PhD in Chemistry/ Biochemistry training program

METHODS OF INVESTIGATION OF THE STRUCTURE AND PROPERTIES OF CHEMICAL COMPOUNDS

Co-funded by the Erasmus+ Programme of the European Union

Curriculum and training manual “Methods of Investigation of the Structure and Properties of Chemical Compounds” was developed by the working group of the Institute Macromolecular Chemistry of the National Academy of Sciences of Ukraine as part of the shared curricula for inter-institutional PhD in Political/Social Science training program, developed by Political/Social Science Research Group in Erasmus+ DocHub project.

Coordination: Vilnius University (Lithuania).

Partners in Chemistry/Biochemistry Research Group: Institute Macromolecular Chemistry of the National Academy of Sciences of Ukraine of Ukraine, Institute of Condensed Matter Pysics of the National Academy of Sciences of Ukraine, National University of Kyiv-Mohyla Academy, Oles Honchar Dnipro National University (Ukraine)

Expert EU partner: Vilnius University (Lithuania).

The project “Structuring cooperation in doctoral research, transferrable skills training, and academic writing instruction in Ukraine's regions” / DocHub / has received funding by the Erasmus+ programme of the European Union under grant no. 574064-EPP-1-2016-1- LT-EPPKA2-CBHE-SP.

INTRODUCTION

METHODS OF INVESTIGATION OF THE STRUCTURE AND PROPERTIES OF CHEMICAL COMPOUNDS (4 ECTS)

Authors, instructors

Dr. Nataliia Hudzenko, Ph.D., Associate Professor, Senior Researcher Scientist, Department of Chemistry of Oigomers and Mesh Polymers, Institute of Macromolecular Chemistry of National Academy of Sciences of Ukraine

Prof. Sergii V. Riabov, PhD, Doctor of Science, Leading Researcher Scientist, Head of the Department of Polymers Modification, Institute of Macromolecular Chemistry of National Academy of Sciences of Ukraine

Dr. Ihor Tkachenko, Ph.D., Senior Researcher Scientist, Department of Chemistry of Oligomers and Netted Polymers, Institute of Macromolecular Chemistry of National Academy of Sciences of Ukraine

Dr. Eugenia Lobko, PhD., Senior Researcher, Department of Polymer Physic, Institute of Macromolecular Chemistry of National Academy of Sciences of Ukraine

Dr. Valeriy Demchenko, Ph.D., Senior Research Scientist, Department of Polymers Modification, Institute of Macromolecular Chemistry of National Academy of Sciences of Ukraine

Dr. Anastasiia V. Hubina, PhD., Senior Researcher, Department of Polymer Physic, Institute of Macromolecular Chemistry of National Academy of Sciences of Ukraine

Prof. Galyna Ushakova, Head of Department of Biophysics and Biochemistry, Oles Honchar Dnipro National University

Dr. Iryna Kolesnyk, Ph.D., Professor Assistant, Chemistry Department, National University of Kyiv-Mohyla Academy

Prof. Yevgen Mamunya, Dr.Sc., Professor, Leading Researcher Scientist, Department of Polymer Composites, Institute of Macromolecular Chemistry of National Academy of Sciences of Ukraine

Dr. Nataliia Babkina, PhD, Senior Researcher, Department of Physics and Chemistry of Polymers, Institute of Macromolecular Chemistry of National Academy of Sciences of Ukraine,

Co-funded by the
Erasmus+ Programme
of the European Union

External evaluation
The curriculum is included in PhD training program in Chemistry, Biochemistry:
102 Chemistry – pre-accredited, NAQA approval is expected in January 2021

Expert Advisor:
Prof. Edita Sužiedėlienė, Vilnius University (Lithuania)

Basing on inter-institutional agreements, the course “Methods of Investigation of the Structure and Properties of Chemical Compounds” may be included in inter-institutional PhD training programs in Chemistry, Biochemistry, Physics and other multi-disciplinary programs.

Course content

Module 1: Spectroscopic Methods of Analysis

- Topic 1: Fourier-Transform Infrared Spectroscopy
- Topic 2: Ultraviolet and Visible Spectroscopy (UV-VIS)
- Topic 3: Nuclear Magnetic Resonance Spectroscopy
- Topic 4: Dielectric Relaxation Spectroscopy (DRS)

Module 2: Structural Methods of Analysis

- Topic 1: X-Ray Diffraction Analysis (WAXS AND SAXS)

Module 3: Chromatographic Methods of Analysis

- Topic 1: Size Exclusion Chromatography

Module 3: Chromatographic Methods of Analysis

- Topic 2: Affinity Chromatography

Module 4: Electrical Kinetic Methods of Analysis

Module 5: Thermophysical methods of analysis

- Topic 1-4: Differential Scanning Calorimetry (DSC),
  Thermogravimetry (TGA),
  Thermal mechanical analysis (TMA)
  Dynamic Mechanical Analysis (DMA)

Co-funded by the
Erasmus+ Programme
of the European Union

DESCRIPTION OF THEMATIC MODULES

Module 1: SPECTROSCOPIC METHODS OF ANALYSIS

TOPIC 1: FOURIER-TRANSFORM INFRARED SPECTROSCOPY (IR)

NUMBER OF CONTACT HOURS

0,5 lecture (1 hour);

2 laboratory practice (6 hours)

BRIEF DESCRIPTION OF THE TOPIC

FT-IR spectroscopy is among of the most powerful analytical method and often used for fundamental and practical researches as well for controlling a range of industrial processes. Currently, FT-IR spectroscopy is widely applied and it combined modern and original achievements in the optics, mechanics, electronics, laser and computer technique.

Involving absorbance and reflectance, FT-IR spectroscopy allows extended information, regarding structure and composition of analyzed substances to be obtained.

Studying of the theoretical background and practical aspects of FT-IR method will provide PhD students with an appropriate basis for understanding and also training experience which would be helpful for interpreting of different spectra and establishing structure and composition of chemical compounds and species.

