ENEA
ITALIAN NATIONAL AGENCY FOR NEW TECHNOLOGIES, ENERGY AND SUSTAINABLE ECONOMIC DEVELOPMENT

CIRMMP
FIRENZE

iNEXT DISCOVERY
Structural biology for translational research & discovery

Abstract

"High- and low-resolution NMR analysis of food samples... and some tricks to improve it"

Krzysztof Kazimierczuk, Centre of New Technologies, University of Warsaw

NNuclear magnetic resonance spectroscopy (NMR) is a potent tool for chemical analysis of mixtures. The exciting examples are food samples such as olive oil, beverages, coffee, honey and many others. NMR can be used to identify compounds present in a mixture. Also, the general statistical analysis of spectral features ("fingerprinting") can detect the sample origin, counterfeiting, adulteration etc.

The high-resolution NMR based on superconductive magnets is the main workhorse of structural analysis ab initio. For general sample screening, however, the low-resolution benchtop spectrometers with permanent magnets are sufficient.

In my presentation, I will discuss simple tips and tricks allowing to enhance the analysis of mixtures from food industry. The discussed methods will include:

- *two-dimensional (2D) NMR with non-uniform sampling [1]*
- *time-resolved 2D NMR [2]*
- *variable-temperature NMR with Radon transform [3]*
- *pH "pseudo-dimension" [4]*
- *quantitative mixture analysis using Wasserstein metric [4]*

I will provide examples of application of high-field NMR (700 MHz) and low-field NMR (43 MHz) on food samples: olive oil, coffee, powdered milk, fermentation, and a fitness supplement.

References:

1. Kazimierczuk, K., & Orekhov, V. (2015). Non-uniform sampling: Post-Fourier era of NMR data collection and processing. Magnetic Resonance in Chemistry, 53(11), 921-926.

2. Dass, R., Kozminski, W., & Kazimierczuk, K. (2015). Analysis of complex reacting mixtures by time-resolved 2d NMR. Analytical Chemistry, 87(2), 1337-1343.

3. Nawrocka, E. K., Urbańczyk, M., Koziński, K., & Kazimierczuk, K. (2021). Variable-temperature NMR spectroscopy for metabolite identification in biological materials. RSC Advances, 11(56), 35321-35325.

4. Putko, P., Romero, J. A., & Kazimierczuk, K. (2024). A pH-enhanced resolution in benchtop NMR spectroscopy. Analyst, 149, 1998-2003.

5. Domżał, B., Nawrocka, E. K., Gołowicz, D., Ciach, M. A., Miasojedow, B., Kazimierczuk, K., & Gambin, A. (2024). Magnetstein: An Open-Source Tool for Quantitative NMR Mixture Analysis Robust to Low Resolution, Distorted Lineshapes, and Peak Shifts. Analytical Chemistry, 96(1), 188-196.

Krzysztof Kazimierczuk

Krzysztof Kazimierczuk did his PhD in 2009 at the University of Warsaw, Poland. A postdoc in the group of prof. Vladislav Orekhov (Swedish NMR Centre, Univers Gothenburg) became a head of Laboratory of NMR Spectroscopy in theCentre of Technologies, University of Warsaw. His research focuses on NMR methods based on signal processing concepts. Over the last decade, prof. Kazimierczuk's group developed new approaches to processing the NMR data from multidimensional, diffu ordered, time-resolved, pure-shift, and other experiments.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 871037

ENEA
ITALIAN NATIONAL AGENCY FOR NEW TECHNOLOGIES, ENERGY AND SUSTAINABLE ECONOMIC DEVELOPMENT

CIRMMP
FIRENZE

iNEXT DISCOVERY
Structural biology for translational research & discovery

Abstract

Protein-ligand interaction by NMR
A case of green tea polyphenols with SARS CoV-2 proteases

Dr. Miguel A Rodriguez, Technological Center of Catalonia (EURECAT)

Nuclear Magnetic Resonance (NMR) spectroscopy holds paramount importance across multiple disciplines, particularly in biology. Its versatility lies in its ability to elucidate molecular structures, dynamics, and interactions in biological systems. In biology, NMR plays a crucial role in studying proteins, nucleic acids, and metabolites, providing invaluable insights into their function, folding, and biochemical pathways (Markley et al., 2017).

