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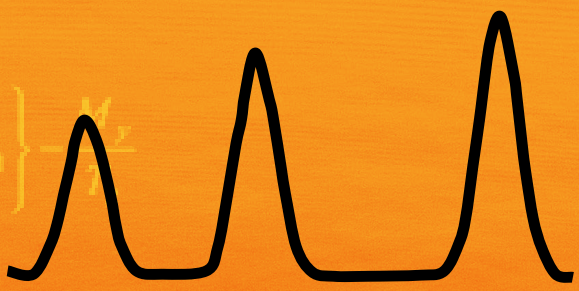

AMPERE NMR SCHOOL

BOOK OF ABSTRACTS

18th – 24th June 2023
ZAKOPANE, POLAND

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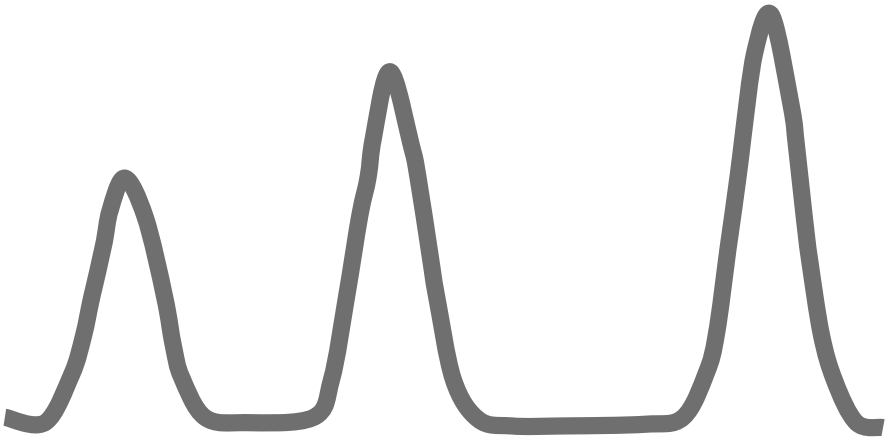

AMPERE NMR SCHOOL

BOOK OF ABSTRACTS

ZAKOPANE

18th – 24th June 2023

AMPERE NMR SCHOOL 2023

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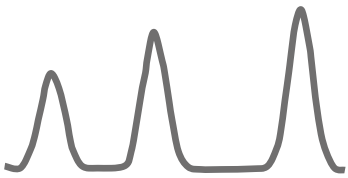

AMPERE **NMR** School

Solid-state NMR of the Multidrug ABC Transporter BmrA

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Membrane proteins presented an interesting target for solid-state NMR studies, since they could be studied in a lipid environment. Also, NMR can reveal chemical properties of the protein, by chemical-shift perturbations caused by binding events. Molecular motors are interesting in this context, since they use ATP as a fuel to power molecular machines. ABC transporters are such machines, since they act as pumps to import and export molecules from cells. This is for instance important in antibiotic resistance, where these large proteins remove the antibiotics from bacterial cells. The pumping mechanisms proceeds as a function of ATP hydrolysis.

The detailed mechanism of ATP hydrolysis in ATPbinding cassette (ABC) transporters is still not fully understood. We here present results from solid-state NMR which we used to probe the conformational changes and dynamics during the catalytic cycle by locking the multidrug ABC transporter BmrA in prehydrolytic, transition, and posthydrolytic states, using a combination of mutants and ATP analogues. The NMR spectra reveal that ATP binds strongly in the prehydrolytic state to both ATP-binding sites, but that in the transition state of wild-type BmrA, the symmetry of the dimer is broken and only a single site is tightly bound to ADP:Mg2+:vanadate. In the posthydrolytic state, weak binding is observed for both sites. Our results show that after ATP binding, the symmetry of the ATP-binding sites of BmrA is lost in the ATP-hydrolysis step, but is then recovered in the posthydrolytic ADP-bound state.

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Acknowledgements
We thank the French ANR and ANRS for support.

BICOMPONENT FLUIDS IN POROUS MEDIA: WHAT NMR CAN DO FOR YOU

Siegfried Stapf, Niklas Siebert, Timo Spalek, Vincent Hartmann, Bulat Gizatullin, Carlos Mattea

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The theoretical description of the T_1 dispersion of liquids in metal-free porous systems has been established decades ago, distinguishing between the limiting cases of strong and weak adsorption of the molecules on the native glass surface [1]. The model predicts vanishing T_1 dispersion in the weak adsorption case, and strongly pronounced dispersion for those molecules that encounter preferential orientation on the surface, experiencing the surface curvature via anomalous diffusion. Therefore, this process is intramolecular by nature and is expected to lead to identical dispersions for protons and deuterons [2].
In very small pores, such as in the 4 nm pores of Vycor or on the surface of silica particles of similar size, T_1 dispersion is present for all liquids, indicating non-negligible surface interaction. We have carried out ^{2}H relaxation dispersion measurements of perdeuterated liquids and ^{1}H and ^{2}H studies of binary systems of two fluids in Vycor that are either miscible or immiscible in the bulk, e.g. protic (water, ethanol) and aprotic liquids (acetone, THF, cyclohexane, n-alkanes) [3,4]. Combining NMRD relaxation between 0.2 mT and 1 T with relaxation and diffusion experiments at 7 T, a particularly strong ^{2}H relaxation dispersion was observed that is at variance with either of the established relaxation models. Moreover, we found a strong tendency of preferential surface adsorption of the non-polar liquid, but also an apparent phase separation between bulk miscible liquids such as acetone and cyclohexane, suggesting that the latter is entirely removed from the silica surface.

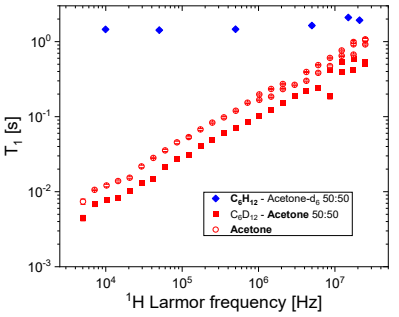

FIGURE 1. Relaxation dispersion $T_1(\nu)$ of the ^{1}H nuclei of liquids in Vycor porous glass for partially deuterated mixtures of acetone and cyclohexane at 50%:50% volume fraction compared with pure acetone in Vycor.

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Protein fast MAS NMR at high field

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The NMR resonances of protein side chain protons provide important information not only about protein structure and dynamics, but also about the mechanisms that regulate interactions between macromolecules. However, these resonances are particularly difficult to resolve spectrally, even in relatively small proteins. The recent development of MAS frequencies up to 160 kHz has been shown to significantly improve resolution. Indeed, NMR spectroscopy, the only technique capable of accessing interactions and dynamic processes in a sample under physiological conditions, has so far been limited when applied to large protein assemblies. We characterise the gain in resolution by increasing the MAS frequency from 110 to 160 kHz MAS frequencies and find a drastic improvement in the spectral resolution of this crowded region in the 1H-13C dimensions, allowing the identification of these essential proton resonances in fully protonated samples. These improvements allowed us to assign 60% of the aliphatic protons of the core protein (Cp), which is organised in a large 120-dimer assembly to form the hepatitis B virus capsid. This opens up new possibilities for further characterisation of protein-protein or protein-nucleic acid interations.

The magical world of pure shift NMR

Adolfo Botana[a], Peter Kiraly[b] and Paul Bowyer[a]

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NMR is an enormously powerful analytical technique, yielding unrivalled insights into the chemical structure and dynamics of organic molecules. Unfortunately, the information that can be readily extracted from the NMR spectrum is sometimes limited by overlap between neighboring peaks, a problem which is often exacerbated by the presence of broad peak "multiplets" due to J-splittings. This is particularly a problem in proton spectra, where the narrow chemical shift range and manifold J-couplings between protons can lead to severe peak overlap. In recent years, much work has been done to develop NMR methods that yield spectra where the peak multiplet structure is completely suppressed; these so-called "pure shift" spectra show much higher resolution than conventional spectra [1]. We introduce the basic principles of pure shift NMR and describe some of the most important pure shift methods that have been developed to date.

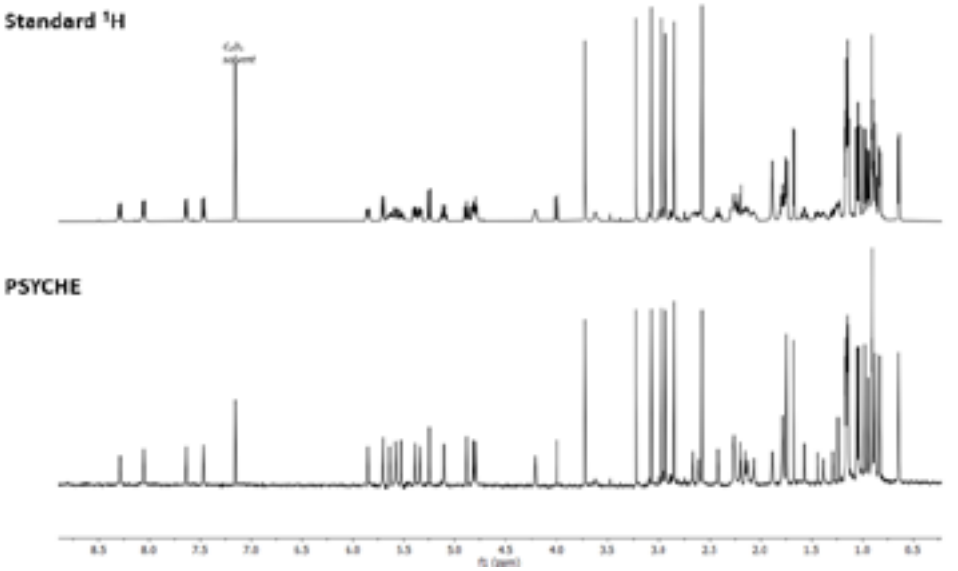

FIGURE 1. Standard single proton pulse acquisition and a pure shift spectrum of cyclosporine.

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High dimensionality and high resolution NMR experiments for biomolecules

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Studies of biomolecular structure and dynamics by NMR spectroscopy at atomic resolution require acquisition of multidimensional spectra. However, the recording time of sufficiently resolved multidimensional spectra is often very long due to the sampling limitations. A variety of different methods, mostly based on non-uniform sampling, were proposed to overcome this limitation in multidimensional NMR spectroscopy. They could be utilized in two different ways, either to shorten the experiment duration without loss of resolution, or to perform experiments that are not obtainable conventionally, i.e. with significantly improved resolution and/or of high dimensionality. Most often first of these two, so called "Fast NMR" approach, is shown as the example of the utility of these methods, as it saves expensive spectrometer time. However, in many cases spectra which are not possible to record conventionally, featuring extraordinary resolution and high number of dimensions may be more interesting from scientific point of view as they reveal effects that are hidden, when spectral lines are broad, or enable resolving spectral ambiguities when peaks are overlapped. This second approach we refer to as "Accurate NMR". Its full potential is manifested when the overall experiment time is less important than a new information available from spectra of high dimensionality (4-6D) or of high resolution approaching natural line-width. The new methods were applied for NMR studies of intrinsically disordered proteins, where the structural disorder in combination with highly repetitive amino-acid sequences causes severe peak overlap in the spectra. Several novel 4-7D pulse sequences are proposed. The new experiments employ non-uniform sampling that enables achieving high resolution in indirectly detected dimensions.