Much attention will be paid to practice - training tasks includes preparing and investigation of samples of different types, like solid substances, liquid and viscous specimens, oriented films, powders. Diverse polymers and organic substances will be tested and identified.

PURPOSE

Studying the course involves providing the PhD students with theoretical knowledge and practical principles on concerning FT-IR spectroscopy. PhD students will also receive practical knowledge on the application of IR-spectra for organic reagents and polymers.

LEARNING OUTCOMES

After finishing the aforesaid course the PhD students will have the following **knowledge:**

1. To report the basic principles of FT-IR spectroscopy.

2. To define such methods and step-by-step procedures for samples' preparation needed for different FT-IR adapters.

3. . To be able to characterize specimens' spectral absorption bands.

and skills:

Co-funded by the
Erasmus+ Programme
of the European Union

1. To interpret IR-spectra and to correlate a main spectral absorption bands
2. To apply FT-IR method in solving a particular tasks
3. To conduct search in the e-library, adjusted to FT-IR spectrometer

TEACHING METHODS

Lecture using slides, practical exercises, seminar.

LEARNING METHODS

- explanation with illustration – informative presentation (lecture);
- research method – practical exercises, analysis and data processing;
- discussions – seminars;
- distance method – students are learning by themselves a scientific literature, make observations, testing and searching an appropriate information.

Assessment

The exam is conducted in the form of a seminar. Each student presents his research work (Power Point presentation), describing results connected with FT-IR spectroscopy.

ADMISSION REQUIREMENTS

Basic knowledge of organic chemistry, physical and analytical chemistry, polymer chemistry and spectroscopy. Operation with PC software. Working experience in a chemical laboratory.

LIST OF TOPICS AND ACTIVITIES

Lecture. Infrared Fourier spectroscopy:

1. Performance capabilities and advantages of FT-IR method in comparison with traditional spectroscopy.
2. Concept for stretching and deformation vibrations.
3. Fundamental transitions in vibration spectra of chemical substances.
4. Dependence of vibrational spectra on molecules' chemical structure.
5. Selection of optimal conditions for monitoring of infrared spectra.
6. Dominant characteristic absorption bands in the area of basic vibrations for organic and polymer compounds.

Practical training:

1. Interpretation of IR-spectra for standard organic substances and polymers.
2. Spectrum -1

Co-funded by the
Erasmus+ Programme
of the European Union

3. Spectrum -2
4. Spectrum - 3
5. Investigation of samples which are in the following state:
   a) solid
   b) liquid
   c) as films,
6. Analysis of spectral data (curves) obtained

CONTROL TASKS

1. Required conditions for emerging IR-spectra of a molecule. Concept for stretching and deformation vibrations.
2. Dependence of absorption band's position on bond order - as it is shown for carbohydrates.
3. Dependence of absorption band's position on bond order - as it is shown for carbonyl agents.
4. Factors which influence on the absorption band intensity. Examples attached.
5. Dependence of absorption band's intensity on bond order.
6. Peculiarities in absorption for amides.

REFERENCE LIST and ONLINE SOURCES

1. Fundamentals of Fourier Transform Infrared Spectroscopy, Second Edition / Brian C. Smith .- Taylor & Francis, 2011.- 207 p.
2. Infrared Spectroscopy: Fundamentals and Applications / B. Stuart .- John Wiley & Sons, 2004.-203p.
3. Fourier Transform Infrared Spectrometry, Second edition / Peter R. Griths and James A. de Haseth:, John WIley and Sons, Hoboken, New Jersey, 2007.
4. Fourier Transform Infrared Spectrometry (2nd ed.) / Griffiths, P.; de Hasseth, J. A. Wiley-Blackwell. – 2007.
5. Fourier Transform Infrared Spectrometry, 2nd ed. // edited by P. R. Griffiths and J. A. De Haseth Wiley-Interscience.- New York, 1986. Google Scholar.
6. Spectrometric identification of organic compounds // RM Silverstein, F.X. Webster, D.J. Kiemle, D.L. Bryce. – 2014.
7. Ferraro J.R. / Practical Fourier transform infrared spectroscopy: industrial and laboratory chemical analysis. - 2012 - books.google.com/
8. ttps://www2.chemistry.msu.edu/faculty/reusch/virttxtjml/Spectrpy/InfraRed/infrared.htm/
9. http://www.bio-rad.com/en-us/product/ir-spectral-databases

MODULE 1: SPECTROSCOPIC METHODS OF ANALYSIS

TOPIC 2: ULTRAVIOLET AND VISIBLE SPECTROSCOPY (UV-VIS)

NUMBER OF CONTACT HOURS

0,5 lecture (1 hour);

2 laboratory practice (6 hours)

BRIEF DESCRIPTION OF THE TOPIC

The course is dealing with basic principles of UV-Vis spectroscopy, equipment involved for this method, and application in the area of organic and polymer chemistry.

Ultraviolet and visible (UV-Vis) absorption spectroscopy is the measurement of the attenuation of a beam of light after it passes through a sample or after reflection from a sample surface. Absorption measurements can be at a single wavelength or over an extended spectral range.

Many molecules absorb ultraviolet or visible light. The absorbance of a solution increases as attenuation of the beam increases. Absorbance is directly proportional to the path length, b, and the concentration, c, of the absorbing species. Beer's Law states that $A = \varepsilon bc$, where ε is a constant of proportionality, called the absorbtivity.

Different molecules absorb radiation of different wavelengths. An absorption spectrum will show a number of absorption bands corresponding to structural groups within the molecule. For example, the absorption that is observed in the UV region for the carbonyl group in acetone is of the same wavelength as the absorption from the carbonyl group in diethyl ketone.

This method allows the following tasks to be determined: the kinetics of a chemical reaction. The reaction, occurring in solution, must present color or brightness shifts from reactants to products in order to use UV/Vis for this application.