Metabolomics is one of the most recent omics techniques that have emerged along with other omics such as proteomics, transcriptomics, or genomics, among others. Its integration to explain biological processes of living organisms has coined systems biology discipline. (Sauer et al. 2007) Systems biology represents an interdisciplinary approach aimed at understanding biological systems in a holistic way, considering interactions among their components at molecular, cellular, and systemic levels. In this context, the integration of genomics, transcriptomics, proteomics, and metabolomics plays a crucial role (Ideker et al., 2001).

NMR, together with mass spectrometry (MS), is widely employed in metabolomics, a field focused on studying the small molecule metabolites present in biological systems. NMR is a gold standard technique for chemical structural elucidation. Therefore, NMR-based metabolomics allows researchers to comprehensively analyze the metabolic profiles of plants, cells, tissues, and biofluids, providing valuable insights into biochemical pathways, biomarker discovery, and disease mechanisms.(Larive, C.K. et al, 2015) Besides, NMR-based metabolomics allows researchers to comprehensively analyze the diverse array of small molecules present in tissues, including primary metabolites such as sugars, amino acids, and organic acids, as well as secondary metabolites like phenolics, alkaloids, and terpenoids. These metabolites play essential roles in growth, development, immunological defense mechanisms, and interactions with other organisms. (Shen et al. 2023)

One of the key advantages of NMR-based metabolomics is its quantitative and non-destructive nature, allowing for the simultaneous detection and quantification of a wide range of metabolites without the need for extensive sample preparation. (Moco, 2022). NMR could deal with complex mixtures without extensive preparation and allow the use of stable isotopes to trace metabolic transformations of biological systems. (Vinaixa et al., 2017) This enables high-throughput analysis of large sample cohorts, facilitating the identification of metabolic signatures associated with various physiological and pathological states. Additionally, NMR metabolic profiles of different plant genotypes, varieties, or species allow identifying metabolic traits associated with specific agronomic characteristics, such as yield, stress tolerance, and nutritional value.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 871037

ENEA
ITALIAN NATIONAL AGENCY FOR NEW TECHNOLOGIES, ENERGY AND SUSTAINABLE ECONOMIC DEVELOPMENT

CIRMMP
FIRENZE

iNEXT DISCOVERY
Structural biology for translational research & discovery

Protein-ligand interaction by NMR
A case of green tea polyphenols with SARS CoV-2 proteases

Dr. Miguel A Rodriguez, Technological Center of Catalonia (EURECAT)

IApart from these applications, in NMR-metabolomics, it is also important to study ligand-macromolecules interactions, where small molecules such as metabolites (ligands) bind to larger biomolecules such as proteins, RNA, or DNA, and modulate their activities and functions. This "from-bottom-to-up flow" changes the classical downwards biological flow of information where DNA and RNA make proteins, and these modify the metabolites with their catalytic action. A powerful technique to study these ligand-protein interactions is saturation transfer difference (STD) technique. It is a specialized NMR method developed for investigating ligand-protein interactions in solution. It is particularly useful for identifying the binding epitopes of small molecules on proteins and quantifying their binding affinities (Mayer and Meyer, 2001).

The STD experiment involves the selective saturation of the signals from the protein protons. These saturated protons then transfer their magnetization to the ligands through intermolecular interactions, leading to a decrease in the intensity of ligand signals in the NMR spectrum. By comparing the spectra obtained with and without saturation, one can identify and quantify the signals arising from the bound ligand and distinguish them from the signals of the free ligand. This experiment is performed in solution without any modification of protein or ligands. This allows the study of interactions under physiologically relevant conditions, preserving the native structure and function of the biomolecules. Furthermore, STD-NMR is sensitive to weak binding events, making it suitable for screening large compound libraries to identify potential drug candidates. It can also provide information about the binding kinetics and thermodynamics, such as the association and dissociation rates and the binding constants. (Angulo, J. et al., 2010)

Polyphenols are a class of natural compounds found in various plant-based foods and beverages such as green tea, known for their antioxidant properties and potential health benefits.

In this context, NMR-STD spectroscopy offers several advantages for investigating polyphenol-protein interactions. Firstly, it allows for the characterization of both the polyphenol and protein in solution, mimicking physiological conditions.