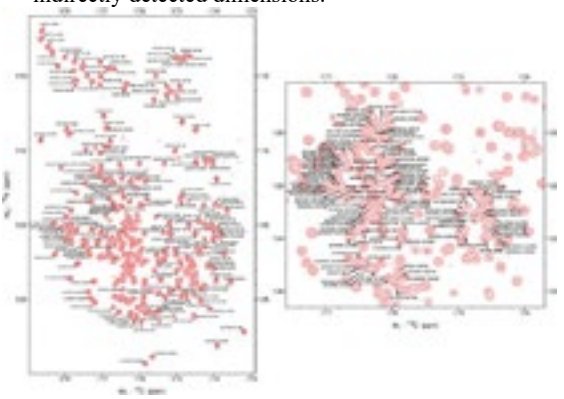

Figure 1 Resonance assignment of Tau3x (354 aa) shown on CON projection from 3D HNCO [1]

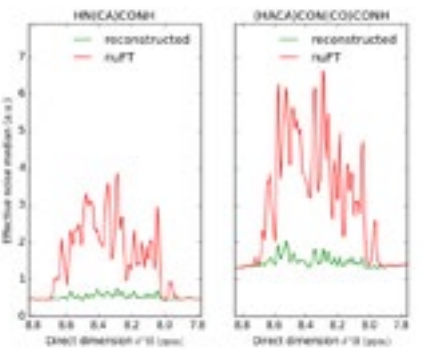

Figure 2 Noise median for 5D HN(CA)CONH (*left*) and 5D (HACA)CON(CO)CONH (*right*) SSA-reconstructed and nuFT spectra with respect to direct dimension ^{1}H chemical shift [2]

Acknowledgements

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Practical aspects of ^{13}C qNMR

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Quantitative NMR is one of the most important tools for the quantification of chemical species in samples. It is most commonly run through the measurement of ^{1}H detected single pulse experiments[1], but severe spectral overlap makes the quantification of analytes a challenging task. ^{13}C detected NMR alleviates the spectral overlap problem, and with modern instruments achieving higher sensitivity, ^{13}C qNMR becomes a suitable alternative to ^{1}H qNMR. For example, the resolution achieved in ^{13}C data enables the determination of long-chain branching in polymers[2].
Several aspects such as the acquisition of data with low signal-to-noise ratio and the larger bandwidth required to excite the ^{13}C spectrum lead to a number of considerations that must be taken into account when acquiring ^{13}C qNMR. Here we describe some of the practical aspects and consideration to take into account to acquire accurate ^{13}C qNMR data[3].

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USE OF A BENCHTOP MAGNETIC RESONANCE IMAGING SYSTEM FOR FOOD APPLICATIONS

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Magnetic Resonance Imaging enables us to visualize the interior tissues of food samples and thus it becomes possible to observe the infected tissues in fresh produce, oil/water distribution in high-fat foods, moisture uptake during storage, and many other processes relevant to food science and processing.
In addition, it can be used to conduct flow imaging, and an MRI system can be transferred to an online viscometer. In this study, steps on how to conduct an image will be explained using a benchtop MR imaging system. T_1, T_2 measurements, Spin Echo Imaging, T_2 mapping, and T_1 mapping will be discussed with examples. In addition, a flow MR imaging experiment will be performed to calculate the rheological properties of some food samples.

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BASICS OF MRI & FIELD-CYCLING IMAGING

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MRI uses magnetic field gradients to encode spatial information into NMR signals. In frequency-encoding, the NMR signal is recorded while a field gradient is applied. Since the magnetic field varies with position along the gradient direction (e.g. X), Larmor frequency is a function of position, so the detected signal contains a range of frequencies; analysing the frequency content generates a one-dimensional projection of the water-distribution within the patient. Phase-encoding is employed in the second in-plane dimension (e.g. Y); here, the gradient is pulsed on and off prior to measurement of the signal, altering the phase of the NMR signal as a function of position. The image slice is defined using selective-excitation, in which the excitation 90° radiofrequency pulse is shaped (typically a sinc function) and is applied in the presence of a field gradient perpendicular to the slice plane (e.g. along Z for a transaxial X-Y slice). An excellent primer textbook on MRI has been published by McRobbie *et al*. [1].

During the last decade, the laboratory in Aberdeen has focused on the development of Field-Cycling Imaging (FCI), based on combining MRI with Fast Field-Cycling (FFC) magnetic resonance. By switching field strength during an experiment, this technique exploits the variation of T_1 with magnetic field (T_1-dispersion), with the aim of increasing the diagnostic potential of MRI [2,3]. FCI aims to obtain spatially-resolved T_1-dispersion data, by collecting images at a wide range of evolution field strengths. A range of FCI equipment has been built, including two whole-body human sized scanners, operating at detection fields of 0.06 T [4] and 0.2 T [5]. The 0.2 T system uses a single resistive magnet, composed of three coaxial coils.

Results have shown that FFC methods can detect changes in human cartilage induced by osteoarthritis [6]. Experiments on resected tissues from breast cancer patients have demonstrated significant differences in the dispersion curves between normal and diseased tissues [7]. Studies on patients with acute ischaemic stroke have been performed; FCI images exhibited increased intensity in stroke-affected regions, with maximum contrast typically at the lowest field used (0.2 mT) [8]. Studies on patients with brain cancer and patients with breast cancer are underway. All human studies were conducted following approval of the relevant Research Ethics Committees and with informed consent. Work to improve the hardware and software is ongoing, including the implementation of improved RF coils [9].

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EFFECT OF SURFACTANT AGGREGATION ON CLOUD FORMATION

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Organic surfactants, which are common constituents of atmospheric aerosols, change both surface tension and water activity of cloud droplets. The aggregation of surfactants significantly affect the cloud droplet growth, inducing yet not well-known potential effects on global climate.

This work aims at improved understanding of aggregation phenomena of surfactants in aqueous solutions as well as the effect of aggregation on cloud droplet growth. We combine advanced relaxation and diffusion NMR techniques with state-of-the-art relaxation modelling, in which dynamic and structural parameters are extracted from MD simulations, to elucidate the aggregation of surfactants. Aqueous sodium decanoate was used as a representative model system. Furthermore, we use thermodynamic models and Köhler theory to estimate the effect of aggregation on growth of cloud droplets.

Contrary to the common understanding, our NMR analysis implies that surfactants form small clusters below the critical micelle concentration (CMC). Furthermore, it shows that the micelle size above the CMC is significantly smaller than the value provided by a conventional empirical model. Water diffusion experiments revealed water-encapsulating aggregates at higher concentrations and exchange rates between the encapsulated and free water pools were determined by ultrafast diffusion exchange spectroscopy. Thermodynamic modelling of aerosol hygroscopic growth and cloud droplet formation showed that the aggregation phenomena may significantly affect the hygroscopic growth factors at lower humidity conditions, leading to misrepresenting the aerosol cooling potential. [1,2]

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OBSERVING SHORT-RANGE ORIENTATIONAL ORDER: FROM LIQUID CRYSTALS TO SMALL-MOLECULE LIQUIDS

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We used NMR relaxometry to demonstrate the existence of dynamic clusters with short-range orientational order in nominally isotropic liquids consisting of elongated molecules. In those clusters the local ordering is driven by polar, steric, and hydrogen-bond interactions between the molecules. In the case of liquid crystals [1-3], measuring the local orientational order fluctuations allowed us to observe the size of these clusters diverging when approaching the phase transition from the isotropic to the nematic phase. These fluctuations are described in terms of rotational elasticity as a consequence of the correlated reorientations of the neighbouring molecules. Our quantitative observations of the dynamic clusters in liquids, numbering about ten or fewer molecules, indicate that this is a general phenomenon in various types of liquids [4].

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LOW FIELD MRI SOLUTIONS: FIXED FIELD, PRE-POLARIZED OR FIELD-CYCLED?

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The development of low-cost & low-field MRI solutions is a hot topic worldwide by these days. A main reason stimulating this increased activity in the field relies in the possibility to popularize the access of MRI for human diagnosis. However, there is also an in-progress impact in education with possibilities for training in engineering, radiology and biomedical sciences, basic & applied research and industrial applications [1]. We will analyze general technical issues of fixed-field instruments operating at low-field. Then we will discuss pros and cons of the pre-polarized approach, from both physical and technical perspectives. Permanent magnet and electromagnet technology will be confronted. Finally, magnetic field-cycling is introduced as an alternative technique, where field-dependent relaxation and MRI experiments are usefull for the development of new contrast mechanisms [2,3]. As field cycled machines usually deals with switched currents in electromagnets, magnetic field instability and inhomogeneity are the main limiting factors affecting image quality [4,5].

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Asymmetry in Three-Site Relaxation-Exchange NMR

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Asymmetry of peak integrals in 2D relaxation maps of exchange between three sites reports circular flow between the relaxation sites. This disagrees with detailed balance according to which the exchange between any pair of sites must be balanced in thermodynamic equilibrium. Confined diffusion of particles jumping randomly under thermodynamic constraints on a 2D checkerboard grid to any of their eight neighbor positions and confined gas diffusion were modelled to explore the impact of topological constraints on diffusion. Both models produce density variations across the pore. The checkerboard simulations reveal that up to 1% of the molecules may move in circular paths near the walls. This motion is argued to result from stochastic pore resonance corresponding to diffusion eigenmodes. If confirmed by experiment, it can be concluded with certainty that T_2-T_2 exchange maps can be asymmetric, and that detailed balance of multi-site exchange is strictly valid only in the absence of topological constraints. The lecture will discuss the significance of detailed balance in textbook physics, how it can be probed in relaxation exchange NMR experiments, and the Monte Carlo simulations executed to test the validity of the principle.

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TRANSLATIONAL MOTION AND MAGNETIC FIELD GRADIENTS

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Micrometer-scale structures in porous media are imprinted on the time/frequency-dependent (“restricted) and anisotropic self-diffusion of the pore liquids, which may be investigated with magnetic resonance methods employing magnetic field gradients to encode the signal with information about translational motion. The simplest and most commonly used methods rely on a pair of gradient pulses to study displacements in the direction of the gradient [1]. These methods are sensitive to a wide range of fundamentally different aspects of motion, including bulk diffusivity, restriction, anisotropy, flow, and exchange. While it may be convenient to have a single method to study multiple phenomena, the lack of specificity prevents unambiguous microstructural interpretation of the data [2]. More detailed information can be obtained by capitalizing on the formal analogies between investigating translational motion with magnetic field gradients and measuring rotational correlation functions and interaction tensor anisotropies in solid-state NMR spectroscopy [3]. This lecture includes the basic relations between structure and translational motion in porous media, the physical principles of using pulsed and modulated gradients to separate and correlate specific aspects of translational motion, and examples of applications to liquid crystals, yeast cells, and brains as illustrated in Figure 1.