The rate constant of a particular reaction can be determined by measuring the UV/Vis absorbance spectrum at specific time intervals. Different rate orders have different integrated rate laws depending on the mechanism of the reaction.

An equilibrium constant can also be calculated with UV/Vis spectroscopy. After determining optimal wavelengths for all species involved in equilibria, a reaction can be run to equilibrium, and the concentration of species determined from spectroscopy at various known wavelengths. The equilibrium constant can be calculated as K(eq) = [Products] / [Reactants].

PURPOSE

The goal of course is to supply PhD students with the theoretical knowledge and practical skills needed for the interpretation of the UV-Vis spectra, followed by identification of organic substances investigated.

Co-funded by the
Erasmus+ Programme
of the European Union

LEARNING OUTCOMES

After learning the topic "UV-Vis spectroscopy", the PhD students will acquire the following:

1. theoretical basic principles and technique of UV-Vis spectroscopy.
2. ability for preparation of samples in solution and solid state.
3. operation skills with equipment and its software and appropriate programs.
4. capability for interpretation and identification of UV-Vis spectra.

TEACHING METHODS

Lectures, seminars, practical tasks and discussions.

LEARNING METHODS

Self-studying of the relevant literature, discussions with tutor, preparing a presentations.

EXAMINATION

The exam will be provided in the frame of a seminar with personal presentations of students involved. Before it, a short individual reports have to be prepared by every students.

ADMISSION REQUIREMENTS

Basic knowledge in Organic chemistry, Polymer chemistry and UV–Vis spectroscopy

LIST OF TOPICS AND ACTIVITIES

Lecture. UV–Vis spectroscopy (introduction and principles)

1. Definition of UV-radiation
2. Principle of UV-Vis spectroscopy
3. Absorption spectrum and Absorbance laws
4. Types of transitions, effect of chromophore, Woodward-Feiser rule, solvent effects
5. Instrumentation and applications

Practical training

1. Working on the UV-Vis spectrophotometer, sample handling and operation with software.
2. Interpretation of UV-Vis spectra of simple and then complicated organic molecules.
3. Short-range presentation regarding of UV-Vis spectroscopy's practical and theoretical aspects and its application in industrial areas.

Co-funded by the
Erasmus+ Programme
of the European Union

CONTROL TASKS

1. Detection of functional groups.
2. Detection of impurities.
3. Qualitative analysis.
4. Quantitative analysis.
5. Single compound without chromophore.
6. Drugs with chromophoric reagent.
7. Tautomeric equilibrium.
8. Electronic transitions: involving π, σ, and n electrons, and transitions involving charge-transfer electrons.

EDUCATIONAL RESOURCES

1. Advantage in UV/Visible Spectroscopy
https://www.agilent.com/cs/library/
2. Ultraviolet and Visible Spectroscopy (S. Kumar)
www.uobabylon.edu.iq/.../publication_11_8282_250.pdf
3. Basic UV-Vis Theory, Concepts and Applications - ResearchGate
https://www.researchgate.net/file.PostFileLoader.html?id...
4. UV-Visible spectroscopy
web.iitd.ac.in/~sdeep/Electronic.pdf
5. http://www.chemguide.co.uk/analysis/uvvisible/theory.html

REFERENCE LIST

1. Robert M. Silverstein, Francis X. Webster, David Kiemle // Spectrometric Identification of Organic Compounds, 7th Edition, Wiley, 2005, 512 p.
2. Robert M. Silverstein, Francis X. Webster, David J. Kiemle, David L. Bryce Spectrometric Identification of Organic Compounds, 8th Edition, John Wiley & Sons, 2014, 464 p.
3. Tony Owen // Fundamentals of modern UV-visible spectroscopy, Agilent Technologies, 2000.
4. Subodh Kumar // Spectroscopy of Organic Compounds, Guru Nanak Dev University, 2006.
5. The Royal Society of Chemistry // Ultraviolet/visible spectroscopy, Unilever, 2011.

Co-funded by the
Erasmus+ Programme
of the European Union

MODULE 1: SPECTROSCOPIC METHODS OF ANALYSIS

TOPIC 3: NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY

NUMBER OF CONTACT HOURS

1 lecture (2 hours);

2 laboratory practice (6 hours)

BRIEF DESCRIPTION OF THE COURSE THEME

The course theme presents an overview of the principles of high resolution nuclear magnetic resonance (NMR) spectroscopy and its application within organic and polymer chemistry. This will mainly focus on the application of solution-state NMR to the study of different types of organic compounds and polymers. Also, this topic will be having a focus on practical skills associated with evaluating NMR data and spectral interpretation. The NMR spectroscopy topic will include one-dimensional 1D NMR experiments, chemical shifts, J-coupling, interpretation of NMR spectrum. The concepts will be demonstrated by NMR spectra of organic molecules and macromolecules. Additionally, different 2D NMR and solution-state NMR experiments will be briefly discussed.

THE GOAL OF THEME

The purpose of the theme is to provide the theoretical and practical background necessary to understand how NMR results can be applied for identification of organic compounds as well as interpretation of the NMR spectra.

LEARNING OUTCOMES

Having successfully followed the topic "Nuclear Magnetic Resonance Spectroscopy", the PhD. students will have following **knowledge:**

1. about the basic principles and techniques of NMR spectroscopy;
2. about sample preparation procedure in solution-state 1D NMR spectroscopy;
3. about the processes responsible for NMR chemical shifts and splitting patterns

and skills:

1. be able to analyze the different information about structure of molecules from solution-state ^{1}H and ^{13}C NMR spectra;
2. be able to interpret both ^{1}H and ^{13}C NMR spectra of simple compounds;
3. be able to use NMR experiment for solving a particular physico-chemical problem;
4. be able to use some NMR sofrware.

Co-funded by the
Erasmus+ Programme
of the European Union

TEACHING METHODS

Lectures using slides, practical NMR exercises and classroom discussion (students will present and discuss selected topics).

LEARNING METHODS

- explanation-illustration - presentation;

- research – acquirement of practical skills with data processing and analysis.

