After completing their Master degree in Chemistry or equivalent will opt for pursuing research either directly or after completing the above program. The program is useful for research students to evaluate and identify the research problems which is related to social and economical valuable issues to the society.

Necessity

It is a platform to learn the basic research methodology and it is easy to identify the current research trends in chemistry.

Importance

This programme helps the students to carry out the literature survey. Based on the literature survey, the students identify the need of research, related to social and industrial needs.

Objectives

After studying the M.Phil. programme, the students will be able to

i. Introduce the purpose and importance of research for future development.
ii. Know the different types of literature search and indexes.
iii. Understand the error analysis, correlation methods and computer application.
iv. Enrich the knowledge in various types of spectral techniques and scientific analysis.
v. Develop their skills for carryout the project.
vi. Make awareness in social and industrial relevant issues.
vii. Expose to present their findings in national and international seminars and conferences.

Outcome

After completing the M.Phil programme, the students will be able to

i. Pursue research programme.
ii. Qualify as Chemist/Scientist in various industries and research institutions

Eligibility Norms:

55% of marks in M.Sc. degree in Chemistry or any other equivalent Master Degree. For SC/ST candidates there will be 5% relaxation in marks.

Admission Procedure:

Admission will be based on (i) the total marks obtained in the entrance test (50%) and the qualifying M.Sc. degree examination (50%) (merit list for a Total of 100 marks) and (ii) by following the govt. norms of reservation.

STRUCTURE & SYLLABUS OF THE COURSE

Semester	Papers	Teaching / contact hours per week	Exam hours	Marks	Credits
Semester I					
Paper I	Research Methodology	8	3	100	8
Paper II	Advanced Paper (Course Work)	8	3	100	8
Paper III	ELECTIVE PAPER - Project oriented **(Any one paper is to be selected by the student out of 5 offered)**	8	3	100	8
Semester II					
Paper IV	**Project & Viva voce**	12	Viva voce	200	16
			Total Marks & Credits	500	40

Evaluation:

The evaluation for Papers I, II, III & IV consists of two components viz. internal and external.

Internal : External = 25 : 75

25 marks for the internal component has been divided as follows:

3 tests, out of which average of the best two tests : 15 marks

Seminar : 5 marks

Assignment : 5 marks

There is no internal passing minimum. There is a passing minimum of 50% for external and overall components.

Question Paper Pattern

For Papers I, II & III : (Max : 75 marks)

Question paper consists of Section A 5 questions each from one unit (5 x 5 = 25 marks) and Section B 5 questions each from one unit (5 x 10 = 50 marks) with internal choice (either a OR b) in each unit. For Paper III the 10 research publications shall be divided equally into 5 units.

Paper IV – Project

The project shall be of one semester duration and is an individual student research project. Each M.Phil student will be allotted to a teaching staff as guide and the student has to do research work under the supervision and guidance of the allotted teaching staff. At the end of the semester the student has to submit the dissertation of the project work which shall be evaluated as follows. The project report evaluation and Viva voce examination will be conducted jointly by the guide and an EXTERNAL EXAMINER.

Maximum mark for project is 200. The break up figures are as follows:

Project report : 150 marks (Internal 75 + External 75)

Viva voce : 50 marks (Internal 25 + External 25)

200 marks

Paper I – Research Methodology

Unit I

Introduction to research, selection of a research topic, reviewing the literature, preparing the proposal and design of study. Experimentation and interpretation of results. Formation, testing and rejection of hypothesis. Application of microcal origin and chemdraw. Preparation and presentation of report; dissertation and thesis writing.

Primary and secondary literature: Journals, patents, Reviews, Chemical abstracts, treatises and monographs. Printed materials and online literature search; websites, search engine for locating information and chemical data bases. E-mail operation and online submission of manuscripts for publication.

Unit II

Limitations of analytical methods; accuracy, precision and minimization of errors. Systematic and random errors and reliability of results. Replicate determination and t-test. Correlation, linear regression and analysis of variance.

Unit III

Principles, sampling techniques and application of UV VIS spectrophotometry, far, near and FTIR spectrophotometry and ICP spectrometry. Thermo analytical techniques: TGA, DTA, DSC and thermometric titrations. Magnetic susceptibility and EPR spectroscopy measurements and characterization of samples.