In addition to studying individual polyphenol-protein interactions, NMR spectroscopy can also be used to investigate complex mixtures of polyphenols and proteins, such as those found in dietary supplements or natural extracts. By analyzing the NMR spectra of these mixtures, researchers can identify specific interactions and elucidate the underlying mechanisms, helping to explain the observed physiological effects. With all this in mind, a mixture of three green polyphenols (Epigallocatechin gallate (EGCG), Epicatechin gallate (ECG), and Gallocatechin-3-gallate (GCG)) and its affinity for SARS-CoV-2 protease Mpro have been studied by STD-NMR and compared with previous "docking" in-silico results (Ghosh et al., 2021) to gain insight into the molecular mechanism of interaction of these green tea compounds and the COVID virus.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 871037

ENEA
ITALIAN NATIONAL AGENCY FOR NEW TECHNOLOGIES, ENERGY AND SUSTAINABLE ECONOMIC DEVELOPMENT

CIRMMP
FIRENZE

iNEXT DISCOVERY
Structural biology for translational research & discovery

Protein-ligand interaction by NMR
A case of green tea polyphenols with SARS CoV-2 proteases

Dr. Miguel A Rodriguez, Technological Center of Catalonia (EURECAT)

Overall, NMR spectroscopy is a powerful tool for studying polyphenol-protein interactions, providing valuable insights into the molecular mechanisms underlying their biological activities. By combining the sensitivity and versatility of NMR spectroscopy with the specific capabilities of techniques like STD-NMR, researchers can advance our understanding of the health benefits of polyphenols and their potential applications in medicine and nutrition.

References:

- Angulo, J., Enríquez-Navas, P. and Nieto, P. (2010), Ligand-Receptor Binding Affinities from Saturation Transfer Difference (STD) NMR Spectroscopy: The Binding Isotherm of STD Initial Growth Rates. Chemistry – A European Journal, 16: 7803-7812. doi:10.1002/chem.200903528
- Bhunia, A., Bhattacharjya, S., & Chatterjee, S. (2012). Applications of saturation transfer difference NMR in biological systems. Drug discovery today, 17(9-10), 505-513. doi: 10.1016/j.drudis.2011.12.016
- Ghosh, R. et al. (2021) 'Evaluation of green tea polyphenols as novel corona virus (SARS CoV-2) main protease (Mpro) inhibitors – an in silico docking and molecular dynamics simulation study', Journal of Biomolecular Structure and Dynamics, 39(12), pp. 4362-4374. doi: 10.1080/07391102.2020.1779818.
- Ideker, T., et al. (2001). Integrated genomic and proteomic analyses of a systematically perturbed metabolic network. Science, 292(5518), 929-934. doi: 10.1126/science.292.5518.929.
- Larive, C.K el al (2015) NMR Spectroscopy for Metabolomics and Metabolic Profiling, Anal. Chem. 2015, 87, 1, 133-146 doi: 10.1021/ac504075g
- Markley, J.L., et al. (2017). The future of NMR-based metabolomics. Current Opinion in Biotechnology, 43, 34-40. doi: 10.1016/j.copbio.2016.08.001
- Mayer, M., & Meyer, B. (2001). Characterization of ligand binding by saturation transfer difference NMR spectroscopy. Angew. Chem Int Ed, 40(15), 2957-2959. doi: 10.1002/(SICI)1521-773(19990614)38:12<1784: AID-ANIE1784>3.0.CO;2-Q.
- Moco, S. (2022) Studying Metabolism by NMR-Based Metabolomics. Front. Mol. Biosci. 9:882487. doi: 10.3389/fmolb.2022.882487
- Sauer, U., Heinemann, M., & Zamboni, N. (2007). Genetics. Getting closer to the whole picture. Science, 316(5824), 550-551. https://doi.org/10.1126/science.1142502
- Shen, S., Zhan, C., Yang, C., Fernie, A. R., & Luo, J. (2023). Metabolomics-centered mining of plant metabolic diversity and function: Past decade and future perspectives. In Molecular Plant Vol. 16, Issue 1, pp. 43-63). Elsevier doi: 10.1016/j.molp.2022.09.007
- Vinaixa, M., Rodríguez M.A. at al. (2017), Positional Enrichment by Proton Analysis (PEPA): A One-Dimensional 1H-NMR Approach for 13C Stable Isotope Tracer Studies in Metabolomics. Angew. Chem. Int. Ed. 2017, 56, 3531-3535 doi:10.1002/anie.201611347