EXAMINATION

The exam is in the form of a seminar. Additionally, written individual report on elucidation of a compound's structure using NMR techniques is required.

ADMISSION REQUIREMENTS

Basic knowledge of organic chemistry and spectroscopy

LIST OF TOPICS AND ACTIVITIES

Lecture. Nuclear Magnetic Resonance Spectroscopy:

1. General introduction to NMR
2. One-dimensional proton NMR spectroscopy: chemical shift, integration and spin-spin coupling
3. Carbon-13 NMR spectroscopy (sensitivity, chemical shift, coupling)
4. Other NMR techniques and applications (19F NMR spectroscopy, solid -state NMR, 2D NMR techniques)

During the lecture the student should choose for one of the selected presentation topics.

Independent work will be connecting with solving some NMR tasks that can help students in their research work. Additionally, the completion of the theme requires presenting some suggested specific topics in a research seminar.

Practical training:

1. Practical exercises: interpretation of ^{1}H NMR spectra and predict ^{1}H NMR spectra for standard organic molecules.
2. Automated spectral prediction using the NMR simulation software
3. Each Ph.D. student will make a (10-15)–minute presentation on the selected topic. A 5-minute question and answer period will follow each presentation.

Co-funded by the
Erasmus+ Programme
of the European Union

CONTROL TASKS

A list of presentation topics:

1. NMR chemical shifts of trace impurities: common laboratory solvents and organics
2. 1D ^{13}C NMR experiments: ATP and DEPT
3. 2D NMR spectra: COSY
4. 2D NMR spectra: NOESY, ROESY and TOSCY
5. Solid state NMR spectroscopy
6. NMR in medicine and biology
7. ^{19}F NMR spectroscopy

A list worked question and problems (some examples)

1. The proton spectrum with integration. This spectrum tells you…..?
2. What (s, d, t…) do you expect to observe in the ^{1}H NMR spectrum of chloroethane CH_3CH_2Cl?
3. How many signals do you expect to see in the ^{1}H NMR spectra of 2-bromopropane $(CH_3)_2CHBr$?
4. A ^{1}H NMR spectrum of compound **X** contains a singlet, a triplet and a quartet. Which of the following compounds might **X** be? ($CH_3CCl_2CH_2CH_3$, $CH_3CH_2CHClCHCl_2$, $CH_3CH_2CH_2CHCl_2$, $CH_3CHClCHClCH_3$)?

REFERENCE LIST

1. Günther H. NMR spectroscopy: basic principles, concepts and applications in chemistry / H. Günther. – John Wiley&Sons, 2013. – 734 p.
2. Modern NMR techniques for chemistry research / A.E. Derome. – Elsevier, 2013. – 299 p.
3. Spectrometric identification of organic compounds / R.M. Silverstein, F.X. Webster, D.J. Kiemle, D.L. Bryce. - John Wiley & Sons, 2014. – 464 p.
4. Keeler J. *Understanding NMR spectroscopy* / J. Keeler. – John Wiley&Sons, 2011. – 526 p.
5. Claridge T.D.W. High-resolution NMR techniques in organic chemistry / T.D.W. Claridge. – Elsevier, 2016. – 552 p.
6. Ning Y.-C. Interpretation of organic spectra / Y.-C. Ning. – John Wiley&Sons, 2011. – 416 p.
7. Bovey F. High resolution NMR of macromolecules / F. Bovey. – Elsevier, 2012. – 476 p.
8. Kwan E.E. Structural elucidation with NMR spectroscopy: practical strategies for organic chemists / E.E. Kwan, S.G. Huang // European Journal of Organic Chemistry. – 2008. – Vol. **16**. – P. 2671-2688.

Educational resources

9. UC Davis Nuclear magnetic resonance facility / http://nmr.ucdavis.edu/useful-links/
10. Tools for NMR spectroscopists / https://www.nmrdb.org
11. Spectral database for organic compounds, SDBS. / http://sdbs.db.aist.go.jp/sdbs/cgi-bin/cre_index.cgi

Co-funded by the Erasmus+ Programme of the European Union

MODULE 1: SPECTROSCOPIC METHODS OF ANALYSIS

TOPIC 4: DIELECTRIC RELAXATION SPECTROSCOPY (DRS)

NUMBER OF CONTACT HOURS

1 laboratory practice (3 hours)

BRIEF DESCRIPTION OF THE TOPIC

Dielectric Relaxation Spectroscopy (DRS) highlights the main practical way of investigation of the dielectric materials, semiconductors, electrical and ion conductive materials (such as proton-conductive membranes, ionic liquids, electrolytes, fuel cells etc.) as well as relaxation properties of the materials.

THE GOAL OF THEME

To study the basic principles of the method of dielectric relaxation spectrometry for investigation of conductivity (electronic and ionic) and relaxation properties of the materials of different nature.

LEARNING OUTCOMES

Having successfully followed the topic "Dielectric Relaxation Spectroscopy", the PhD. students will have following **knowledge:**

1. about the basic principles and techniques of DRS;
2. about the possible application of DRS.

and skills:

1. be able to prepare samples for measurement;
2. be able to use the specific equipments (impedance meter, alternating current bridge);
3. be able to process data and perform analysis.

TEACHING METHODS

Laboratory (samples preparation, data registration, data processing and analysis).

LEARNING METHODS

- explanation-illustration - presentation, demonstration materials;
- research – acquirement of practical skills with equipment, data processing and analysis.

EXAMINATION

The exam is in the form of a seminar.

Co-funded by the
Erasmus+ Programme
of the European Union

ADMISSION REQUIREMENTS

Basic knowledge of organic chemistry and macromolecular chemistry.

LIST OF ACTIVITIES

Laboratory. Dielectric Relaxation Spectroscopy

1. Investigation of electrical and dielectrical properties of materials using DRS.

2. Preparation of samples for analysis.

3. Registration of DRS spectra.

4. Data processing (including the calculation of sample conductivity, dielectric constant, tangent of dielectric loss and others). Analysis of the data.