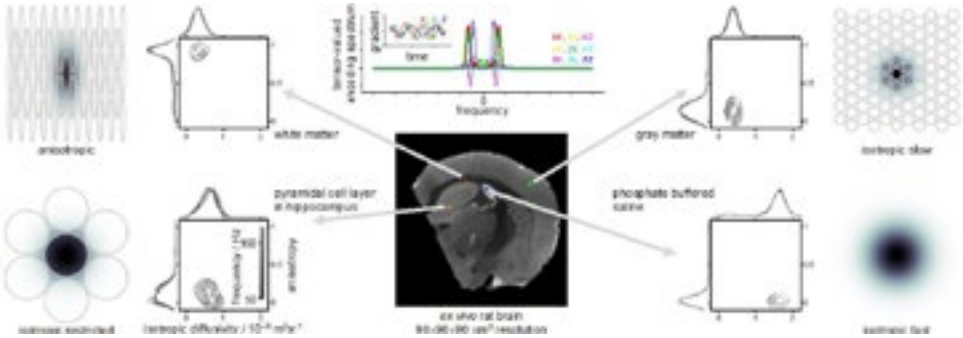

FIGURE 1. Diffusion encoding by double-rotation gradient waveform [4] (top center) and frequency-dependent diffusion tensor distributions for selected voxels of an *ex vivo* rat brain. The multidimensional distributions are visualized as projections onto the 2D plane of isotropic diffusivity and normalized anisotropy for a series of frequencies (gray scale of contour lines), where restriction results in a shift of the distribution with frequency (bottom left). For each voxel, the underlying tissue structure is shown schematically with gray lines representing partially permeable cell membranes and “ink stains” illustrating water diffusion during the 10 ms timescale of diffusion encoding.

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Exploring NMR Laboratory Operations in Medicinal Chemistry: Insights from Everyday Challenges at Celon Pharma SA

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Working in a pharmaceutical company's NMR lab combines the need for fast data analysis and accurate results, particularly in the medicinal chemistry department. The intricacies of this work differ from those found in academic settings, and the solutions employed are uniquely tailored to the fast-paced world of drug discovery.

In this presentation, I will provide an insider's perspective on our NMR laboratory at Celon Pharma SA. From compound characterization to reaction monitoring, NMR spectrometers are crucial tools that enable us to analyze and validate our synthesized molecules. My talk will focus on presenting practical aspects such as quantitative analysis and stoichiometry control. Furthermore, I will delve into a few interesting examples of structural analysis problems. I will also demonstrate how to effectively use titration methods and variable temperature measurements to obtain desired results and plan experiments. Additionally, I will briefly discuss how NMR can be used to screen libraries of small-molecule compounds and how to work without the use of deuterated solvents for rapid analyses of reaction mixtures straight from the reaction flask. Moreover, I will present the spectrometers we work with and the software that facilitates our daily tasks. I will also discuss our future plans, including further laboratory equipment, potential techniques, and analytical methods.

Furthermore, this short presentation goes beyond basic measurements and delves into more interesting cases encountered in our NMR laboratory. I will present a few real-life case studies and examples that highlight the intricacies of working with challenging samples and problems.

NMR OF QUADRUPOLAR NUCLEI IN SOLIDS AT ULTRA-HIGH MAGNETIC FIELDS

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Quadrupolar nuclei with spin $I \geq 1$, such as ^{11}B, ^{14}N, ^{17}O, ^{33}S, ^{35}Cl, ^{63,65}Cu, ^{67}Zn and ^{71}Ga, represent over 74% of stable NMR-active nuclei and are present in a broad range of solids. Nevertheless, the solid-state NMR of these nuclei remains often challenging because of their large the density matrix and the large anisotropic quadrupolar interaction, which broadens the NMR spectra and complicates the spin dynamics. As a result, NMR spectra of quadrupolar nuclei often lack resolution and many techniques developped for spin-1/2 isotopes are not suitable for quadrupolar nuclei.

This lecture will present a brief overview of the main techniques used to improve the resolution and the sensitivity of NMR spectra of quadrupolar nuclei. In particular, multiple-quantum magic-angle spinning (MQMAS) experiments can be employed to remove the broadening of the signals by quadrupolar interaction. The sensitivity for the detection of quadrupolar nuclei can be improved by the acquisition of a train of echoes using Carr-Purcell Meiboom-Gill (CPMG) sequence as well as population transfers between the different energy levels of the quadrupolar nucleus.

More recently the sensitivity and the resolution of NMR spectra of these quadrupolar nuclei has been greatly improved using ultra-high field magnetic fields produced either by persistent hybrid superconducting magnets built from high- and low-temperature superconductors (LTS) with a static magnetic field (B_0) of 28.2 T, *i.e.*, ^{1}H Larmor frequency ($\nu_0(^1H)$) of 1.2 GHz, and DC powered hybrid magnets combining LTS and resistive wires with B_0 = 35.2 T and $\nu_0(^1H)$ = 1.5 GHz. The sensivity for the detection of quadrupolar nuclei has also been improved using indirect detection via protons [1,2] and dynamic nuclear polarization (DNP) [3]. These innovative approaches open new avenues for the detection of quadrupolar nuclei with low gyromagnetic ratio, low natural abundance or large quadrupolar coupling, such as ^{33}S, ^{63,65}Cu and ^{67}Zn, in a wide range of solids.

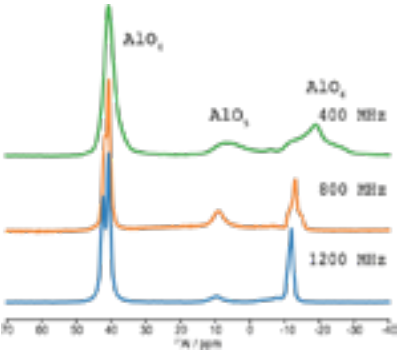

FIGURE 1. 1D ^{27}Al NMR spectra of a sample of the crystalline microporous aluminophosphate VPI-5 recorded on 400, 800 and 1200 MHz NMR spectrometers.

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^{69}Ga AND ^{71}Ga AS PROBES OF LOCAL SYMMETRY IN DIAMAGNETIC COMPLEX METALLIC PHASES

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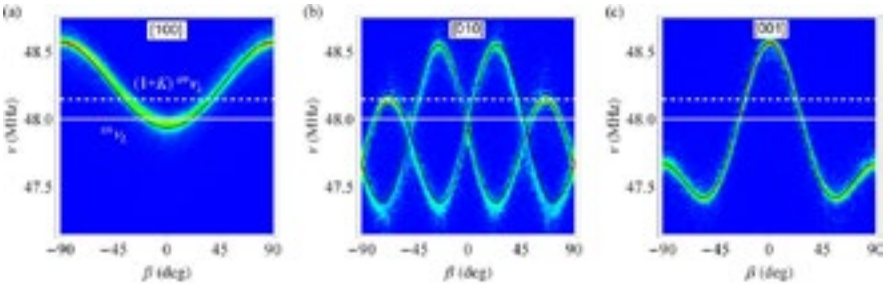
The ^{69}Ga and ^{71}Ga of the GaPd, $GaPd_2$ and Ga_3Ni_2 intermetallic catalysts NMR experiments allow to determine the electronic density of states at the Fermi level (DOS) from the Knight shift and the spin-lattice relaxation rate, which are both determined by the magnetic interaction of the nuclear spins with the spins of conduction electrons. The resolution of the Knight shift and the quadrupolar shift can be done for monocrystalline samples, by recording rotation patterns of the NMR spectra about different orthogonal crystallographic directions. Our determination of the ^{69}Ga and ^{71}Ga gallium electric-field-gradient (EFG) tensor, the Knight shift and the spin-lattice relaxation rate on monocrystalline samples were performed [1, 2]. In a case of the orthorhombic $GaPd_2$ intermetallic compound we could determine the rotation patterns of the ^{69}Ga and ^{71}Ga central lines (-1/2 ↔ +1/2 transition of a spin I = 3/2 spectrum) because of the large quadrupole frequency $\nu_Q = e^2qQ/(2h)$ of both isotopes (where $eq = V_{ZZ}$ is the largest EFG tensor eigenvalue, $Q(^{69}\text{Ga}) = 17.1 \times 10^{-30}$ m^2 and $Q(^{71}\text{Ga}) = 10.7 \times 10^{-30}$ m^2 are the nuclear electric quadrupole moments of the two isotopes and h is the Planck's constant), which made the satellite transitions unobservable. The figure below shows the rotation patterns of the ^{69}Ga NMR central lines at T = 300 K around the [010] – the (a) panel, [010] – (b) and [001] – (c) crystal axes. A small nonzero value of the quadrupolar asymmetry parameter η = 0.034 in the $GaPd_2$ and η = 0.030 in the Ga_3Ni_2 as compared to η = 0 in the cubic GaPd means that the local charge distribution around the Ga site in the $GaPd_2$ is close to be axially symmetric, whereas it is fully axially symmetric in the GaPd. More advanced structurally examples of ^{27}Al-based complex phases like a high-entropy alloy and a decagonal quasicrystal will be discussed during the lecture as well [3, 4, 5].



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LOOKING INTO POROUS MATERIALS WITH SOLID STATE NMR AND RELAXOMETRY

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Solid porous materials find widespread domestic and industrial applications, including construction and building, gas separation and storage, filtration and purification, ion exchange, energy storage, and heterogeneous catalysis. This class of materials encompasses fully inorganic or organic systems and organic/inorganic hybrid systems, with both crystalline and amorphous structure. The macroscopic behaviour of porous materials is strongly related to the structural and dynamic properties of their building units at molecular level, as well as to the interactions of the porous matrix with guest molecules.
Solid-state NMR (SSNMR) spectroscopy and NMR relaxometry represent very powerful techniques for characterizing microscopic properties over wide space and time scales and drawing structure-properties relationships in porous materials. Thanks to the possibility of exploiting many different NMR-active nuclei and a variety of nuclear observables, the application of NMR methods allows molecular and supramolecular structure and motions of specific moieties of the porous materials to be investigated. In addition, information can be acquired on the dynamics of gaseous or liquid guests hosted in the porous matrix, as well as on host-guest interactions.
In this lecture, the applicability of SSNMR spectroscopy and relaxometry experiments for looking inside porous materials will be discussed presenting several examples taken from the recent research work of the Pisa SSNMR group. In particular, the lecture will focus on structural properties and hydration of innovative cements characterized by multinuclear SSNMR [1,2] and ^{1}H relaxometry [3,4] and on structural and dynamic properties and host-guest interactions of metal organic frameworks (MOFs) [5] and polymers of intrinsic microporosity (PIMs) for CO_2 capture investigated by multinuclear SSNMR.

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FIELD-CYCLING NMR RELAXOMETRY OF PARAMAGNETIC-LABELED PROTEINS

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Field-cycling relaxometry can provide the nuclear longitudinal relaxation rates as a function of the applied magnetic field over 4 orders of magnitude. These measurements permit to have a direct access to spectral density functions, so that information on molecular structure and dynamics of macromolecules in solution can be obtained. ^{1}H relaxometry profiles are very useful for the characterization of paramagnetic molecules, especially in the field of the development of contrast agents for MRI.