Unit IV

Chromatographic methods of analysis – column, paper, thin layer, gas, ion exchange, gel permeation, VPC and HPLC

Reagents in Organic Synthesis:

Gilman's reagents – DCC – Girard reagents – NBS – crown ethers – NBS – BF_3 complexes – SeO_2 – 1,3-dithiane, tri-n-butyltin hydride – phase transfer catalysts – Wilkinson's catalyst.

Unit - V

Teaching – Objectives of Teaching. Phases of Teaching- Teaching Methods: Lecture method, Discussion Method, Discovery Learning, Inquiry, Problem Solving Method, project Method, Seminar- Integrating ICT in Teaching: Individualised Instruction, Ways for Effective Presentation with Power Point – Documentation- Evaluation: Formativem Summative & Continuous and Comprehensive Evaluation- Later Adolescent Psychology: Meaning, Physical, Cognitive, Emotional, Social and Moral Development- Teaching Later Adolescents

References:

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4. Jerry March, Advanced Organic Chemistry, 4th Edn. John Wiley & Sons, 1992.
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7. Chromatographic Methods – R. Stock and B.R. Rice (Chapman & Hall 1974)
8. Reaction Mechanism and Reagents in Organic Chemistry – Gurdeep R. Chatwal
9. A.J. Bard and L.R. Faulkner, Electrochemical Methods : Fundamentals and Applications, 2nd Edn., John Wiley and Sons, New York, 2004.
10. L. Antropov, Theoretical Electrochemistry, Mir Publication, Moscow, 1972.
11. D.A. Skoog and J.J. Leary, Principles of Instrumental Analysis, 4th Edn., Saunders College Publishing, 1992.
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14. G. E. Bacon, Neutron diffraction, Oxford Universtiy Press, Oxford, 1975.
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18. Vedanayagam, E.G. (1989), Teaching technology for college teachers, New York: Sterling Publishers.

Paper II – Advanced Paper (Course work)

Unit I: Retrosynthetic Analysis

Introduction to disconnections – one group disconnections – two group disconnections – pericyclic reactions – Heteroatoms and heterocyclic compounds – small rings: three membered, four membered, and five membered.

Unit II: Applications of Advanced Organic Spectroscopy

NMR: Basic principles of two-dimensional NMR spectroscopy – HOMOCOSY, HETCOSY and NOESY spectra and their applications – use of INEPT and DEPT methods and their applications.

Mass: Molecular ions, isotope peaks, fragmentation pattern – McLafferty rearrangement - measurement techniques (EI, CI FI, FD, FAB, SIMS, MALDI) – M + 1 and M + 2 ions – calculation of molecular formula from P_{M+1} and P_{M+2}

Road-map problems covering UV, IR, ^{1}H-NMR, ^{13}C-NMR and mass spectral data.

Unit III: Advanced Inorganic Chemistry

Mixed ligand complexes – Stabilities dynamics of formation and dissociation of ternery complexes. Reactions of coordinated ligands mimicking biological systems.

Isomerization and racemization reactions of bidentate mixed ligand and tris chelate complexes of unsymmetrical ligands; application of ORD & CD for study of such reactions. Atom (or group) transfer processes.

Enzymes in redox catalysis: NAD, ascorbate oxidase, peroxidases and catalases, superoxide dismutase and cysteine – cystine system. Neurotransmitters and related hormones.

Supramolecular Chemistry: Cation binding hosts of crown ethers, cryptands and spherands and their metal ion selectivity.

Unit IV: Advances in Linear Free-Energy Relationships

An introduction to linear free-energy relationships (LFER) – the Hammett equation – the duality of substituent constants and the Yukawa-Tsuno equation – the general validity of the Hammett equation – deviations from the Hammett equation in its various forms; the separation of polar, steric and resonance effects – Taft's equations; the ortho-effect; application of LFER to organic reactions; Influence of solvent on organic reactivity; the reactivity-selectivity principle.

Unit V: Advanced Photochemistry

Artificial photosynthesis and solar energy conversion – Photoelectrochemical cells – dynamics of excited state processes (excited state energy, redox properties, emission lifetime and its temperature dependence) in micelles, reverse micelles and biomembranes – Fluorescence – quenching and anisotropy concepts; fluorescence sensing – mechanism and applications; Radioactive decay engineering – metal-enhanced fluorescence and surface plasmon-coupled emission.