5. Investigation of relaxation characteristics of materials using DRS. Registration DRS spectra in temperature and frequency ranges. Data processing. Analysis of relaxation transitions in materials.

REFERENCE LIST

1. Broadband Dielectric Spectroscopy / Ed. by F. Kremer, A. Schönhals. – Springer, 2003. – 729 p.

2. Impedance Spectroscopy: Theory, Experiment, and Applications / Ed. by Evgenij Barsoukov and J. Ross Macdonald. – Wiley Interscience, 2nd Edition, 2005. - 616 p.

Co-funded by the
Erasmus+ Programme
of the European Union

MODULE 2: STRUCTURAL METHODS OF ANALYSIS

TOPIC 1: X-RAY DIFFRACTION ANALYSIS (WAXS AND SAXS)

NUMBER OF CONTACT HOURS

1 lecture (2 hours);

1 laboratory practice (3 hours)

BRIEF DESCRIPTION OF THE TOPIC

This course encompasses the modern methods for investigation of structure of materials including the polymers. The prime accent will be focused on the following methods: Wide- and small-angle scattering of X-rays (WAXS and SAXS). Radiographic methods provide powerful ways of studying the structure of materials and often used for the fundamental and practical researches. Currently, the WAXS and SAXS methods are widely applied for the study of structure various materials such as polymers, nanocomposites, fillers of non-polymer nature.The course contains both theoretical foundations of X-ray structural an and the application of SAXS and WAXS methods to study the corresponding structure of the materials. Acquaintance with the basic laws of the study of the structure of chemical compounds, polymeric materials, composites and nanocomposites by methods of wide- and small-angle scattering of X-rays.

PURPOSE

Studying the course involves providing the PhD students with theoretical knowledge on of X-ray structural analysis methods, familiarity with the principles and approaches of each method of research, with the structural characteristics that can be measured by each of these methods. PhD students will also receive practical knowledge on the application of X-ray structural analysis methods, as well as acquire skills in the equipment on each research method and the ability to analyze the experimental data received.

LEARNING OUTCOMES

After finishing the aforesaid course the PhD students will have the following **knowledge:**

1. To report the basic principles of X-ray structural analysis methods of research (WAXS and SAXS), which structural characteristics of materials can be determined by each of the methods.

2. To define such structural parameters as Bragg's period, relative level of crystallinity, size of the crystallites, the period of alternation in space (volume of polymers) of regions of heterogeneity of the same local electron density, the size of the range of heterogeneity (Rouland's technique) and other characteristics of materials including polymers, composites and their constituents.

Co-funded by the
Erasmus+ Programme
of the European Union

and skills:

1. To recognize the equipment for each structural method.

2. To analyze the XRD.

LEARNING METHODS

1. Explanation with illustration – informative presentation (lecture).

2. Research method – practical exercises with equipment, analysis of the processing data.

3. Distance method – students are learning by themselves a scientific literature, make observations, testing and searching an appropriate information.

EXAMINATION

The exam is conducted in the form of a seminar. Each student presents his research work (Power Point presentation), describing results obtained by one of the structural method.

ADMISSION REQUIREMENTS

Basic knowledge physical chemistry, polymer physics and chemistry, physico-chemistry of composite materials. Operation with PC software. Working experience in a laboratory equipment.

LIST OF TOPICS AND ACTIVITIES

Lecture. X-ray diffraction analysis (WAXS and SAXS):

1. The method of wide-angle scattering of X-rays. Bragg's equation, methodology for determining:

a) additive X-ray diffraction patterns and their comparison with experimental ones;

b) the relative level of crystallinity (according to Matthew);

c) the size of the crystallites (Scherrer's method).

2. The method of small-angle scattering of X-rays:

a) the period of alternation in space (volume of polymers) of regions of heterogeneity of the same local electron density;

b) the size of the range of heterogeneity (Rouland's technique);

Practical training:

1. Interpretation of SAXS and WAXS profiles standard chemical substances and polymers.
2. Experiment testing of samples which are in the following state:
   a) solid;
   b) liquid;
   c) powder.
3. Analysis of SAXS and WAXS curves of chemical compounds and polymeric material.

Co-funded by the
Erasmus+ Programme
of the European Union

CONTROL TASKS

1. Classification of X-ray methods for polymer research (WAXS and SAXS). X-ray optical scheme of these methods.

2. Calculation of the additive WAXS diffractograms materials.

3. Determination of Bragg's period, relative level of crystallinity (according to Matthew); size of the crystallites (Scherrer's method).

2. Calculation of the period of alternation in space (volume of polymers) of regions of heterogeneity of the same local electron density; the size of the range of heterogeneity (Rouland's technique).

REFERENCE LIST

.

1. V. I. Shtompel' and Yu. Yu. Kercha, Structure of Linear Polyurethanes (Naukova dumka, Kiev, 2008) [in Russian].
2. Porod G. General theory / G. Porod // Small-angle x-ray scattering /Ed. by O. Glatter, O. Kratky. – London : Acad. Press, 1982. – P. 17–51
3. Demchenko V., Riabov S., Rybalchenko N., Goncharenko L., Kobylinskyi S., Shtompel' V. X-ray study of structural formation, thermomechanical and antimicrobial properties of copper-containing polymer nanocomposites obtained by the thermal reduction method, Eur. Polym. J., 2017, 96: 326–336.

Co-funded by the Erasmus+ Programme of the European Union

MODULE 3: CHROMATOGRAPHIC METHODS OF ANALYSIS

TOPIC 1: SIZE EXCLUSION CHROMATOGRAPHY

NUMBER OF CONTACT HOURS

1 lecture (2 hours);

1 laboratory practice (3 hours)

BRIEF DESCRIPTION OF THE TOPIC

The course concerns the main principles and application of size exclusion chromatography (SEC). The course describes SEC as a powerful modern method to separate compounds with different molecular weight and to investigate molecular mass characteristics of polymer compounds, including the number average molecular weight (Mn), the weight average molecular weight (Mw) , the size average molecular weight (Mz), or the viscosity molecular weight (Mv) and polydispersity index (PDI). Among the main topics of this course are the following: the principle of SEC performance, principles of separation, instrumentation for the method, sample preparation, data interpretation.