The interaction of paramagnetic complexes with macromolecules present in biological systems can cause an increase of the paramagnetic relaxivity [1,2], which in turn increases the MRI contrast. The paramagnetic relaxivity is defined as the relaxation rate enhancement due to the presence of 1 mmol/dm^3 concentration of the paramagentic species. The strategy to reduce the risks associated with the administration of MRI gadolinium(III) contrast agents can pass through the use of complexes with higher efficiency, i.e. higher relaxivity, so that the injected dose can be sizably reduced. Molecular reorientation times in the nanosecond timescale are needed to achieve the highest efficiency at the fields of MRI scanners. This can be achieved by functionalizing low molecular weight gadolinium(III) complexes to bind noncovalently to macromolecules, by confining them within nanosized matrices, like nanogels, or by exploiting nanosized gadolinium(III)-based compounds.

The relaxivity profiles of Gd-labeled large protein assemblies, as the tetrameric protein asparaginase or the protein cages AaLS-13 and OP, show a remarkably high relaxivity at MRI fields, ca. five times higher than that of clinically used contrast agents. The analysis of the relaxivity profiles can shed light on the origin of the observed relaxivity enhancement, mainly ascibed to a correlation time modulating the proton-electron dipole-dipole interaction of few nanoseconds [3,4].

Hydrogel nanoparticles composed of chitosan and hyaluronan and incorporating Gd-complexes with different hydration number were also characterized through ^{1}H NMR relaxometry [5]. These nanoparticles exhibit enhanced relaxivity, over six times that of the free complexes, thanks to a restricted molecular dynamics and fast exchange of the metal-bound water molecule(s). Also the ^{1}H relaxivity profiles of Gd-HP-DO3A in crosslinked hyaluronic acid, used for monitoring the relaxation properties of contrast agents in tissues, show a modest interaction of the Gd(III) complex with the hydrogel and a slowed mobility of the water molecules [6].

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NMR RELAXATION PROCESSES IN SOLUTIONS OF SUPERPARAMAGNETIC NANOPARTICLES

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Superparamagnetic nanoparticles are considered as a novel solution for achieving high contrast in NMR imaging (MRI). The concept lies in exploiting the high magnetic moment of superparamagnetic nanoparticles that leads to very strong dipole-dipole interactions with magnetic moments of ^{1}H nuclei of surrounding water (solvent) molecules.

The mechanism of ^{1}H (generally, spin ½) spin-lattice relaxation in solutions of superparamagnetic nanoparticles includes numerous factors that affect the relaxation rates. As already stated, the relaxation pathway is constituted by dipole-dipole interactions between the magnetic moment of the nanoparticle and the nucleus. The interactions fluctuate in time as a result of translation diffusion of the solvent molecules. One should, however, also consider the possibility that the solvent molecules (typically water) form a layer (hydration shell) around the nanoparticle staying attached to it for a time comparable (or longer) to the rotational correlation time of the nanoparticles. In such a case there are also exchange processes between the bound and the "free" fractions of the solvent molecules. As far as the magnetic moment of the nanoparticle is concerned, it has two components: a mean (average) part which is parallel (longitudinal) to the external magnetic field and does not fluctuate in time, and a component that fluctuates around this average value. Both components depend on the applied magnetic field. The mean component increases, according to the Langevin function, from zero towards a saturation value at high fields [1,2]. As the mean component grows the fluctuating part of the longitudinal magnetic component gets lower, i.e. it reaches maximum at zero field and decreases towards zero with increasing the magnetic field. In consequence, the measured ^{1}H relaxation rate is a sum of two relaxation contributions. The first contribution is associated with the mean component of the magnetic moment and is referred to as Curie relaxation [3] and dominates at higher magnetic fields. The second one, associated with the fluctuating component of the nanoparticle magnetization is responsible for the low field relaxation [3,4]. The relaxation scenario will be explained and illustrated by experimental examples.

Acknowledgements

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ANALYSIS OF STRONGLY DIPOLAR COUPLED SOLIDS BY TIME-DOMAIN NMR

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Transverse relaxation in rigid-state materials below glass-transition temperature happens to be exponential in very rare cases, mostly showing up complex shape that many authors try to fit within a mixture of Gaussian, Abrahamian, Pake and Lorentzian lines, though for the majority of samples even such variative models do not work perfectly.

The approach to characterize structures and dynamics of solids that involves moments of spectral lines is becoming more adequate and promising because the moments can be directly calculated using integration of FFT spectrum and not rely on ambiguous muti-component approximation of the TD-NMR Free Induction Decay (FID).

On the other hand, the huge community of scientists in applied areas where TD-NMR is considered as only one of the measuring tools are educated in the way of simple relaxation times approach and the use of second moments M_2 expressed in squared Tesla occurs far less convenient and demonstrative than the T_2 in microseconds.

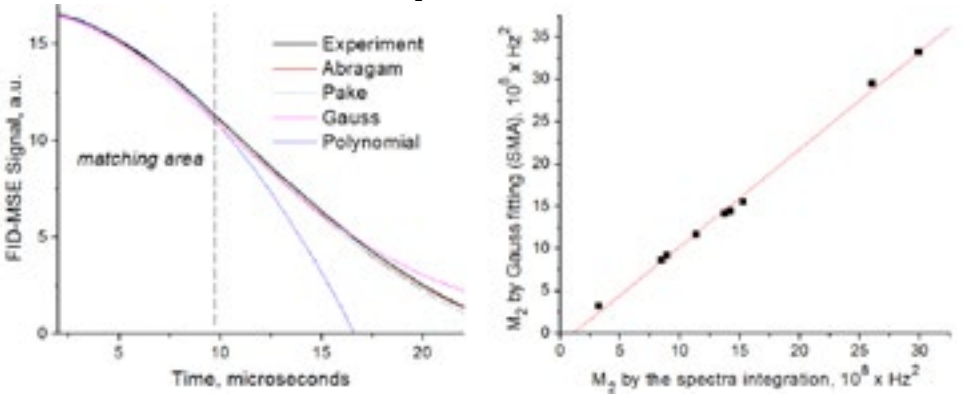

FIGURE 1. The idea of the Second Moment Approximation (SMA) applied to processing of Starch samples

The contribution pretends to bring up the way to recalculate second moments corresponding to various line shapes into timely parameter T_2^{**} with minimal distortion of information about structure of solid-state materials.

Besides the FID analysis we are discussing the use of the resdual dipolar constant, acquired via Double Quantum Build-up Curve experiment for the additional insights at solids structure.

Acknowledgements

This work was partially funded from the European Union's Horizon 2020 Research and Innovation programme – MSCA-RISE under grant agreement No 101008228.

CHEMICAL EXCHANGE RATE STUDIED BY NMR CPMG METHOD

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Various nuclear magnetic resonance method, which manipulate the spin dynamic of atomic nuclei in order to convey information about their environment i.e. the structure and dynamics in matter, are used in chemistry, physics, material science, biology, and medicine. Particularly useful NMR method for the study of the molecular dynamics is the Carr-Purcell-Meiboom-Gill sequence (CPMG)[1] with a train of π-radiofrequency pulses, which was originally introduced to provide reliable spin measurements relaxation, but later on it turned out that it in conbination with an inhomogeneous magnetic field provides the direct insight into the spectrum of the molecular velocity auto-correlation[3,4], which is the key dynamic quantity of molecular translation motion. It is less known that by using CPMG sequences we can also obtain detailed information about the fluctuation of molecular electronic orbitals, which are the result of chemical processes such as also chemical exchange[2].

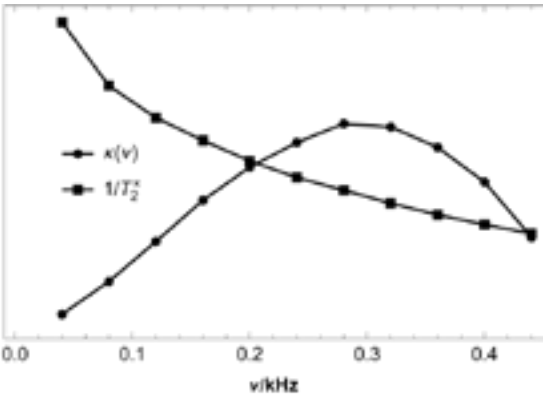

FIGURE 1. Spectrum of chemical exchange rate κ(ν) in aqueous sugar solutions obtained from the effective relaxation decay $1/T_2^*$ measured by the CPMG method

We will show, that with the CPMG measurements of liquids[5] in which there is a processes, such as chemical exchange, the decline of the peak of the spin echo is directly proportional to the value of the spectrum of the exchange rate at the value of the spin phase modulation frequency, which is determined by the frequency of π pulses. The method not provide just chemical exchange spectrum between only two molecular conformations, but it could result from several exchanges effecting the molecular conformation.

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ASSESSING THE MOLECULAR DYNAMICS IN PARAMAGNETIC LIQUID SYSTEMS

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NMR relaxometry is a widely used experimental technique that enables the study of dynamical processess ranging from fast internal molecular rotations to slower aggregate motions. This technique is particularly sensitive to the presence of paramagnetic species, such as iron, gadolinium, manganese and cobalt, just to name a few. In Figure 1 it is possible to observe the highlighted paramagnetic relaxation enhancement induced by the presence of around 1mM of gadolinium in a phosphonium-based ionic liquid system.

Paramagnetic relaxation can be induced by a temporary binding to the paramagnetic center (inner-sphere, IS) or by translational diffusion in its vicinity (outer-sphere, OS). The deconvolution of these two processes is non-trivial and generally requires one to perform multiple experiments, such as analyzing systems with different solvent proportions[1], varying the paramagnetic species concentration or varying the temperature at which the experiments are performed.

In the present work we achieve a consistent analysis of ionic liquid- and PEG-based paramangetic systems, relying on the simultaneous analysis of data obtained for different temperatures and concentration of magnetic particles. Moreover, we show that the spin-spin relaxation time, T_2, might be a more efficient alternative to discriminate between IS and OS paramagnetic relaxation. Finally, the subtly significant influence of metal impurities is also evidenced in the PEG-based systems.

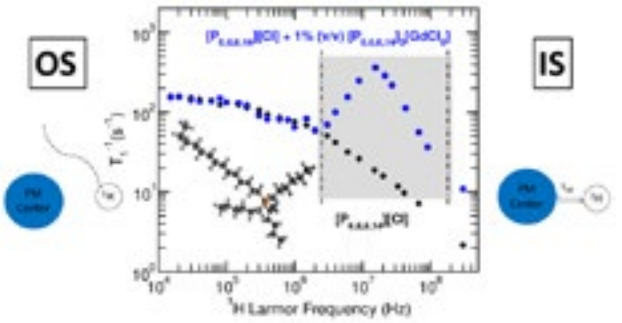

FIGURE 1. Gadolinium-induced paramagnetic relaxation enhancement in ionic liquid systems

Acknowledgements
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IONIC-LIQUID BASED HYBRID GEL POLYMER ELECTROLYTES AS SEEN BY NMR

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Gel polymer electrolytes (GPEs) combine the advantages of liquid and solid electrolytes. They exhibit both high ion conductivity, similar to liquids, and the mechanical stability and high safety of solids, which makes them ideal candidates energy storage applications. Since GPCs are multicomponent systems, they exhibit quite complex behavior that can be studied at the molecular level by a variety of NMR methods using different nuclei. In this lecture, an introduction to NMR investigations of GPEs will be provided, focusing on our studies of GPEs based on the random copolymer poly(vinylidene fluoride co-hexafluoropopylene) (PVdF-HFP) swollen with electrolyte solutions (ES) of salt in an ionic liquid (IL) and containing inorganic fillers, making them hybrid organic/inorganic materials. The fillers are added to optimize conductivity [1] or electromagnetic shielding [2]. Figure 1 shows examples of diffusion coefficients and line shapes, reflecting molecular mobilities.