References:

1. Designing Organic Synthesis: A Programmed Introduction to the Synthon Approach – Stuart Warren
2. Spectrometric Identification of Organic Comounds – Silverstein, Bassler and Morril.
3. Organic Spectroscopy – William Kemp
4. Basic and Two-dimensional NMR Spectroscopy – V. Fibrolein, VCH, 1982.
5. Principles of Instrumental Analysis – S. Skoog, Holler and Nieman, Saunders, 1998.
6. Practical Spectroscopy – P.R. Young – Brooks / Cole, 2000.
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11. S.J. Lippard and J.M. Berg, Principles of Bioinorganic Chemistry, University Science Books, California, 1994.
12. Bertini, Gray, Lippard, Valentine, Bioinorganic Chemistry, Viva Books Pvt. Ltd., New Delhi, 1998.
13. N.B. Chapman and J. Shorter, Eds., Advances in Linear Free-Energy Relationships, Plenum Press, London, 1972.
14. J. Shorter, Correlation Analysis in Organic Chemistry – An Introduction to Linear Free-Energy Relationships, Clarendon Press, Oxford, 1973.
15. N.B. Chapman and J. Shorter, Eds., Correlation Analysis in Chemistry-Recent Advances, Plenum Press, New York, 1978.
16. J. Shorter, Correlation Analysis of Organic Reactivity, Research Studies Press, England, 1982.
17. A. Juris, V. Balzani, F. Barigelletti, S. Campagna, P. Belser, A. Von Zelewsky, Coordination Chemistry Reviews, 84, 1988, pp. 85-227.
18. J.R. Lakowicz, Principles of Fluorescence Spectroscopy, Plenum Press, New York, 2006.
19. K. Kalyanasundaram, Photochemistry in Microheterogeneous Systems, Academic Press, Orlando, 1987.

Paper III Elective Papers

Elective Paper I HETEROGENEOUS CATALYSIS

Unit I

Zeolite-based photocatalysts - Zeolites and molecular sieves acting as hosts for photoactive guests - Electron donor photosensitisers - organic dye - electron acceptor photosensitisers - Zeolites encapsulating clusters of semiconductor oxides - Zeolites having photocatalytically active framework.

Efficient photocatalytic degradation of organics diluted in water and air using TiO2 designed with zeolites and mesoporous silica materials.

Unit II

Effect of metal-doping of TiO_2 nanoparticles on their photocatalytic activities toward removal of organic dyes.

Solar photocatalytic degradation of phenol using nanosized ZnO and α-Fe_2O_3.

Unit III

Network Structured SnO_2/ZnO Heterojunction Nanocatalyst with High Photocatalytic Activity.

Green synthesis of copper nanoparticles for the efficient removal (degradation) of dye from aqueous phase.

Unit IV

Visible Light Photodegradation of Phenol Using Nanoscale TiO_2 and ZnO Impregnated with Merbromin Dye: A Mechanistic Investigation.

Fe(III)/TiO_2-Montmorillonite Photocatalyst in Photo-Fenton-Like Degradation of Methylene Blue.

Unit V

TiO_2 nanoparticles immobilized on carbon nanotubes for enhanced visible-light photo-induced activity.

Preparation of a Titania/X-Zeolite/Porous Glass Composite Photocatalyst Using Hydrothermal and Drop Coating Processes.

References

1. C h e m . C o m m u n . , 2 0 0 4 , 1 4 4 3 – 1 4 5 9.
2. J. Mater. Chem., **2011**, 21, 2407–2416 | 2407
3. Egyptian Journal of Petroleum (**2014)** 23, 419–426
4. Journal of Chemical Engineering and Materials Science**,** Vol. 4(7), pp. 87-92, November **2013**
5. Inorganic Chemistry**,** Vol. 48, No. 5, **2009 1819-1825**
6. ENVIRONMENTAL SCIENCE AND POLLUTION RESEARCH · AUGUST **2015**, DOI 10.1007/s11356-015-5223-y
7. Iran. J. Chem. Chem. Eng, Vol. 33, No. 2, **2014**
8. International Journal of Chemical Engineering, Volume **2015**, Article ID 485463,
9. J. M a t e r . R e s. T e c h n o l . **2 0 1 5;4(2)**:126–132
10. Molecules **2015**, *20*, 2349-2363; doi:10.3390/molecules20022349.

Elective Paper II – **Adsorption and Catalysis**

ADSORPTION AND CATALYSIS

Unit I

Concept of adsorption – types of adsorption, monolayer and multilayer adsorption. Adsorption - activation energy and temperature relationships, different between adsorption and catalysis, catalysis - homogeneous catalysis, heterogeneous catalysis, Acid -- base catalysis

Unit II

Adsorbent - adsorbent preparation from plant materials, activated carbon preparation, synthetic adsorbent/catalyst - Molecular sieves – microporous & mesoporous molecular sieves – silicates, Aluminosilicates, Aluminophosphates – structure, acidic and basic sites.