PURPOSE

The goal of the course is to deliver the basic knowledge of the method, which allows to choose appropriate instrumentation and analysis conditions to investigate molecular mass characteristics of the polymers.

LEARNING OUTCOMES

On having completed the “Size Exclusion Chromatography” course, the students will gain following **knowledge:**

1. basic principles and instrumentation of SEC
2. choosing the appropriate instrumentation for polymers separation and investigation of molecular-mass characteristics
3. data interpretation

and skills:

1. choose instrumentation and prepare the sample for analysis
2. use SEC to solve an actual physico-chemical problem
3. determine molecular mass characteristics M_n, M_w, M_z, Mv, PDI using SEC.

TEACHING METHODS

Lectures with visual aids (presentation), practical exercises, discussion.

Co-funded by the
Erasmus+ Programme
of the European Union

LEARNING METHODS

- explanation-illustration - presentation;
- research – acquirement of practical skills with data processing and analysis.

EXAMINATION

The exam is held as a seminar, including solving individual task on chosen polymer compound characterization.

ADMISSION REQUIREMENTS

Basic knowledge of colloid chemistry and macromolecular chemistry.

LIST OF TOPICS AND ACTIVITIES

Lecture. Size-Exclusion Chromatography:

1. Main principles of SEC as a HPLC method
2. Instrumentation and sample preparation
3. Main characteristics provided by the method
4. Peculiarities and tips

During the lecture the student should choose for one of the selected presentation topics.

Independent work concerns choosing the instrumentation and analysis conditions for a defined polymer (preferably the polymer, synthesized by the student).

Practical training

1. Practical exercises: choise of the column, solvent and standards for analysis of the selected polymers or their separation from the low-molecular compounds.
2. Presentations on the selected topic concerning the course.

CONTROL TASK

A list of presentation topics:

1. Sample preparation
2. Columns and column sets
3. FIPA (Flow Injection Polymer analysis)
4. SEC of proteins
5. SEC in aqueous solutions

Co-funded by the
Erasmus+ Programme
of the European Union

A list worked question and problems (some examples)

1. If a protein cannot be detected by UV, what can we do?

2. How is resolution affected in columns of different heights?

3. Does amount of sample loaded onto the column impact the resolution? How much sample is usually loaded for analysis?

4. Why do SEC columns have different bead sizes? Can we calculate how much beads of different sizes are available in a specific volume of resin?

5. What is the difference between resolution and sensitivity?

REFERENCE LIST

1. Data Reduction in Multidetector Size Exclusion Chromatography, Y Brun J. Liq. Chrom & Rel. Technology, 21 (13), 1979 - 2015 (1998).

2. "Polymer Hardbook", J. Brandrup, E.H. Immersut: John Wiley & Sons Publisher.

3. Modern Size-Exclusion Liquid Chromatography, W.W. Yau, J.J. Kirkland, D.D. Bly John Wiley & Sons, Inc., New York, 1979.

4. Molecular Biomethods Handbook, Ralph Rapley, John M. Walker, Springer, 1998. Sigma-Aldrich Gel Filtration

https://www.sigmaaldrich.com/content/dam/sigma-aldrich/docs/Sigma-Aldrich/General_Information/1/ge-gel-filtration.pdf

Co-funded by the
Erasmus+ Programme
of the European Union

MODULE 3: CHROMATOGRAPHIC METHODS OF ANALYSIS

TOPIC 2: AFFINITY CHROMATOGRAPHY

NUMBER OF CONTACT HOURS

1 laboratory practice (3 hours)

BRIEF DESCRIPTION OF THE TOPIC

This course topic covers all the practical theory you need to know about Affinity Chromagraphy and its related techniques including column, injection techniques, eluters, sampling, data analysis. Affinity chromatography is a separation method based on a specific binding interaction between an immobilized ligand and its binding partner. Examples include antibody/antigen, enzyme/substrate, and enzyme/inhibitor, hormone/receptor interactions. This topic course is packed full of examples and practical tips, makes full use of props and includes a Protocol for lab., Questions and Answers session at the end of topic.

PURPOSE

The aim of the topic is to develop basic knowledge for affinity chromatography. This course is for PhD student who have basic knowledge of chemistry and biochemistry and would like to enhance it to practical skills in industrial and medical affinity chromatography.

LEARNING OUTCOMES

Having successfully followed the topic "Affinity Chromatography", the PhD. students will have following **knowledge:**

1. to learn the basic points for affinity chromatography (AfCH);
2. to learn how to select and use differ ligands for AfCH;
3. to learn about various techniques for AfCH in industry and medicine;
4. to learn about the principles and components of AfCH for protein purification.

and skills:

1. to choose sorbents and ligands for affinity chromatography;
2. to separate specific protein with affinity chromatography;
3. to develop colums (or strips) for affinity chromatography.

TEACHING METHOD

Tutorial lab, face-to-face classroom.

Co-funded by the
Erasmus+ Programme
of the European Union

EXAMINATION

The exam is conducted in the form of a seminar. Each student presents his research work (Power Point presentation), describing results connected with affinity chromatography.

ADMISSION REQUIREMENTS

Basic knowledge of organic chemistry, physical and analytical chemistry, polymer chemistry, biochemistry, protein chemistry, spectroscopy. Operation with PC software. Working experience in a chemical and biochemical laboratory.

LIST OF TOPICS AND ACTIVITIES

Practical training.

1. Practical Essentials of Affinity Chromatography (3 h).

2. The Protocol for lab includes all information for column preparation with heparin-sepharose for affinity purification of heparin-binding protein from the rat brain extract.