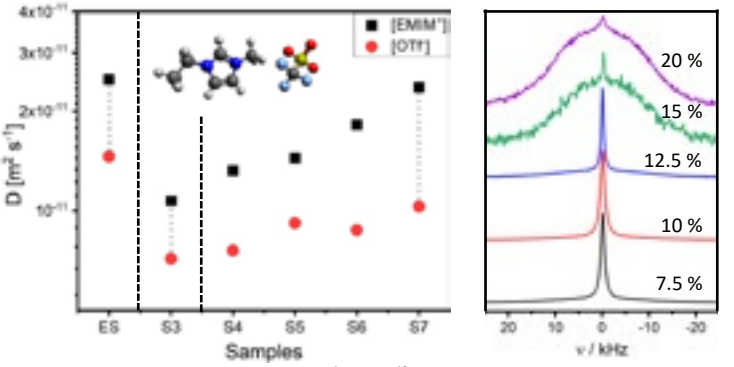

FIGURE 1. (a) Diffusion constants obtained from ^{1}H and ^{19}F measurements of the IL [EMIM][OTf] in the system PVDF-HFP/SiO_2/ES, where ES is the electrolyte solution of LiOTf in the IL, and S3 to S7 are GPE samples (S3: 65 % ES, no SiO2; S4 to S7: 50, 55, 60, and 65 % ES, respectively, with SiO_2) [1]. (b) Static ^{7}Li spectra of the system PVDF-HFP/$BaTiO_3$/ES, where ES is $LiBF_4$ in [EMIM][BF_4], containing different mass fractions of $BaTiO_3$ [2].

Acknowledgements

The work by the (former) students and postdocs in my group, Dr. W. Keil, R. Duo, M. Beerbaum, S. Lansab, M. Siebrecht, Y. Zhao, and Dr. A. Ordikhani-Seyedlar, is gratefully acknowledged. Thanks go to Prof. A. Chandra (University of Delhi) and her former Ph.D. students, Dr. M. Kumar Vyas and Dr. S. Khurana, who provided most of the materials, to Dr. M. Dvoyashkin (University of Leipzig) for diffusion measurements, to Dr. R. Graf (Max-Planck-Institute for Polymer Research) for stimulating discussions, and to the Alexander von Humboldt Foundation for funding the Delhi-Paderborn research linkage.

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CHALLENGES OF NON-EXPONENTIAL NMR RELAXATION PROCESS

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NMR relaxation processes give insight into molecular motion. In NMR experiments one monitors time evolution of magnetization (^{1}H, ^{19}F, *etc*.) The non-exponentiality can result from two sources: dynamical heterogeneity of the system or/and the quantum-mechanical framework of the relaxation process. It is well-known that even a very simple spin system, like two non-equivalent ½ spins (such as ^{1}H, ^{19}F or ^{1}H, ^{13}C) exhibit, in principle, bi-exponential relaxation (and, hence, bi-exponential evolution of magnetization) as a result of joint spin transitions (cross-relaxation) caused by mutual dipole-dipole interactions [1-3]. However, to experimentally observe a bi-exponential magnetization evolution, two conditions must be met: the first one is a significant difference (let us say at least by an order of magnitude) between the relaxation rates, while the second one is comparable contributions of both relaxation components in terms of their amplitudes. The effect depends on several factors, such as: the magnetic field, the difference in the gyromagnetic factors of the participating spins, the amplitude of the dipole-dipole coupling, the time scale and the mechanism of the molecular motion– as shown in Fig.1. The non-exponentiality of relaxation processes will be discussed in detail and illustrated by experimental examples.

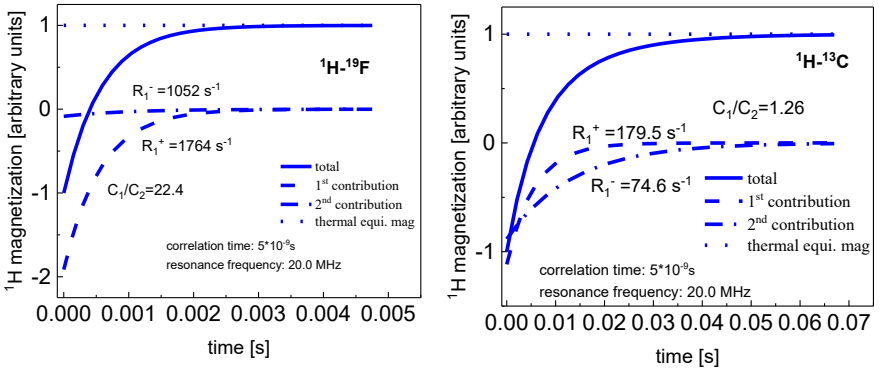

FIGURE 1. . Examples of bi-exponential magnetization evolution.

Acknowledgement

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ON-LINE LABORATORY TRAININGS

AMPERE **NMR** School

JASON workshop: practical aspects

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JASON is a new vendor agnostic analytical software with special emphasis on the automatic processing, analysis and reporting of NMR data [1]. In particular, JASON features a large work space, "Canvas," where 1D NMR, 2D NMR, and chemical structures are interlinked to support structural analysis. In this workshop we will present an overview of the software and how it can be used for structure verification and generation of reports. To fully benefit from this workshop it is recommended to bring a laptop with the software already installed.

FIGURE 1. Overview of JASON capabilities

References

[1] JASON https://jeoljason.com/

MAGIC ANGLE

Jacek Jenczyk

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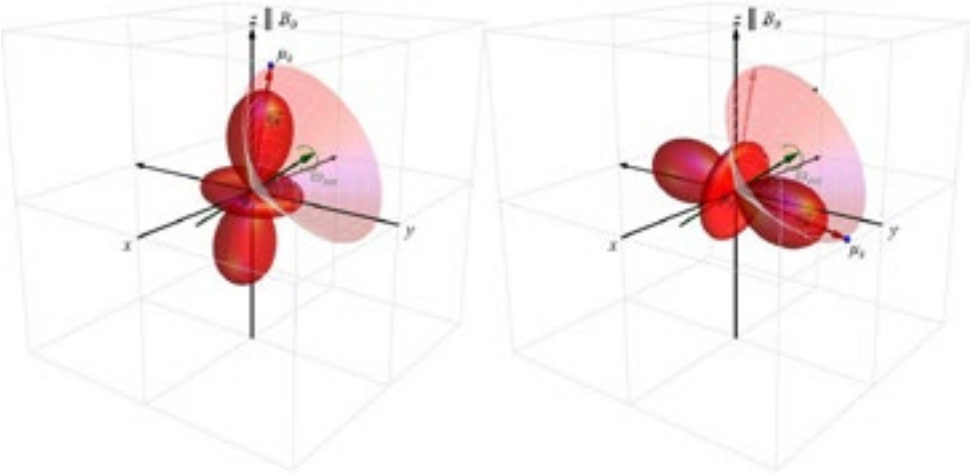
There are number of exceptional examples indicating the unique position of tetrahedral symmetry in the vast landscape of different spatial organization pathways which can be sampled by matter. Very often the tetrahedral angle remains an intrinsic feature of the system or emerges spontaneously due to interactions. Quite remarkably, the same angle reappears in fundamental spin interactions i.e. the dipolar coupling between a pair of polarized spins (magnetic moments) vanishes when the inter-spin vector is tilted one half of the tetrahedral angle with respect to the polarization field direction. This specific condition is known as the magic angle in the NMR community and the effect is directly related to the inherent symmetry of a magnetic field generated by an isolated magnetic moment. Developed conceptually in the 1950s [1, 2] Magic Angle Spinning (MAS) has become a routine technique in solid state NMR spectroscopy. This unique concept, together with the exceptional progress made in the field of NMR probe engineering, has made possible the acquisition of high resolution spectra for a rigid phase samples. General idea regarding spin interactions under MAS conditions can be demanding to comprehend due to an experimentally introduced time evolution of the spin system and its anisotropic character of interactions. In order to thoroughly understand the concept of a MAS experiment it is vital to first describe the structure of coupling tensors and their subsequent orientational evolution during NMR experiments.



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FFC-NMR TUTORIAL

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One of the fundamental topics of molecular science is to learn dynamical properties of biomolecules. Nuclear Magnetic Resonance (NMR) methods, especially FFC-NMR relaxation studies, give a deep insight into the dynamics of biomolecular systems i.e. tumbling-like motion, translation diffusion and internal dynamics. In FFC-NMR relaxation experiments at low frequencies (magnetic fields) one probes slow molecular motions in the scale of microseconds, while with increasing the frequency progressively faster dynamics up to nanoseconds are detected. The FFC-NMR relaxation studies stems as an extremely valuable source of information about the dynamics of biomolecules, nevertheless, they pose several challenges from the experimental as well as theoretical point of view.

In this context, the FFC-NMR tutorial will be divided into three parts: First, some general introduction to the Fast Field Cycling technique will be provided, along with the theory of basic pulse sequences and essential knowledge for setting of proper parameters to start an experiment. Next, step by step a simple FFC experiment will be performed remotely using one of the FFC-NMR Relaxometers (Stelar s.r.l.), located at the University of Warmia and Mazury in Olsztyn laboratory, including practical advice for beginner users. In other words, the tutorial Participants will simply learn "How to get started with the FFC-NMR measurements" - i.e. sample preparation, sample tuning, choosing the right sequence (depending on the type of sample), setting proper parameters, acquiring and evaluating FFC data. In the last part of the tutorial, some theoretical aspects of data analysis will be presented.

MRI: BASIC PRINCIPLES AND APPLICATION

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The tutorial aims to introduce the basics of the NMR phenomena and procedures in the MRI lab. Classes will be divided into two parts. In the first part, some basic information about NMR and MRI will be provided, and in the next step will be shown the preparation of an MRI research scanner, ie. tuning coil, and magnetic field shimming. Moreover, 1D NMR experiments will be carried out.

The second part will show the basic experiments using Fourier imaging methods with the introduction of the spin echo imaging technique. The building of k-space during MRI experiments will be shown, to highlight the significance of Fourier transform in Magnetic Resonance Imaging. Finally, the images for the object with a well-defined structure will be obtained with spin echo and gradient echo techniques.

The tutorial will be carried out with the use of ICT technology and remote access to an MRI scanner at NanoBioMedical Centre in Poznan.

NMR DIFFUSION

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The word ***diffusion*** derives from the Latin word *diffundere*, which means "*to spread out*".