Miguel A Rodriguez

Dr. Miguel A Rodriguez has his degree in Chemistry, Biochemistry, and his Ph.D. thesis in organic synthesis of natural products. Currently he is working at Center of Omic Sciences (COS), a joint unit of Technological Center of Catalonia (EURECAT) and University of Tarragona (URV). As part of his role as NMR-metabolomics researcher, he is developing new NMR analysis to characterize metabolites, to track stable isotopes in biological models and to study dynamics interactions to give valuable biochemical, nutritional, and medical data.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 871037

ENEA
ITALIAN NATIONAL AGENCY FOR NEW TECHNOLOGIES, ENERGY AND SUSTAINABLE ECONOMIC DEVELOPMENT

CIRMMP
FIRENZE

iNEXT DISCOVERY
Structural biology for translational research & discovery

Abstract

Functional properties of whey proteins and their hydrolysates

Špela Gruden and Nataša Poklar Ulrih, University of LjubljanaDr. Miguel A Rodriguez, Technological Center of Catalonia (EURECAT)

Whey is the most important by-product of cheese and casein production, which is one of the greatest environmental burdens of the food industry due to its uneconomical management. However, whey is a rich source of proteins such as lactoferrin (Lf), lactoperoxidase (LPO) and beta-lactoglobulin (BLG) with many structural and functional properties that have great potential for use in the food and pharmaceutical industries.

By enzymatic hydrolysis of lactoferrin with different enzymes and enzymatic reaction conditions, we obtained various hydrolysates with antibacterial and antioxidant activity and demonstrated for the first time the antibacterial activity of Lf and Lf peptides against Latilactobacillus sakei.

The structural and enzymatic activity of LPO depended on pH, temperature, type and molar concentration of buffer, while the presence of 10 mM Ca2+, Mg2+ and Na+ only affected the structural stability of LPO. Partial removal of Fe3+ and Ca2+ from LPO resulted in lower temperature stability and lower enzymatic activity of LPO.

Beta-lactoglobulin forms gels by the heat and cold gelation method, into which we successfully encapsulated vitamin D3 and Lf peptides. The gels formed under different conditions showed different stability in simulated gastric fluid due to structural differences. In addition, we were able to show that BLG A binds two vitamin D3 molecules at different pH values, which influences its quaternary structure.

With the investigations carried out, we were able to demonstrate many positive physicochemical and functional properties of the proteins studied, which will make a positive contribution to the efficient utilisation of whey.

Nataša Poklar Ulrih

Prof. Dr. Nataša Poklar Ulrih is a full professor of biochemistry at Biotechnical Faculty University of Ljubljana. She obtained her PhD in 1994 at Faculty of Chemistry and Chemical Technology at University of Ljubljana. As a post-doctoral fellow, she had an appointment at The Rutgers, The State University of New Jersey, as a visiting professor at University of Toronto and University of California Santa Cruz (as Fulbright fellow). She has been coordinating the research program and many national and international projects. Her research interests are the interactions of small molecules with biological macromolecules, identification of small molecules, polyphenols and other natural compounds, developing the new technologies (nanobased) for encapsulation of small molecules and plant extracts. She has published more than 200 peer review articles.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 871037

Abstract

Combining high-resolution mass spectrometry and nuclear magnetic resonance to improve metabolite detection in response to agro-sustainable treatments

Federica Bianchi, University of Parma

Metabolomics is based on cutting-edge analytical techniques providing a snapshot of small molecules present in complex biological samples, being nuclear magnetic resonance (NMR) and high-resolution mass spectrometry (HRMS) the techniques of choice for running omics experiments. In contrast to targeted metabolomics, which is focused on the identification and quantitation of a limited number of defined metabolites, the untargeted approach aims at acquiring data related to all ions within a certain mass range, thus providing a comprehensive pattern of metabolites present in complex biological samples.

Advances in HRMS allowed a comprehensive profiling of food samples, representing a valuable technique for studying the metabolic changes developed by organisms in the presence of environmental and physiological factors. Ultra-high performance liquid chromatography coupled to HRMS (UHPLC-HRMS) currently represents the best tool to face challenges related to the complexity of metabolome [1]. UHPLC provides efficient chromatographic separation of compounds, whereas HRMS is characterized by unparalleled specificity, sensitivity, and availability of large spectral databases. In addition, ion mobility spectrometry (IMS) offers great potential for improving depth of coverage in metabolomic studies, separating ions according to their collisional cross-section (CCS) and providing unique features for metabolite identification.