CONTROL TASKS

1. The main principles to choose the sorbent for affinity chromatography of neurospecific proteins.
2. Effect of ions strength and pH for binding and elution in affinity chromatography.
3. The main characteristics for microstrips for affinity chromatography in medicine.
4. Affinity chromatography for industrial affinity columns.

REFERENCE LIST

1. Affinity Chromatography Handbook
https://research.fhcrc.org/content/dam/stripe/hahn/methods/biochem/Affinity_Chromatography_Handbook-Specific_Groups_of_Biomolecules.pdf

2. Stranska R, Gysbrechts L, Wouters J, Vermeersch P, Bloch K, Dierickx D, Andrei G, Snoeck R.Comparison of membrane affinity-based method with size-exclusion chromatography for isolation of exosome-like vesicles from human plasma. J Transl Med. 2018 Jan 9;16(1):1. doi: 10.1186/s12967-017-1374-6.

3. King SL, Joshi HJ, Schjoldager KT, Halim A, Madsen TD, Dziegiel MH, Woetmann A, Vakhrushev SY, Wandall HH. Characterizing the O-glycosylation landscape of human plasma, platelets, and endothelial cells. Blood Adv. 2017 Feb 23;1(7):429-442. doi: 10.1182/bloodadvances.2016002121.

4. Li Z, Kim J, Regnier FE. Mobile Affinity Sorbent Chromatography. Anal Chem. 2018 Jan 9. doi: 10.1021/acs.analchem.7b03117.

Co-funded by the
Erasmus+ Programme
of the European Union

5. Mikel P, Vasickova P, Kralik P. One-plasmid double-expression His-tag system for rapid production and easy purification of MS2 phage-like particles. Sci Rep. 2017 Dec 13;7(1):17501. doi: 10.1038/s41598-017-17951-5.

6. Tkach VL, Ushakova GA, Lepekhin EA. [Isolation and purification of a membrane hyaluronate-binding protein from embryonic human brain]. Ukr Biokhim Zh (1978). 1998 Sep-Oct;70(5):69-74.

Co-funded by the
Erasmus+ Programme
of the European Union

MODULE 4: ELECTRICAL KINETIC METHODS OF ANALYSIS

NUMBER OF CONTACT HOURS

1 laboratory practice (3 hours)

BRIEF DESCRIPTION OF THE TOPIC

The topic is devoted to electrokinetic methods of analysis such as electrophoresis, electro-osmosis, measurement of sedimentation and streaming potentials. Electrokinetic phenomena can be defined as all those phenomena associated with the tangential motion of a fluid adjacent to a charged surface. Their study is one of the classic branches of colloidal science, electrokinetics, which was developed in connection with the theories of electric double layer and electrostatic surface forces.

Studying of the theoretical background and practical aspects of electrokinetic methods of analysis will supply PhD students with an appropriate basis for understanding and also training experience which would be helpful for data interpreting of electrokinetic surface properties. Much attention will be paid to practice. Training tasks include measurement of the streaming potential of membrane surface at different pH range.

PURPOSE

The aim of the topic is to acquire knowledge and practical principles concerning electrokinetic methods of analysis and to provide analysis of electrokinetic characteristics of the surface.

LEARNING OUTCOMES

After finishing the above course students will have the following knowledge:

1. basic principles of electrokinetic methods of analysis;
2. methods and procedures for measurement electrokinetic characteristics of disperse systems.

and skills:

1. to measure the streaming potential of surface;
2. to apply electrokinetic methods in solving particular tasks;
3. to analyze and interpreter data of electrokinetic methods of analysis.

LEARNING METHODS

- Explanation with illustration – informative presentation;
- Research method – practical exercises, analysis and data processing;
- Distance method – students are learning by themselves a scientific literature, make observations, testing and searching an appropriate information.

Co-funded by the Erasmus+ Programme of the European Union

EXAMINATION

The exam is conducted in the form of a seminar.

ADMISSION REQUIREMENTS

Basic knowledge of organic chemistry, analytical, physical and surface chemistry. Operation with PC software. Working experience in a chemical laboratory.

LIST OF TOPICS AND ACTIVITIES

Practical training:

1. Measurement of streaming zeta-potential of polymeric membranes *vs* pH.

2. Analysis and interpretation of obtained data.

CONTROL TASKS

1. Electrokinetic phenomena: electrophoresis, sedimentation potential, electro-osmosis, streaming potential, streaming current, electro-capillarity, electro-acoustic effects.

2. Electrochemical double layer and double layer models.

3. Electrokinetic potential, streaming potential.

4. Charge formation mechanisms on solid surfaces.

REFERENCE LIST

1. Measurement and interpretation of electrokinetic phenomena / [A.V. Delgado, F. González-Caballero, R.J. Hunter et al.] // Journal of Colloid and Interface Science. – 2007. – Vol.309. – P. 194–224.

2. Electrical phenomena at interfaces and biointerfaces / Edited by H. Ohshima, 2012. – New Jersey: Willey, 850 p.

3. Use of electrophoretic mobility and streaming potential measurements to characterize electrokinetic properties of ultrafiltration and microfiltration membranes / [L. Ricq, A. Pierre, J.-C. Reggiani et al.] // Colloids and Surfaces A: Physicochemical and Engineering Aspects. – 1998. – Vol. 138, Is. 2–3. – P. 301-308.

Co-funded by the Erasmus+ Programme of the European Union

MODULE 5: THERMOPHYSICAL METHODS OF ANALYSIS

TOPIC 1-4: DIFFERENTIAL SCANNING CALORIMETRY (DSC), THERMOGRAVIMETRY (TGA), THERMAL MECHANICAL ANALYSIS (TMA) AND DYNAMIC MECHANICAL ANALYSIS (DMA)

NUMBER OF CONTACT HOURS

1 lecture (2 hours);

4 laboratory practice (12 hours)

BRIEF DESCRIPTION OF THE TOPIC

This course encompasses the modern methods for investigation of structure and thermophysical properties of materials including the polymers. The prime accent will be focused on the following methods: Differential scanning calorimetry (DSC), Thermogravimetry (TGA), Thermal mechanical analysis (TMA) and Dynamic mechanical analysis (DMA).