In the most general case, diffusion is a phenomenon that refers to the net movement of an object driven by a gradient of some magnitude factor. The process has a stochastic nature, and its concept plays an important role in many areas of physics, chemistry, biology, sociology, economics, and finance, constituting a broad field for research. In natural sciences, diffusion is not limited to a given state of matter but can occur in solids, liquids, and gases. It conditions the life processes by determining the transport through membranes, cells eventually, the whole body. In chemical processes, it is often the central rule driving many reactions. In physics, it defines many transport processes for atoms, ions, or molecules. A distinguishing feature of diffusion is that it depends on particle random walk and results in mixing or mass transport without requiring directed bulk motion. The first description of the diffusion phenomena was given by Adolf Fick in 1855. Fick's laws can be used to solve for the diffusion coefficient, ***D***. A diffusion process that obeys Fick's laws is called *normal* or *Fickian diffusion*. On the other hand, it is called anomalous diffusion or non-Fickian diffusion if the process does not follow these laws. This tutorial aims to give an overview of the wide range of applications of diffusion NMR and principles of NMR diffusometry methods that allow insight, for example, for accurate molecular size determination, in nanomedicine drug delivery, or separation of complex mixtures. The tutorial will be carried out with the assistance of dr hab Kosma Szutkowski from the NanoBioMedical Centre in Poznan and remote access to an NMR spectrometer at the NanoBioMedical Centre in Poznan.

PARTICIPANTS

POSTERS

AMPERE **NMR** School

Insights into Gelation Mechanisms and Gel Properties of LMWG/PC: A Comparative Study using DSC/TGA, NMR, and Confocal Fluorescence Microscopy

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ABSTRACT

In this study, we present the LMWG, also referred to as a candidate for the gel-electrolytes functional material that exhibits promising potential for utilization in various electrochemical applications. Acquiring an understanding of the distinct properties exhibited by LMWG at the Sol-gel phase is crucial for analyzing and predicting the features they offer. The current investigation presents a comparative analysis including thermal analysis (DSC/TGA), intermolecular interactions (NMR spectroscopy), and microstructure elucidation (Confocal Fluorescence Microscopy) of a physical gel based on a low molecular weight gelator, specifically cyclo (L- β -2-ethylhexylasparaginyl-L-phenylalanine)2-ethylhexyl 2-((2S,5S)-5-benzyl-3, 6-dioxopiperazin-2-yl) acetate and solution of propylene carbonate. A physical gel's molecular structure, dynamics, and interactions were analyzed through NMR. The DSC/TGA technique provided us with the thermal characteristics of the gel. Optical polarization microscopy was employed to examine the internal microstructure of the gel. The studies were performed on an LMWG-based Gel using a propylene carbonate solvent.

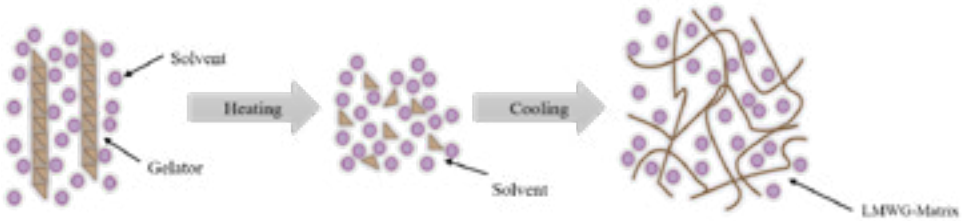

FIGURE 1. The preparation route of LMWG-based gel through the physical method

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Magnetic resonance imaging of hydrocarbon gas at various pressures

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Thermal reactions using heated gases are the driving force behind industrial production processes. However, a lack of understanding regarding the distribution of gas flow within reactors hampers the optimization of these chemical processes. We are proposing using magnetic resonance imaging (MRI) to vizualize the gas flows inside the reaction chambers. In the current work, we study thermally polarized hydrocarbon gas, ethane, under slightly elevated pressures (1.5 – 6 bar) and evaluate their relaxation parameters and assess their utility for rapid imaging.

MRI experiments were done on a 7 T pre-clinical MRI (BioSpec, Bruker), using a ^{1}H transmit-receive quadrature volume resonator (86 mm diameter, Bruker). We used a 75 mL borosilicate flask (Synthware) filled with ethane (C_2H_6, 99.95% purity, Linde plc) as a phantom of the reactor. The longitudinal relaxation time constant, T_1, and the transverse relaxation time, T_2, of ethane were measured at various pressures in the range of 1.5 – 6 bar using an inversion recovery (IR) and a Carr-Purcell-Meiboom-Gill (CPMG) pulses sequences with a very short constant echo spacing of 2 τ = 600 µs (Figure 1 (a, insert)), respectively. Based on the measured relaxation times (Figure 1 (a)), we adjusted the MRI sequence parameters and acquired images of ethane using a fast low angle shot (FLASH), and a rapid acquisition with relaxation enhancement (RARE) (Figure 1 (c)) sequences. The SNR of sequences was maximised by taking into account dependence of relaxation on pressure. Each image was acquired within 100 s only with an signal to noise ratio (SNR) between 140 and 190 for FLASH and 170 – 280 for RARE pulse sequence. The experimental data were compared to the values of the gas-phase SNR expected for the corresponding pressures (Figure 1 (d)). Thus, we show the feasibility of fast imaging of gases within the reaction chamber.

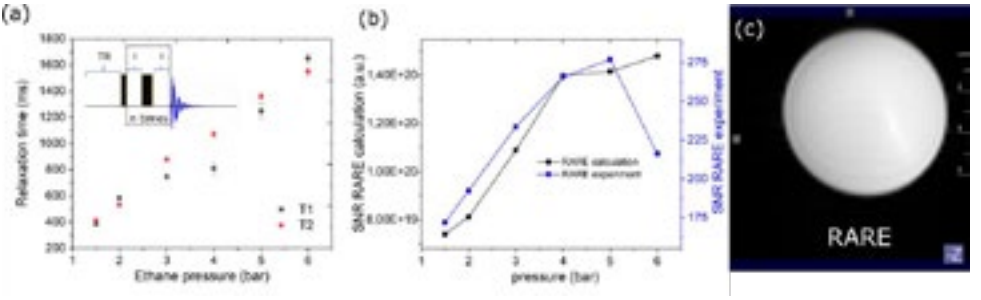

FIGURE 1. (a) Summary of of ethane longuitudinal and transverse relaxation times measured in IR and CPMG with τ = 0.3 ms (insert) experiments at different pressures. (b) Comparison of the calculated (black dots) and experimental (blue dots) SNR values for RARE imaging. (c) Axial RARE image of the flask filled with 4 bar ethane (slice thickness 80 mm, resolution 0.906 mm x 0.906 mm, TE = 15.36, RARE factor = 2, TR = 1.6 s, 2 averages, bandwidth = 5 kHz).

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Synthesis of novel tracers for deuterium magnetic resonance imaging

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Deuterium magnetic resonance imaging (DMI) is a non-invasive technique capable of generating high-resolution metabolic maps. This study explores the potential of deuterium-labeled DMI tracers for in vivo diagnostics and treatment monitoring. We present a novel deuterium based pH sensor for MRI, deuterium-labeled zymonic acid (ZA)-d4.

Protonated ZA was synthesized from sodium pyruvate (Pyr) and its pH sensitivity was evaluated. Pyr (0.1-0.2 M) was dissolved in water and maintained at a pH of 12 for two days. Acidification with HCl (pH ~1) was followed by water removal using a freeze dryer. The resulting crude ZA was diluted in acetone, filtered to eliminate NaCl, and purified using flash column chromatography with a Biotage SNAP Ultra C18 60g column and a 2-10% acetonitrile linear gradient. The purified ZA was freeze-dried, yielding a dark yellow viscous mass with an experimental yield of 50.8%.

pH-dependent changes in the chemical shifts of CH and CH_3 resonances of ZA were investigated using ^{1}H NMR analysis at varying pH values (2-3, 6-8). Two pKa values, 1.5±0.7 and 7.06±0.02, were confirmed. This chemical shift variation is planned to be utilized for pH mapping with DMI. Future steps involve synthesizing deuterated ZA and acquiring corresponding DMI pH maps.

In conclusion, deuterium-labeled zymonic acid (ZA)-d4 shows promise as a pH sensor for MRI, with its pH-dependent chemical shifts opening a new avenues for non-invasive in vivo diagnostics and treatment monitoring.

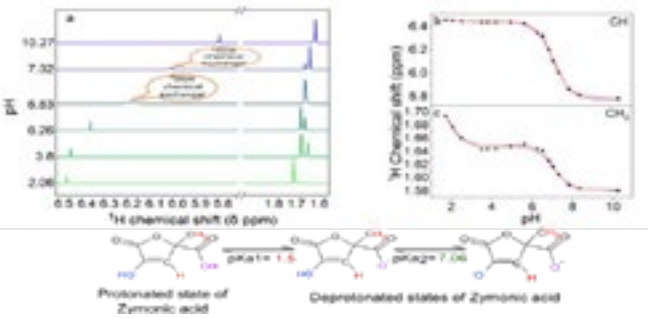

FIGURE 1: ^{1}H NMR spectra of ZA (a) and chemical shifts (b,c) as a function of pH. This experiment was conducted in 9.4 T at 290 K using 1:10 of H_2O:D_2O 49.64 mM ZA solution. Chemical shift variation of CH and CH_3 protons revealed to pH sensitive areas near pKa values of ~1.5 and 7.06.

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INSIGHTS TO NMR RELAXATION AND SUSCEPTIBILITY REPRESENTATION

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Nuclear Magnetic Resonance Relaxation is a powerful experimental tool to study a spin particle's system interacting with its surroundings. Behind the experimental technique's success, there is a theoretical background well established, such as the Redfield master equation, which is one of the main formal backgrounds used to decode information from an environment monitoring the spin particle system. Consequently, applying the Redfield master equation, the magnetization dynamics is non-exponential for some heteronuclear cases. The highlighted dynamics are less frequent in experimental implementations, often mixed or overlapped with the mono-exponential relaxation evolution of the magnetization. Therefore, it is interesting to establish some procedures to identify the non-exponential signature from the magnetization evolutions. In this study, we considered that the magnetization time evolutions are well known, such that the relaxation rates can be computed and established, showing the characteristic dependence of the spectral density functions, which allows us to figure out the most suitable kinds of molecular motion. Together with the relaxation rates, there are the susceptibility functions. It is a physical definition that will help us find some insights about the non-exponential relaxation and the regimes in which the non-exponential signature could, or not, be more evident.

In this discussion, we explored the relaxation rates of a heteronuclear spin system generated from a theoretical development found in Ref. [1,2]. Next, we applied the definition of susceptibility function $\chi = \omega R_1$, where ω is the angular frequency and R_1 is the relaxation rate. The susceptibility function was analyzed at different angular frequency regimes and using the spectral density function of the force-free hard-sphere model [1]. Those results were compared with susceptibility functions considering the mono-exponential relaxation, and the differences were highlighted.

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POSTER N^{O} 5

SYNTHESIS AND CHARACTERIZATION OF Gd-BASED DENDRITIC POLYMERS AS A POTENTIAL CONTRAST AGENT

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As new multifunctional materials, dendritic hyperbranched polymers have drawn much attention because of their unique fluorescence properties, perfect mechanical toughness, and good chemical stability. These features allow to use it in the fields of photoluminescence, electroluminescence, optics, lasers, and solar–energy conversion systems [1,2]. Due to high water solubility and low toxicity, dendrimers have emerged as one of the most promising nanocarrier systems for biomedical applications, especially for drug delivery or medical imaging. Among rare earth elements gadolinium Gd due to the 7 unpaired electrons in its 4f subshell is a well-known MRI/NIR dual-modality imaging agent due to their high r_1 relaxation and paramagnetism [3]. At the same time, Gd is a well-known green-emission phosphor ($\lambda_{lum.}$ at 545 nm) and can be used as a biomarker for the detection of cancer cells.