As for NMR, it is non-destructive technique, requires minimal sample preparation and allows for absolute quantitation. However, it is affected by sensitivity issues, since only compounds at micromolar/millimolar concentration levels can be detected.

Despite their complementarity, to date, metabolomics studies have been carried out using either HRMS or 1H-NMR: however, the use of a single platform does not allow a full coverage of the metabolome.

The outcomes of metabolomics can be successfully translated to agricultural research to assess the availability of natural resource sustainability. Sustainable agriculture aims to achieve a healthy food production while reducing the use of fertilizers, pesticides, and greenhouse gasses emission. In this context, metabolomics has frequently been applied to detect alterations in plants exposed to different treatments and to disclose the effects related to innovative and more sustainable treatments for crop cultivation.

The aim of the study herein proposed is the identification of biomarkers able to differentiate among the treatments with biostimulants in durum wheat (cultivar Svevo) grain, one of the most important cereal crops of Mediterranean region. In fact, although durum wheat is an important staple food for human nutrition, the studies that involve the metabolomics of durum wheat are less than 10% of those of wheat in general.

The combined use of UHPLC-HRMS and 1H-NMR is proposed as a promising strategy for metabolomics studies by increasing the metabolic coverage.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 871037

ENEA
ITALIAN NATIONAL AGENCY FOR NEW TECHNOLOGIES, ENERGY AND SUSTAINABLE ECONOMIC DEVELOPMENT

CIRMMP
FIRENZE

iNEXT DISCOVERY
Structural biology for translational research & discovery

Combining high-resolution mass spectrometry and nuclear magnetic resonance to improve metabolite detection in response to agro-sustainable treatments

Federica Bianchi, University of Parma

Untargeted metabolomics by UHPLC-HRMS was applied to investigate the metabolome of durum wheat in response to sustainable treatments, i.e., the addition of biochar, commercial plant growth promoting microbes, and their combination. After data filtering, 3587 and 4686 features were extracted for 2020 and 2021 wheat grain, respectively. Both unsupervised (PCA) and supervised (PLS-DA) analyses were performed: clustering of the samples according to the treatments was obtained, and the discrimination was assessed by k-fold cross validation. Variable important in projection (VIP>2) were extracted and used for PLS-DA, obtaining a good separation among the treatments. K-fold cross validation showed sensitivity, specificity, and a non-error rate close to 1. The features responsible for the differentiation were identified by comparing the data with online databases, considering the accurate mass of both precursor and fragment ions, the isotope similarity, the CCS value and the score fit. A total of 88 and 45 discriminant compounds having biological, nutritional, and technological implications were tentatively identified in samples grown in 2020 and 2021 [2]. 1H-NMR data obtained on the same samples by applying two different extraction procedures were combined with UHPLC-HRMS data using both a low level and a mid-level data fusion approach to test the possibility of a more comprehensive metabolome coverage.

The findings achieved using the combined analytical approach suggest the safe use of the combined biochar-biostimulant treatment for sustainable wheat cultivation. The addition of biochar-biostimulants produced the highest up-regulation of lipids and flavonoids, with the glycolipid desaturation being the most impacted pathway, whereas carbohydrates were mostly down-regulated. Additional expected benefits based on increased use of natural amendments and biofertilizers will rely on: i) the reduction in both water consumption and greenhouse gas emissions, ii) a considerable decrease in the carbon footprint of food production, and iii) a reduction of soil pollution due to a lower use of fertilizers. Finally, the results achieved in this study could pave the way for the establishment of harmonized conditions to make fertilizers based on recycled or organic materials available on the market.

[1] Ultra-high performance liquid chromatography high-resolution mass spectrometry for untargeted metabolomic analysis of dental calculus from Duke Alessandro Farnese and his wife Maria D'Aviz
N. Riboni, F. Bianchi, M. Mattarozzi, M. Peracchia, M. Meleti, M. Careri, Sci Reports 13 (2023) 8967
[2] Ultra-high performance liquid chromatography-ion mobility-high-resolution mass spectrometry to evaluate the metabolomic response of durum wheat to sustainable treatments
N. Riboni, F. Bianchi, M. Mattarozzi, M. Caldara, M. Gulli, S. Graziano, E. Maestri, N. Marmiroli, M. Careri, J. Agric. Food Chem. 71 (2023) 15407-15416

