SYLLABUS

1. Information regarding the programme

1.1 Higher education institution	Babeş-Bolyai University, Cluj-Napoca
1.2 Faculty	Chemistry and Chemical Engineering
1.3 Department	Chemical Engineering
1.4 Field of study	Chemical Engineering
1.5 Study cycle	Master
1.6 Study programme / Qualification	ICAP / master degree

2. Information regarding the discipline

2.1 Name of the discipline	Structural characterization methods for materials and precursors – CMX7142						
2.2 Course coordinator	Prof. dr. Ion Grosu Assoc. Prof. dr. Radu Silaghi-Dumitrescu Lecturer dr. Gabriela Nemeş Lecturer dr. Richard Varga Prof. dr. Liana Mureşan Assoc. Prof. dr. ing. Sanda Andrada Măicăneanu						
2.3 Seminar coordinator	Prof. dr. Ion Grosu Assoc. Prof.dr. Radu Silaghi-Dumitrescu Lector dr. Gabriela Nemeş Lector dr. Richard Varga Prof. dr. Liana Mureşan Assoc. Prof. dr. ing. Sanda Andrada Măicăneanu						
2.4. Year of study	I	2.5 Semester	2	2.6. Type of evaluation	C	2.7 Type of discipline	Compulsory

3. Total estimated time (hours/semester of didactic activities)

3.1 Hours per week	3	Of which: 3.2 course	2	3.3 seminar/laboratory	1
3.4 Total hours in the curriculum	42	Of which: 3.5 course	28	3.6 seminar/laboratory	14

Time allotment:	hours
Learning using manual, course support, bibliography, course notes	30
Additional documentation (in libraries, on electronic platforms, field documentation)	28
Preparation for seminars/labs, homework, papers, portfolios and essays	42
Tutorship	5
Evaluations	3
Other activities:	-

3.7 Total individual study hours	108
3.8 Total hours per semester	150
3.9 Number of ECTS credits	6

4. Prerequisites (if necessary)

4.1. curriculum	• none
4.2. competencies	• none

5. Conditions (if necessary)

5.1. for the course	• Students will be present at courses with their mobile phones turned off • Late presence at courses will not be accepted
5.2. for the seminar /lab activities	• Students will be present at seminaries/labs with their mobile phones turned off • Homework solving is requested for the next meeting • A penalty of 0.5 points /day will be applied if the papers (essays) are not submitted as requested

6. Specific competencies acquired

Professional competencies	• Use the advanced concepts of analysis and synthesis of processes, and equipment specific to process engineering • Creative usage of analysis and synthesis of specific knowledge, methods and concepts in development of new chemical processes • Integrated usage of analysis and synthesis of chemical processes to develop innovative processes and the production of innovative products • Application of modern evaluation techniques to new systems in order to improve the decision making processes in process synthesis • Creative use of analysis and synthesis in developing innovative products / technologies • Ability to understand and interpret additional data to characterize materials, to express and argue the interpretation of data based on the correlation results and comparison with data from the literature • Ability to understand the techniques, to use and interpret information obtained using spectroscopic methods of chemical compounds structure investigation, nuclear magnetic resonance and mass spectrometry • Ability to identify samples where analysis of ESR method is applicable, and to extract useful information on the identity and properties of materials • Ability to identify issues where analysis of UV-VIS method is applicable, and to extract useful information on the identity and properties of materials • Description and interpretation of the methods and techniques used to determine the structure and properties of chemical compounds, processing and interpretation of the results obtained from X-ray diffraction analysis • Ability to explain and interpret electrochemical principles and methods used in structural characterization of materials • Familiarity with BET method and its variants for determination of the specific surface area and pore distribution; ability to understand the structure-property relationship (examples for catalytic materials), by correlating SEM and BET results
Transversal competencies	• Execution of the tasks required under specified requirements and the deadlines imposed, in compliance with professional ethics and moral conduct, following a predetermined work plan • Solving tasks in accordance with the general objectives established by integrating into a working group • Permanent information and documentation in its field, in Romanian • Seeking to improve working results by engaging in professional activities • Ability to prepare written reports and to make these reports public

7. Objectives of the discipline (outcome of the acquired competencies)

7.1 General objective of the discipline	• Acquiring notions of structural characterization of materials and precursors using specific techniques
7.2 Specific objective of the discipline	• Acquiring theoretical knowledge on the use of nuclear magnetic resonance method for investigating the structure of chemical compounds • Acquiring theoretical knowledge on the use of mass spectrometry to investigate and identify the structure of chemical compounds • Acquiring knowledge and practical skills required to implement notionally ESR spectroscopy in the study of materials and precursors • Acquiring the necessary theoretical knowledge and their application in UV-VIS techniques • Acquisition of basic theoretical knowledge on single-crystal X-ray diffraction as investigative method of the crystalline structure • Acquiring basic theoretical knowledge on the main electrochemical methods for investigating structure of materials • Acquiring notions of surface characterization by gas adsorption method and electron microscopy.