The present study focused on the synthesis of Gd-based dendritic hyperbranched polymers, *in vitro* biocompatibility evaluation for potential use in magnetic resonance imaging contrast agents.

The binding of Gd^{3+} ions to the dendrimer complex was confirmed by photoluminescence, FT-IR, and NMR spectroscopies. The complexes show characteristic photoluminescence peaks in the visible green region - emission characteristic of gadolinium. FT-IR spectroscopies confirmed that the carboxyl group of the dendrimer is coordinated with the Gd ions. Results from NMR measurements showed, that Gd ions may reside within terminal, dendritic regions and thus lead to vanishing of NMR signals characterized by higher chemical shift values. Cytotoxicity studies showed that Gd-based dendrimers do not have a significant negative effect on human cardiomyocyte cell line up to the highest concentration tested - 100 μg/ml.

Acknowledgment
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Fast pKa determination for lead optimisation Application of Chemical Shift Imaging and Chemical Gradients into Analysis of APIs

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^{1}H Chemical Shift Imaging [1] with a chemical gradient (CSI-CG) provides an efficient, one-shot method for the determination of pKa, using little analyte [2]. The technique can be readily used in DMSO-d6 [3] and has been used to analyse basicity patterns in a homologous series of bridged anilines. Density-Functional Theory (DFT) geometry optimisation and charge distribution calculations have been used to rationalise the results [4,5].

We have developed mixed solvent water-DMSO pH indicator ladders. That allows the determination of pKa in any water-DMSO ratio in a single-shot experiment and the reduction in the number of experiments required to extrapolate mixed-solvent pKa determinations to an aqueous value. As a result, we can successfully extrapolate to an aqueous pKa regardless of solvation effects on the analyte with a precision greater than that offered by computational methods.

Several pulse sequences for water suppression have been evaluated, and automation routines developed to allow the use of sample changers for high throughput of data acquisition in an industrial setting [3]. Water signals are suppressed using excitation sculpting, while pre-saturation can be used to suppress a second solvent peak if required, Figure 1.

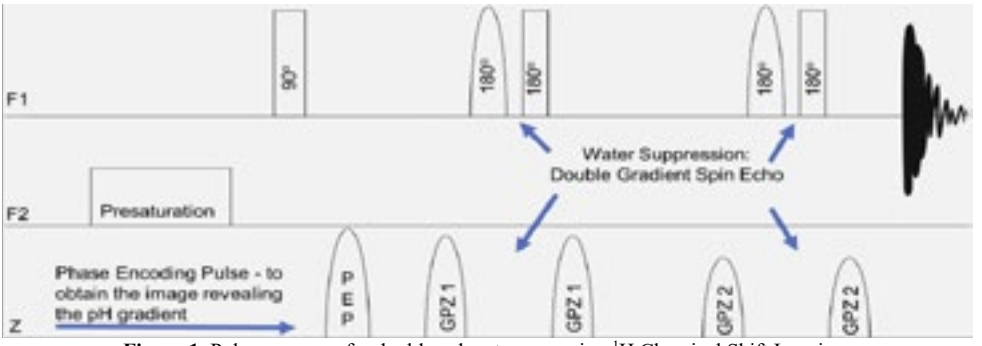

Figure 1. Pulse sequence for double solvent suppression ^{1}H Chemical Shift Imaging.

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POSTER N^{O} 7

UNDERSTANDING WATER AND OIL HOLDING PROPERTIES OF COCOA FIBER-GUM MIXTURES WITH NMR RELAXOMETRY

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Cocoa bean husk is a byproduct of chocolate industry and discarded as waste or animal feed. The husk has a high fiber content with considerable water and oil holding properties, but the cocoa aroma and flavor limits its use in ranging food applicants. Once the aroma and flavor of the husk is removed through a solvent extraction process, the aroma free mass is called cocoa fiber which is a potential stabilizer. In addition, its physical properties can be improved by incorporation of commercially available gums such as xanthan and gum arabic. In this study, water holding properties of cocoa fiber mixed with varying arabic gum (5, 10, 20%) and xanthan gum (5, 10, 20%) were measured. In order to understand the interactions of the hydrocolloid mixtures at a molecular level, T_2 (*spin-spin relaxation time*) times of the water and hydrocolloid mixes were also measured. Relaxation spectra were also obtained for the wet mixes. Addition of arabic gum did not improve the water holding properties of cocoa fiber significantly but increased T_2 of the samples. A strong correlation between water holding capacity and T_2 was obtained (R^2>0.81). Differences were also detected in the effect of gum type and oil binding capacities.

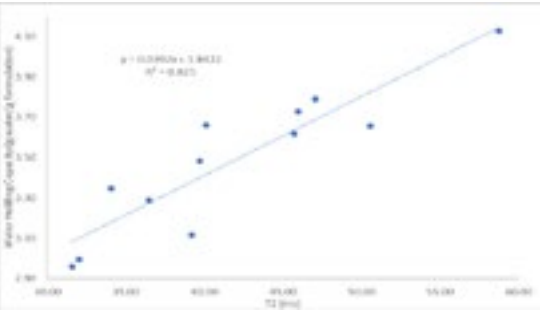

FIGURE 1. Water Holding Capacity and T_2

THREE MUSKETEERS OF MRI CONTRASTING: INFLUENCE OF METAL ADDITION ON PHOTOTHERMAL AND RELAXIVITY PROPERTIES OF POROUS POLYDOPAMINE NANOPARTICLES

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Polydopamine (PDA) nanoparticles have been extensively studied for their photothermal and therapeutic properties, making them promising candidates for cancer treatment [1]. During the synthesis of PDA nanoparticles, metal ions can be incorporated into the polymer matrix through coordination bonds with the functional groups of dopamine. The addition of metal ions can influence the physicochemical properties of PDA nanoparticles, including photothermal and relaxivity properties [2]. However, the influence of different metal dopants on the photothermal and relaxivity properties of porous PDA nanoparticles has yet to be fully explored. In this study, we aim to assess the influence of different metal dopants on the photothermal and relaxivity properties of PDA nanoparticles.

We synthesized porous PDA nanoparticles using a simple one-pot method. The nanoparticles were then doped with different metal ions: iron, manganese, and gadolinium. The physicochemical properties of the nanoparticles were characterized using various techniques, including HR-TEM with EDX, DLS, ICP-OES, and SQUID. We also assessed the photothermal properties of the nanoparticles using a laser irradiation system and evaluated the relaxivity properties of the nanoparticles using NMR spectroscopy. The photothermal and relaxivity properties of the nanoparticles were found to be dependent on the type of metal dopants. Our findings provide insights into the potential of metal-doped PDA nanoparticles for cancer theranostics and combined therapy. The porosity of nanoparticles enables loading additional cargo such as small molecule drugs, whereas the metal doping-induced paramagnetism of the nanoformulations makes them suitable for use as MRI contrasting agents.

Acknowledgments
The research was financed by The National Science Centre (NCN), Poland, under project number UMO-2018/31/D/ST8/02434.

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SPEEDING UP RESTRICTED DIFFUSION NMR MEASUREMENTS FOR POROUS STRUCTURE DETERMINATION OF BIOCOMPATIBLE GELS

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Restricted diffusion measurements with Nuclear Magnetic Resonance spectroscopy provide us detailed information about porous structures. Restricted diffusion measurements utilize the behavior of molecules with movement limited by porous structures since the diffusion coefficient is lower than expected for free movement. The disadvantage of this measurement is it's duration, which can be shortened significantly by applying fast Time-Resolved methodology[1]. In our research, we study samples of Gelatin Methacryloyl hydrogels dissolved in water. The sample is used as a backbone structure for a 3D-printed bionic pancreas. In our work, we show the relationship between the concentration of Gelatin Methacryloyl, its degree of substitution, and its structure.

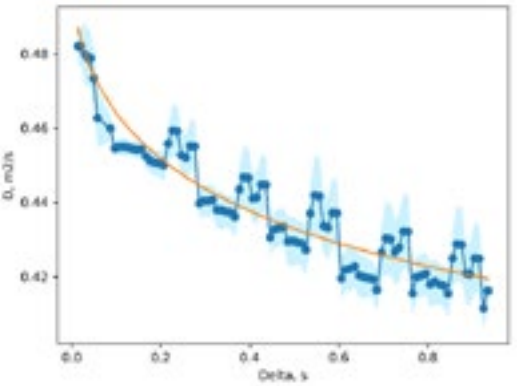

FIGURE 1. The exemplary plot of restricted diffusion of Gelatin Methacryloyl sample. Diffusion coefficient values are multiplied by the factor of 10^{-9}. Light blue color is error of diffusion calculation.

Acknowledgements
The authors would like to thank the National Science Centre, Poland, for its support in the form of an OPUS Grant (2021/41/B/ST4/01286).

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GMO@DTPA-BSA-GD NANOPARTICLES AS NOVEL POTENTIAL MRI CONTRAST AGENTS

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Current challenges in the design of new contrast agents is a search for new Gd-based paramagnetic CAs or their substitutes, whose low concentration (micromolar or even submicromolar) would make it possible to achieve the desired contrast with a low risk of systemic toxicity [1]. Nanoparticle-based CAs represent promising new platforms that next to the biocompatibility can provide high resolution and sensitivity MR imaging. Another alternative nanocarrier-approach is lipid-based nanoparticles modified with Gd-chelating lipids, where the Gd-chelating lipids are a part of the formed lipid bilayer next to the GMO lipid matrix. Among lipid nanoparticles used in bioimaging, lipid-based lyotropic liquid crystal nanoparticles (LLCNPs) stand out, particularly cubosomes and hexosomes, next to solid lipid nanoparticles (SLNPs) or other types [2]. These structures exhibiting long-range internal ordering are promising nanocarriers of CAs. In particular, these self-organized structures appear to offer a large loading of coordinated Gd(III) ions, and their extensive water channels may enable efficient water exchange between the inner and outer spheres surrounding the Gd(III) ions, resulting in better relaxivity.

Undertaken studies confirmed that proposed new lipid-based NPs show great potential as new CAs for MR imaging, particularly obtaining T_1-weighted images. At a magnetic field induction of 0.47 T, GMO@DTPA-BSA-Gd dispersed phases showed increased relaxivity r_1 compared to commercial contrast agents: Magnevist, Gadovist and Dotarem [3]. These features of LLCNPs, therefore, allow to conclude that proposed Gd-chelating lipid-enriched GMO-lipid nanoparticles may be the promising new contrast agents in MR imaging, thereby the type of nanocarriers for further development of multifunctional systems combining diagnostics and therapy in a single system.