Unit III

Characterization of adsorbent and catalyst - X-Ray Diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), Differential thermal analysis(DTA) , Nuclear magnetic resonance spectroscopy (NMR), Temperature programmed desorption (TPD), Electron spin resonance spectroscopy(ESR) Scanning electron microscopy(SEM), BET Surface Area, pore size analysis.

Unit IV

Liquid phase - heterogeneous reaction conditions optimization - Temperature, pH, time and molar ratios. Vapor phase reaction, Regeneration of catalyst.

Adsorption – adsorption of dye molecules, metal ions, sugar molecules and other suitable molecules, conditions optimization – time, temperature, PH, concentration and adsorbent dosage.

Unit V

Product analysis in catalysis reactions – Gas chromatographic technique, conversion and product selectivity

Interpretation of adsorption parameters - Adsorption kinetics, adsorption isotherms and adsorption thermodynamics

References

1. Environmentally stable adsorbent of tetrahedral silica and non tetrahedral alumina for removal and recovery of malachite green dye from aqueous solution, *J.Hazardous materials*, 157 (2008) 137-145.
2. Plant poisoning organic dyes adsorption on tomato plant root and green carbon from aqueous solution, *Desalination*, 249 (2009)1132-1138.
3. Film and pore diffusion modeling for the adsorption of direct red 81 on activated carbon prepared from balsamodendron caudatum wood waste, *Digest Journal of Nanomaterials and Biostructures*, Vol. 5, No 3, July 2010, p. 911 – 919
4. Plant toxic and non-toxic nature of organic dyes through adsorption mechanism on cellulose surface, *Journal of Hazardous materials*,189 (2011) 294–300.
5. Adsorption of cationic and anionic organic dyes from aqueous solution using Silica, *J. Environmental Science and Engineering*, volume 52, No.4 **(2010)** 361-366
6. Hazardous dyes removal from aqueous solution over mesoporous aluminophosphate molecular sieves with textural porosity by adsorption, Journal of Hazardous Materials 244– 245 (**2013**) 10– 20.
7. A Simple Method for the Synthesis of Thermally Stable Large Pore Mesoporous Aluminophosphate Molecular Sieves, Materials letters, 113 (**2013**) 93–95.
8. Aniline methylation over AFI and AEL type molecular sieves, *App. Catal*., Vol. 174, **1998**, 213.
9. Adsorptive removal of metanyl yellow on mesoporous Nickel aluminophosphate molecular sieves from aqueous solution, Asian J. of chemistry, vol. 24, no.12(2012), 5775-5778
10. Recent trends in catalysis**,** Narosa publication, 1st edition 2000.

Elective Paper III - **Nanomaterials and their applications to solar energy conversion**

Unit I Nanomaterials

Introduction to Nanoscience: Introduction- definition of nanoscience, nanochemistry- classification of the nanomaterials

Synthesis of nanomaterials: Precipitative methods – hydrothermal and solvothermal methods - chemical methods - reduction methods – colloidal and micellar approach – sol-gel method – chemical vapor deposition method.

Specialized Nanomaterials: Metal oxide nanoparticles, semiconductor nanoparticles and core/shell nanoparticles

Unit II Dye-sensitized solar cells

Solar energy conversion and storage – photoelectrochemical cells – dye-sensitized solar cells – design and fabrication - power conversion efficiency

Use of metal and metal-free dye sensitizers in photovoltaic devices.

Unit III

Review of published literature – Water-soluble silica-coated semiconductor quantum dots – synthesis, characterization and properties.

Thickness-controllable silica coating of quantum dots – synthesis by microemulsion method and application in the growth of rice.

Unit IV

Review of published literature – Silica coated cadmium sulfide nanocomposites – synthesis, structure, optic and its photocatalytic properties.

Zirconia-coated carbonyl iron particles – synthesis and corrosion study.

Unit V

Review of published literature – Ruthenium(II) sensitizer in dye-sensitized solar cells using an organic dye as cosensitizer – Fabrication and device characterization - photovoltaic performance.

Dye-sensitized solar cells - Co-sensitization strategy – electrochemical properties – Photoelectrochemical performances – Electrochemical impedance spectroscopy – dark current measurement – Open-circuit voltage decay.