Federica Bianchi

Federica Bianchi is Full Professor in Analytical Chemistry at the Dept. of Chemistry, Life Sciences and Environmental Sustainability of the University of Parma, Italy. The main research topics include the development and validation of analytical and bioanalytical methods based on hyphenated techniques in environmental and food analysis and innovation in materials for sample treatment devices and mass-spectrometry instrumentation. Expertise in chemometrics is widely exploited for data elaboration.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 871037

ENEA
ITALIAN NATIONAL AGENCY FOR NEW TECHNOLOGIES, ENERGY AND SUSTAINABLE ECONOMIC DEVELOPMENT

CIRMMP
FIRENZE

iNEXT DISCOVERY
Structural biology for translational research & discovery

Abstract

Metabolomics for pathway discovery and food quality improvement

Gianfranco Diretto
ENEA, C.R. Casaccia

Metabolic engineering has revolutionized classic breeding, allowing the production of new plant materials enriched or devoid of molecules of pro- and anti-nutritional interest, respectively; historically, the greatest efforts and the most promising results have concerned the obtaining of lines of species of agricultural interest (rice, corn, potato and tomato) in which the vitamin content (A, B, C, E, K) has been increased or accumulated de novo. Subsequently, metabolic engineering found a further field of application in the use of plants as biofactories ("molecular agriculture") for the production of metabolites with high added value and, therefore, of strong industrial interest (pharmaceutical, cosmetics, etc).
In this context, ENEA has a long tradition in the field of metabolic engineering, specifically of carotenoids and, more recently, of apocarotenoids: after a general introduction and on the main metabolic engineering strategies for different classes of natural molecules, there will be presented (i) the approaches that led to the creation of potato and tomato lines enriched in b-carotene and total carotenoids (therefore defined as "Golden"); (ii) characterization studies, through the use of omics sciences, of these materials, with particular reference to the accessory effects on metabolism and agronomic traits; (iii) the "molecular farming" strategies that have led to the generation of plants (Nicotiana benthamiana, potatoes and tomatoes) capable of accumulating saffron apocarotenoids. Finally, the research developed in ENEA for the generation and characterization of tomato lines free of anti-nutritional molecules (glycoalkaloids and allergens), produced through genome editing technology, will be presented.

Gianfranco Diretto

Dr. Diretto got his Laurea degree in Plant Biotechnology with full marks in 2002 at the University of Naples "Federico II", Italy. He holds a Ph.D. in Cell and Molecular Biology at the University of L'Aquila and spent 16 months (2006-2007) at Boyce Thompson Institute (BTI), Ithaca (NY), USA, and 3 months (2008) at University of Freiburg, Germany, 10 months (May-December 2015) at the Istituto de Biologia Molecular y Celular de Plantas (IBMCP), Valencia, Espana, and 2 months (May-June 2017) at INRA-INP Research Unit, working on omics sciences (transcriptomics and metabolomics), bioinformatics and pharmacological treatments and physiological analyses of tomato fruits.
Since December 2008, he has a Junior Research Scientist position at ENEA, where he presently is responsible for the LC-HRMS Metabolomics facility and for a series of activities of plant and microalgae metabolic engineering. Since March 2022, he is head of the ENEA Biotechnology Unit.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 871037

ENEA
ITALIAN NATIONAL AGENCY FOR NEW TECHNOLOGIES, ENERGY AND SUSTAINABLE ECONOMIC DEVELOPMENT

CIRMMP
FIRENZE

iNEXT DISCOVERY
Structural biology for translational research & discovery

Abstract

NMR relaxometry for accessing food quality and authenticity

Danuta Kruk, Department of Physics and Biophysics, University of Warmia and Mazury in Olsztyn

From the point of view of molecular science, food products are very complex systems containing several fractions of interacting molecules of very different structure. The macroscopic properties of foods are determined not only by their composition, but also (or even primarily) by their molecular arrangement and dynamics.

Nuclear Magnetic Resonance (NMR) methods are widely appreciated and used as very valuable tools to reveal dynamical and structural properties of a variety of molecular systems, from simple liquids to macromolecules (polymers, proteins) to tissues.NMR relaxation plays a special role in such studies. In contrast to "classical" relaxation experiments, which are performed at a single magnetic field, Fast Field Cycling (FFC) technology allows the magnetic field to be varied over a wide range. The unique advantage of covering a broad range of magnetic fields (resonance frequencies) is the possibility to probe dynamical processes occurring on much different time scales (from milliseconds to nanoseconds) in a single experiment. Already at this stage one can see the exceptional potential of NMR relaxometry – the method can be used to reveal dynamical properties of different molecular fractions present in food products.