8. Content

8.1 Course	Teaching methods	Remarks
MODULE 1 Spectroscopic methods for structural characterization of organics and organometallic compound		
8.1.1. Structural characterization by NMR – principles (2 h)	Lecture; Explanation; Conversation; Description	6 hours
8.1.2. NMR method – data interpretation, applications (2 h)		
8.1.3. Structural characterization by mass spectrometry (2 h)		
8.1.4. ESR spectroscopy (electronic spin resonance spin) – principle, application domains (2 h)	Lecture; Explanation; Conversation; Description	4 hours
8.1.5. ESR spectroscopy – experiments for material analysis, biomedical applications (2 h)		
8.1.6. UV-VIS spectroscopy, theoretical aspects and principles. UV-VIS spectroscopy utilization for molecular structure identification (2 h)	Lecture; Explanation; Conversation; Description	4 hours
8.1.7. UV-VIS spectra analysis (2 h)		
MODULE 2 XRD Structural characterization	Lecture; Explanation; Conversation; Description	4 hours
8.1.8. X-ray diffraction. Experimental methods. X-ray diffraction as analytical method, single-crystal, symmetry. (2 h).		
8.1.9. Interpretation of data obtained by single-crystal X-ray diffraction. Description and interpretation of Cif file, atomic parameters, crystallographic tables, intra-and intermolecular interactions, hydrogen bonds, covalent radii, Van der Waals radii. (2 h).		
MODULE 3. Structural characterization by electrochemical methods	Lecture; Explanation; Conversation; Description	6 hours
8.1.10. Principles of electrochemical investigation;		

Classification of methods by the nature and type of perturbation signal and by the type of response; Electrochemical cell, experimental setups; Elementary steps of electrode processes; Thermodynamic and kinetic parameters that can be determined and their importance. (2 h)		
8.1.11. Classical and non-conventional polarography: principles and instrumentation. Hydrodynamic voltammetry: principles, instrumentation, parameters. Levich and Koutecky-Levich relationships and their utility (2 h).		
8.1.12. Cyclic voltammetry, chronoamperometry and electrochemical impedance spectrosopy. Reversibility criteria. Experimental parameters and approaches for estimation of characteristic physico-chemical parameters (2 h).		
MODULE 4 – Materials and precursors characterization using evaluation of: specific surface area, pore size distribution and surface morphology by microscopic techniques (SEM, TEM)	Lecture; Explanation; Conversation; Description; Case study; Questioning	4 hours
8.1.13. Adsorption. Solid porous structure. Specific surface area and pore size distribution determination (2 h)		
8.1.14. Solid structure characterization by electronic microscopy (2 h)		

Bibliography MODULE 1

1. N. E. Jacobsen, *NMR Spectroscopy Explained*, Ed Wiley-Interscience, 2007.
2. H Friebolin, *Basic One- and Two-dimensional NMR Spectroscopy*, Wiley-VCH, 2004.
3. S. Mager, *Analiza Structurala Organică*, Ed. Ştiinţifica si Enciclopedica, Bucureşti 1979.
4. David L., Crăciun C., Cozar O., Chiş V., *Rezonanţă Electronică de Spin. Principii, metode, aplicaţii*, Presa Univ. Clujeană, Cluj-Napoca, 2001.
5. H.H. Perkampus, *UV-VIS spectroscopy and its applications*, Springer-Verlag, 1992.

Bibliography MODULE 2

1. W. Massa, *Crystal Structure Determination*, Editura Springer, Berlin, 2000. (accesibila la titularul de modul)

Bibliography MODULE 3

1. Oniciu L., Mureşan L., *Electrochimie aplicată*, Presa Universitară Clujeana, 1998.
2. A. J. Bard şi L. R. Faulkner, *Electrochemical Methods. Fundamentals and Applications*, John Wiley and Sons, New-York, 1980.