References

1. H. R. Allcock, Introduction to Materials Chemistry, John Wiley & Sons, Inc. Publication, 2008.
2. T. Pradeep, Nano: The Essentials, Tata Mc Graw-Hill, 2007.
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4. J. Gong, J. Liang, K. Sumathy, Renewable and Sustainable Energy Reviews, 2012, 16, 8, 5848-5860.
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6. A. Wang, Y. Zheng, F. Peng, J. Spectros. 2014, Article ID 169245, 1-5.
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<u>Elective Paper IV</u> - Phyto-biosynthesis and applications of Metal nanoparticles

Unit I - Extraction and Isolation of some Indian Medicinal plants

i) Solid-Phase Extraction and LC−MS analysis of Pyrrolizidine Alkaloids in Honeys.

ii) Comparative study of phytochemical screening, antioxidant and antimicrobial capacities of fresh and dry leaves crude plant extracts of *Datura metel* L.

Unit II – Biosynthesis of Metal Nanoparticles

i) Green synthesis of silver nanoparticles using *Ixora coccinea* leaves extract.

ii) Ultrasmall Copper Nanoparticles Synthesized with a Plant Tea Reducing Agent.

Unit III – Characterization of Nanoparticles

i) Phytosynthesis of silver nanoparticles using *Coccinia grandis* leaf extract and its application in the photocatalytic degradation.

ii) A facile synthesis of high optical quality silver nanoparticles by ascorbic acid reduction in reverse micelles at room temperature.

Unit IV – Biological Applications of Nanoparticles

i) The green synthesis, characterization and evaluation of the biological activities of silver nanoparticles synthesized from *Iresine herbstii* leaf aqueous extracts

ii) In vitro evaluation of antioxidant and anticancer potential of *Morinda pubescens* synthesized silver nanoparticles.

Unit V – Green catalytic activity of Nanoparticles

i) Catalytic Reduction of 4-Nitrophenol using Biogenic Gold and Silver Nanoparticles Derived from *Breynia rhamnoides*.

ii) Catalytic degradation of organic dyes using biosynthesized silver nanoparticles.

References

1. K. A. Beales, K. Betteridge, S.M. Colegate, J.A. Edgar. Journal of Agric. Food Chem. 2015, 63, 7421−7427
2. Tahiya Hilal Ali Alabri, Amira Hamood Salim Al Musalami, Mohammad Amzad Hossain, Afaf Mohammed Weli, Qasim Al-Riyami. Journal of King Saud University – Science 2014, 26, 237–243
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4. Aaron D. Brumbaugh, Katelyn A. Cohen, and Sarah K. St. Angelo. ACS Sustainable Chem. Eng. 2014, 2, 1933−1939.
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6. Debabrata Singha, Nabajeet Barman, Kalyanasis Sahu. Journal of Colloid and Interface Science 413 (2014) 37–42.
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Elective Paper V – **Biomaterials and Corrosion**

BIOMATERIALS AND CORROSION

UNIT I - Basic biomaterial and biomedical device knowledge, to identify the material properties that are critical for metallic, polymer and ceramic biomaterials, or their combination.

UNIT II - Application areas for different types of biomaterials, biomedical deivices and their failure analysis

UNIT III - Basic properties of physical, chemical and mechanical and coating processes that may occur on biomaterials in use.

UNIT IV - Analysis and evaluation of corrosion degradation reactions that occur for different biomaterials and their consequences.

UNIT V - Suggest proper type of biomaterial for given applications, taking into account function, health risk and economic aspects.

References:

1. Monika Saini, Yashpal Singh, Pooja Arora, Vipin Arora, and Krati Jain Implant biomaterials: A comprehensive review World J Clin Cases. 2015 Jan 16; 3(1): 52–57.
2. Geetha Manivasagam , Durgalakshmi Dhinasekaran and Asokamani Rajamanickam Biomedical Implants: Corrosion and its Prevention - A Review Recent Patents on Corrosion Science, 2010, 2, 40-54
3. Patterson, S.P.; Daffner, R.H.; Gallo, R.A. Electrochemical corrosion of metal implants. AJR. Am. J. Roentgenol. 2005, 184, 1219-1222.
4. A. Balamurugan, S. Rajeswari, G. Balossier, A. H. S. Rebelo and J. M. F. Ferreira Corrosion aspects of metallic implants — An overview Materials and Corrosion 2008, 59, No. 11 855-869
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