It is much easier to counterfeit the composition of food products than the molecular arrangement that results from specific technological procedures. Following this line – compromised quality of food products is also related to alterations in molecular arrangement (and, hence, molecular dynamics). Consequently, one can determine characteristic relaxation properties (markers) of food products that can be treated as fingerprints of their authenticity and quality. This concept has been verified for different kinds of food with special focus on sugar and dairy products, as well as oil and honey.

The key to identifying the characteristic markers is the fact that in addition to providing information about the timescale of the molecular motion, the shape of the frequency dependencies of 1H spin-lattice relaxation rates reflects the mechanism of the molecular motion. To be more specific, one can, for instance, distinguish between isotropic diffusion of water molecules and cases when the macromolecular arrangement prevents small (water) molecules to move freely in all directions. One can describe the confinement effects in a quantitative way – for instance one can determine the effective translation diffusion coefficient and/or the residence lifetime on the macromolecular surface (the exchange lifetime).

After providing a short introduction to the principles of NMR relaxometry, I will illustrate the potential of this method by several examples and demonstrate how to obtain quantitative information about dynamical properties of different molecular fractions in a straightforward way.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 871037

ENEA
ITALIAN NATIONAL AGENCY FOR NEW TECHNOLOGIES, ENERGY AND SUSTAINABLE ECONOMIC DEVELOPMENT

CIRMMP
FIRENZE

iNEXT DISCOVERY
Structural biology for translational research & discovery

NMR relaxometry for accessing food quality and authenticity

Danuta Kruk, Department of Physics and Biophysics, University of Warmia and Mazury in Olsztyn

The key to identifying the characteristic markers is the fact that in addition to providing information about the timescale of the molecular motion, the shape of the frequency dependencies of 1H spin-lattice relaxation rates reflects the mechanism of the molecular motion. To be more specific, one can, for instance, distinguish between isotropic diffusion of water molecules and cases when the macromolecular arrangement prevents small (water) molecules to move freely in all directions. One can describe the confinement effects in a quantitative way – for instance one can determine the effective translation diffusion coefficient and/or the residence lifetime on the macromolecular surface (the exchange lifetime).

After providing a short introduction to the principles of NMR relaxometry, I will illustrate the potential of this method by several examples and demonstrate how to obtain quantitative information about dynamical properties of different molecular fractions in a straightforward way.

Danuta Kruk

Danuta Kruk – physicist interested in spin resonance phenomena from both theoretical and applied points of view. She applies NMR relaxometry in combination with other NMR methods (diffusometry, spectroscopy) complemented with dielectric spectroscopy and ESR to get deep insight into molecular and ionic dynamics of complex systems, exploiting a deep theoretical framework of these methods.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 871037

ENEA
ITALIAN NATIONAL AGENCY FOR NEW TECHNOLOGIES, ENERGY AND SUSTAINABLE ECONOMIC DEVELOPMENT

CIRMMP
FIRENZE

iNEXT DISCOVERY
Structural biology for translational research & discovery

Abstract

NMR and Food Matrix Studies: Structure, Bioaccessibility and Digestion

Carlo Mengucci, Bio-NMR Group, DISTAL University of Bologna

Food is a complex matter, literally. Studying and characterizing food structure and function, involves the investigation of a plethora of different length scales and phenomena unfolding at very different time scales. In this presentation, a brief overview on how to characterize food matrix structure and its interaction with the human digestive system, with a focus on NMR techniques, is provided. First, a framework to integrate SEM imaging with TD-NMR to study structure and water mobility/dynamics in cooked pasta is presented. Then, a study on bioaccessibility coupling in-vitro digestion and omic techniques is discussed. Finally, an example of fingerprinting individual variability in digestion of various foods in a cohort that was administered a nutritional intervention, is proposed.

Carlo Mengucci

Carlo Mengucci is a researcher at Bio-NMR Group, DISTAL, University of Bologna. With a background in statistical and mathematical physics applied to omic sciences, its research activity focuses on machine learning for 1H NMR Spectroscopy metabolomics, omic data modelling, TD-NMR and data integration for food structure characterization.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 871037