Bibliography MODULE 4

1. E. Rouquerol, J. Rouquerol, K. Sing, *Adsorption by Powders and Porous Solids. Principles, Methodology and Applications*, Academic Press, San Diego, 1999.
2. J. M. Thomas, W. J. Thomas, *Principles and Practice of Heterogeneous Catalysis*, VCH, Weinheim, 1997.
3. J.W. Niemantsverdriet, *Spectroscopy in Catalysis. An introduction*, VCH, Weinheim, 1993.

8.2 Seminar / laboratory	Teaching methods	Remarks
MODULE 1		
- ^{1}H-RMN, ^{13}C-RMN, COSY, HMQC, HMBC spectra interpretation - EI, CI, ESI, APCI, MALDI mass spectra analysis and interpretation	Explanation, conversation, description, questioning	Total 3 ore
- ESR spectra, sample preparation, spectra recording, spectral parameters interpretation	Explanation, conversation, description,	Total 2 ore

	questioning	
- UV-VIS spectra interpretation for various chemical combinations	Explanation, conversation, description, questioning	Total 2 hours
MODULE 2		
- Specific software utilization for data preparation for publication	Explanation, conversation, description, questioning	Total 2 hours
MODULE 3		
- Investigation of redox systems by cyclic voltammetry	Experiment, conversation, description, questioning	Total 3 hours
- Electrochemical impedance spectroscopy		
MODULE 4		
- S_{sp} determination of some zeolitic materials	Experiment, conversation, description, questioning	Total 2 hours – according to the specific situation, one of the proposed themes will be considered
- Retrieval and image interpretation of SEM / TEM for characterization of some natural adsorbents		

Bibliography MODULE 1

1. David L., Crăciun C., Cozar O., Chiş V., *Rezonanţă Electronică de Spin. Principii, metode, aplicaţii*, Presa Univ. Clujeană, Cluj-Napoca, 2001
2. R. M. Silverstein, F. X. Webster, D. J. Kiemle *Spectrometric Identification of Organic Compounds*, Wiley, New-York, 2005.
3. N. E. Jacobsen, *NMR Spectroscopy Explained*, Wiley-Interscience, 2007.
4. H.H. Perkampus, *UV-VIS spectroscopy and its applications*, Springer-Verlag, 1992.
5. Referate laborator şi fişe de lucru.

Bibliography MODULE 2

1. W. Massa, *Crystal Structure Determination*, Editura Springer, Berlin, 2000. (accesibila la titularul de modul)

Bibliography MODULE 3

1. I.C. Popescu, G. Turdean, A. Nicoara, P. Ilea şi L. Muresan, *Lucrări practice pentru Ciclul de Studii Aprofundate în Electrochimie*, Lito. UBB, Cluj-Napoca, 1998.
2. C. Brett, A. M. Oliveira Brett, Electrochemistry. Principles, methods and applications, Oxford Science Publications, 1993.

Bibliography MODULE 4

1. E. Rouquerol, J. Rouquerol, K. Sing, *Adsorption by Powders and Porous Solids. Principles, Methodology and Applications*, Academic Press, San Diego, 1999.
2. J. M. Thomas, W. J. Thomas, *Principles and Practice of Heterogeneous Catalysis*, VCH, Weinheim, 1997.
3. J.W. Niemantsverdriet, *Spectroscopy in Catalysis. An introduction*, VCH, Weinheim, 1993.
4. referate de laborator

9. Corroborating the content of the discipline with the expectations of the epistemic community, professional associations and representative employers within the field of the program

- By acquiring theoretical and methodological concepts and addressing practical aspects included in the **Structural characterization methods for materials and precursors** discipline, students are acquiring a solid knowledge base, according to partial competences required for possible occupations listed in grid 1 - RNCSIS.

10. Evaluation

Type of activity	10.1 Evaluation criteria	10.2 Evaluation methods	10.3 Share in the grade (%)
10.4 Course	The correctness of answers - learning and understanding of the treated issues	Written examination - access is conditioned by essays submission. Fraud intention punishable by removal from the examination room. Examination fraud is punishable by expulsion according to UBB ECST regulation.	60%
	Way of thinking, correctness and proposed solutions argumentation		
10.5 Seminar/lab activities	Essay submission	Essays must be delivered no later than the last week of teaching activity	40%
	Essay quality, correct utilization of literature data		
	Laboratory/seminar activity		
10.6 Minimum performance standards			
• 5 (five) grade for each module • Knowledge of one characterization technique and interpretation of the corresponding results for each module (1 technique/module)			

Date	Signature of course coordinator	Signature of seminar coordinator
15.05.2014	.....	.....
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Date of approval

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Signature of the head of department

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