Thermophysical methods provide powerful ways of studying the fundamental properties of materials and often used for the fundamental and practical researches as well as for controlling of variety industrial processes. Currently, the DSC, TGA, TMA and DMA methods are widely applied for the study of various materials such as polymers, composite materials and their constituents of non-polymer nature

The course contains both theoretical foundations of thermophysics and the application of thermophysical research methods to study the corresponding properties of the materials. Also, the course includes the application of thermophysical methods for the research of polymer materials, composite materials and their constituents of non-polymer nature. During the course, the students will been familiar with the principles of each method, also the students will be provided illustration of the ability of each method, explain what information about the structure and properties of different materials can be defined by these methods.

PURPOSE

Studying the course involves providing the PhD students with theoretical knowledge on thermophysical methods, familiarity with the principles and approaches of each method of research, with the possibilities of methods and amount of scientific data that can be obtained by each thermophysical method. PhD students will also receive practical knowledge on the application of thermophysical methods, as well as acquire skills in the equipment on each research method and the ability to analyze the experimental data received.

Co-funded by the
Erasmus+ Programme
of the European Union

LEARNING OUTCOMES

After finishing the aforesaid course the PhD students will have the following **knowledge:**

1. To report the basic principles of thermophysical methods of research, which structural, physical and mechanical characteristics of materials can be determined by each of the methods.

2. To define such thermophysical parameters as glass transition, melting, thermal stability, temperature expansion, elastic modulus and other characteristics of materials including polymers, composites and their constituents.

3. To choose the temperature and/or frequency ranges of research and to explain the difference between the dynamic and static methods,

and skills:

1. To recognize the equipment for each thermophysical method.

2. To analyze the experimental data and select the define functions and represent them graphically.

LEARNING METHODS

1. Explanation with illustration – informative presentation (lecture).

2. Research method – practical exercises with equipment, analysis of the processing data.

3. Distance method – students are learning by themselves a scientific literature, make observations, testing and searching an appropriate information.

EXAMINATION

The exam is conducted in the form of a seminar. Each student presents his research work (Power Point presentation), describing results obtained by one of the thermophysical method.

ADMISSION REQUIREMENTS

Basic knowledge physical chemistry, polymer physics and chemistry, physico-chemistry of composite materials. Operation with PC software. Working experience in a laboratory equipment.

LIST OF TOPICS AND ACTIVITIES

Lecture. Thermophysical methods of analysis:

1. Basic concepts of the principles of thermophysical methods of research.
2. Thermal transitions in polymers and non-polymeric materials.
3. Short characteristics of the DSC, TGA, TMA and DMA methods of investigations.
4. Ways of the samples preparation for each thermophysical method.

Co-funded by the
Erasmus+ Programme
of the European Union

Practical training:

1. DSC method. Carrying out an experiment test of certain solid polymer material to determine the temperature transition of melting. Testing of a thermosetting polymer (e.g., an epoxy resin) to location the temperature region of the glass transition. Evaluation of the parameters of glass transition and melting, calculation of the degree of crystallinity.

2. TGA method. Investigation of a certain material in the temperature range of 20-700 ^{0}C, identify of areas of thermal destruction, thermal decomposition rates, coke residue values, calculation of the content of the thermally resistant component in the case of composite material research.

3. TMA method. Experiment testing of thermosetting or thermoplastic material in penetration mode and thermal expansion mode. Distinguishing of the region of glass transition and melting of the sample. Measurement of the coefficient of thermal expansion. Determination of the thermal stability of the material under load.

4. DMA method. Investigation of viscoelastic properties of the elastic material in a wide range of temperatures. Determination of modulus of elasticity, coefficient of mechanical losses, obtaining a spectrum of temperature transitions, connected with the molecular structure of material.

CONTROL TASKS

1. Thermal transitions in certain solids, polymers, composites. Determination of glass transition, analysis of the parameters of vitrification. Identification of melting transition. Analysis of characteristics of the melting process.

2. Temperature range and ambient conditions of the TGA measurements. Determination of areas of thermal destruction and thermal decomposition rate.

3. Temperature and stress region of the measurements. TMA curve and differential (DTMA) curve, definition of thermal transitions. Modes (penetration and expansion) of device, the purpose of each mode. Calculation of the coefficient of thermal expansion.

4. Viscoelastic properties of materials in a wide range of temperatures. Determination of the modulus of elasticity, coefficient of mechanical losses. Relation a spectrum of temperature transitions with the molecular structure.

REFERENCE LIST

1. Godovsky Yu.K. Thermophysics of polymers. – Moscow, Chemistry, 1982. – 280

2. Hatakeyama T., Quinn F.X. Thermal analysis. Fundamentals and applications to polymer science. - Willey: N-Y, 1995, 158 p.

3. Thermal analysis of rubbers and rubbery materials. Eds.: N.R. Choudhury, P.P. De, N.K. Dutta. - Smithers: Shawbury, UK, 2010, 216 p.

Co-funded by the
Erasmus+ Programme
of the European Union

4. Thermal analysis of polymers. Fundamental and applications. Eds.: J.D. Menczel, R.B.Prime. - Willey: Hoboken, 2009, 385.

5. Metal, ceramic and polymeric composites for various uses. Ed.: J. Cuppoletti. – InTech, Rijeka, 2011, 682 p.

6. John D. Ferry. Viscoelastic properties of polymers. - Third Edition. John Wiley and Sons, New York (1980), 641 p.

7. Michael P. Sepe. Dynamic mechanical analysis for plastics engineering. - Plastics Design Library, Norwich, NY (1998) 189 p.

Co-funded by the
Erasmus+ Programme
of the European Union