Acknowledgments
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CHARACTERIZATION OF POLYMER-CERAMIC COMPOSITE ELECTROLYTES USING MULTIDIMENSIONAL SOLID-STATE NMR

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Electrification of vehicles, which is an effective approach to mitigate climate change requires the development of safer batteries with solid-state electrolytes. A composite polymer electrolyte (CPE) consisting of Li+ conductor inorganic particles dispersed in a polymer matrix is a promising concept that combines the high ionic conductivity and electrochemical stability of ceramics with the flexibility and processability of the polymers.[1] Although CPEs have improved the performance and stability of the electrolytes, their ionic conductivity is insufficient for high-rate applications. One possible approach to improve the ionic conductivity is the heat treatment of the ceramic particles to modify the ceramic surface and form a better interphase with the polymer.[2]

In this work, solid-state NMR spectroscopy was used to closely follow the chemical and phase evolutions of LLZO garnets during the heat treatment process and its impact on the local Li^+ dynamics. The presence and degradation of LiOH and Li_2CO_3 as secondary phases on the LLZO surface were monitored by relaxometry and 7Li-1H heteronuclear correlations. Thereafter, 2D ^{7}Li-^{7}Li and ^{6}Li-^{6}Li EXSY NMR experiments were employed to analyze the polymer-ceramic interface and detect the Li-ion exchange, as well as to quantify the exchange rates.[3,4] Using pseudo-3D experiments, LiOH was identified as an intermediate phase for the interfacial Li exchange. Finally, using $^7\text{Li} \rightarrow {}^6\text{Li}$ trace exchange experiments, the Li-ion pathway through the electrolyte was determined.

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INVESTIGATING SYNERGISTIC EFFECTS BETWEEN CELLULOSE AND LIGNIN FOR ADVANCED FOREST CARBON FIBERS: MASS TRANSPORT CHARACTERIZATION WITH MAGNETIC RESONANCE METHODS

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Carbon fibers (CFs) have a high specific strength and stiffness, which makes them very efficient reinforcements in composite materials for light-weight applications. The production of CFs requires a polymeric precursor that later can be carbonized. Forest-based products: lignin and cellulose are an attractive alternative to the fossil-based polymer commonly used. To obtain fibers, these materials need first to be dissolved in e.g. ionic liquids before being coagulated in a non-solvent e.g. water. It has been shown that wet spinning of lignin-cellulose ionic liquid solutions is a promising method for producing forest-based CFs precursors [1]. Several synergistic effects of using lignin and cellulose have been identified and affect the performance during spinning and consequently the produced CFs. These effects are very poorly understood and not deeply explored.

In this study, a special focus is on the mass transport of the solvent and non-solvent occurring during the coagulation process, where cellulose and lignin precipitate to form a rigid fiber. Complementary to some theoretical and experimental studies [2,3], in-situ NMR- and MRI-based methodologies are used to characterize the mass transport and solvent exchange under different coagulation conditions, as well as morphological analysis of the coagulated cellulose-lignin films.

A single shot RARE sequence combined with slice saturation was applied and 2D signal intensity maps were recorded throughout the coagulation process. In addition, spectroscopic information in a voxel was measured to characterize and follow the coagulation bath composition over time. ^{13}C solid-state NMR spectroscopy was used to study the morphological structure of cellulose and its crystallinity of the obtained cellulose-lignin films.

The results showed that the coagulation conditions, such as the use of different solvent, non-solvent, or types of lignin, affect the dynamics of the mass transport during the coagulation process, as well as the morphological properties of the obtained cellulose-lignin films.

The use of NMR/MRI methods reveals how variations in the raw materials affect the quality of the fibers, and how the process, and consequently the CFs, can be further optimized

a. b.

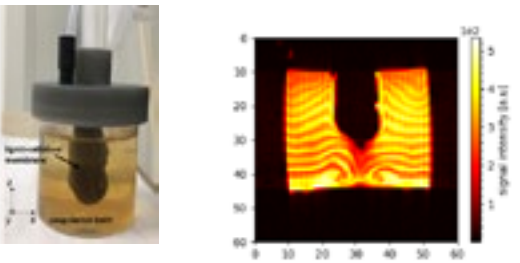

FIGURE. Mass transport measurements: (a) study set-up and (b) 2D signal intensity map in xz plan (geometric axis in mm) showing the dynamics of solvent exchange.

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1D 1H NMR SPECTRA OF NOVEL GLYCERYL MONOOLEATE / GLYCERYL MONOLAURATE LIPID LIQUID CRYSTALLINE NANOPARTICLES

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In recent years, lipid liquid crystalline nanoparticles (LLCNPs) have garnered significant attention as potential drug delivery systems due to their biocompatibility and versatile properties. In this study, we present a preliminary investigation of newly developed lipid liquid crystalline nanoparticles composed of glyceryl monooleate (GMO) and glyceryl monolaurate (GML) using nuclear magnetic resonance (NMR) spectroscopy.

The synthesis of the LLCNPs was accomplished using a top-down approach with sonication as the homogenization method. The nanoparticles were characterized using dynamic light scattering (DLS), cryogenic transmission electron microscopy (cryo-TEM), and small-angle X-ray scattering (SAXS). The DLS analysis revealed a narrow size distribution with an average diameter of 140.23 nm. Cryo-TEM images demonstrated the presence of spherical nanoparticles with a well-defined structure. SAXS results suggest that the nanoparticles exhibit a crystalline structure with cubic Pn3m symmetry.

In addition to the aforementioned methods, nuclear magnetic resonance is another approach to gain insights into the internal structure and molecular dynamics of the LLCNPs [1]. In the presented study, a one-dimensional proton nuclear magnetic resonance (1D 1H NMR) spectrum was evaluated to investigate the degree of internalization of GMO and GML into the structure of the nanoparticles.

In conclusion, this study presents the successful synthesis and characterization of novel lipid liquid crystalline nanoparticles composed of GMO and GML. Further investigations are warranted to explore their performance as drug carriers and optimize their formulation for specific therapeutic applications.

Acknowledgements
The studies were financially supported by The National Centre for Research and Development through grant DWM/WPC2/285/2020.

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^{1}H-NMR low temperature spectroscopy and relaxometry studies of hydration from gaseous phase of foliose lichenized fungi: *Psoroma Hypnorum* from Antarctica.

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The rehydration process of the extremely dehydrated lichenized fungi (*Psoroma Hypnorum*) from the Antarctica were studied. Low temperature (210-295K) relaxometry and ^{1}H-NMR spectroscopy were used - methods allowing one to monitor molecular dynamics of bound water in order to distinguish several fractions of water present in a living cryptobiotic organism (in thallus of dehydrated lichenized fungus). For the dehydrated thallus of *P. hypnorum*, an Antarctic lichenized fungus, the course of gas phase hydration shows a sequential bonding of very tightly bound water fraction, tightly bound water, and finally loosely bound water. The rehydration process for *P. hypnorum* is slower, compared to desert species from less humid region, reflecting the availability of water in the environment[1]. For *P. hypnorum*, the ^{1}H-NMR spectra make it possible to distinguish several water signals, namely the signal from loosely bound water, and the signal from tightly bound water fraction. In the ^{1}H-NMR spectra a chemical shift of the lines can be seen.

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POSTER N^{O} 15

Insights into structure and dynamics of minimal HDV like ribozyme Drz-Mtgn-1

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Hepatitis delta virus (HDV)-like ribozymes are self-cleaving RNAs belonging to the class of small ribozymes, and their widespread occurrence [1] in all aspects of life implies crucial functions in various biological processes. The catalytic reaction follows a general acid-base mechanism assisted by divalent metal ions and directly involves a conserved cytosine to initiate the nucleophilic attack of the 2'-hydroxyl group on the adjacent phosphodiester bond. This ribozyme family differs greatly in length and sequence but adopts a conserved nested double pseudoknot structure with the catalytically important cytosine in the core [2]. So far, only the HDV ribozyme, the eponym and first representative of this family, has been studied in detail regarding structure and catalytic mechanism. However, to understand which structural motives are responsible for the wide range of cleavage rates observed in this family, further structural studies are required.

Here we investigate the Drz-Mtgn-1 ribozyme, which originates from the human gut microbiome and shows a unique bell-shaped self-cleavage activity in the presence of divalent metal ions. This ribozyme belongs to the minimal HDV-like ribozymes, as it lacks a non-essential domain for catalytic activity [3]. Using NMR spectroscopy, we want to determine the three-dimensional structure and disentangle the intricate interplay of this RNA with metal ions. Initial NMR studies reveal that the Drz-Mtgn-1 ribozyme folds into the nested double pseudoknot structure even in the absence of divalent metal ions, thereby forming two critical G·U wobble pairs that are potentially important for the generation of specific metal ion binding sites in the catalytic core.
This study will enlighten the structural basis and the role of metal ions that determine the dynamics of the cleavage reaction and help to bridge the gap between ribozyme's structure and function.

Acknowledgements:
I thank to Dr. Silke Johannsen and Prof. Roland K.O. Sigel for their guidance and all Sigel lab members for their input in this project.

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POSTER N^{O} 16

MECHANISMS OF MOLECULAR MOTION BY MEANS OF NMR RELAXOMETRY

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The development of Fast Field Cycling technology has enabled NMR relaxation experiments ina broad frequency range from about 10KHz to 40 MHz (referring to ^{1}H resonance frequency). There are two main advantages of NMr relaxometry – the first one is the oportunity to probe dynamical processes occuring onmucg different time scales from ms to ps in a single experiment, while the second one is the unique oportunity of revealing the mechanism of the motion.
This is due to the fact that relaxation rates can be described by linear combination of spectral density functions. As spectral density functions are Fourier transforms of the tcorrespinding ime correlation functions, their mathematical forms depend on the mechanism of the motion.
The poster presents a guideline for analysing NMR relaxometry data for hydrated macromoelcules, ilustrated by examples. In the first step, it is shown that the data can always be parametrized in term sof relaxation contributions asscoiated with Lorentzian spectral densities. Such approach, although useful, does not give a deeper insight into the mechanism of motion. Thus, in the next step, it is demonstrated how to attempt to reveal the mechanism of translation diffusion on the basis of the mathematical form of the frequency dependencies of the erlaxation rates, exploiting characteristic properties of the spectral density functions.

Acknowledgements
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A TD-NMR investigation for the food-grade pickering emulsions

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TD-NMR relaxometry is a powerful, non-invasive method to investigate the molecular interactions in food systems. Food systems usually show a multi-component relaxation behaviour thus generating a relaxation spectrum from a T_2 decay curve can and be a great tool to examine the components that are present [1]. T_2 relaxation spectra can provide information about the *water/oil contents and status*, *physical properties of the water*, and *interaction of the water with the surrounding macromolecules* [2]. In this study, we examined pickering emulsions which are emulsions stabilized by hydrocolloidal particles. Pickering emulsions were prepared by pea protein isolate and quince seed mucilage as the hydrocolloids. Sunflower oil was used at a ratio of %20. In order to better visualize this interaction, emulsions with different polysaccharide-protein ratios of 0.01, 0.02, 0.05, 0.10 were prepared and T_2 relaxation times were measured for 14 days. T_2 times were expressed as a biexponential model and the two components were associated with the protons coming from (T_{2a}) water that is restricted by the pickering particles and (T_{2b}) oil droplets that were stabilized. Higher hydrocolloid concentrations (0.5 & 1) were expected to decrease the T_2 times due to higher viscosities. However, this was not the observed case until the end of 1st week. It is likely that pickering particles adsorbed to the oil droplets and decreased the relaxation times. The increase in both T_{2a} and T_{2b} values was less in high concentration emulsions after the 7th day. This trend was also confirmed by the particle size measurements, and low concentration emulsions (0.01 & 0.02) were observed to undergo phase separation in the following days. Differences in the relaxation times were interpreted as different stability mechanisms for the studied emulsions.

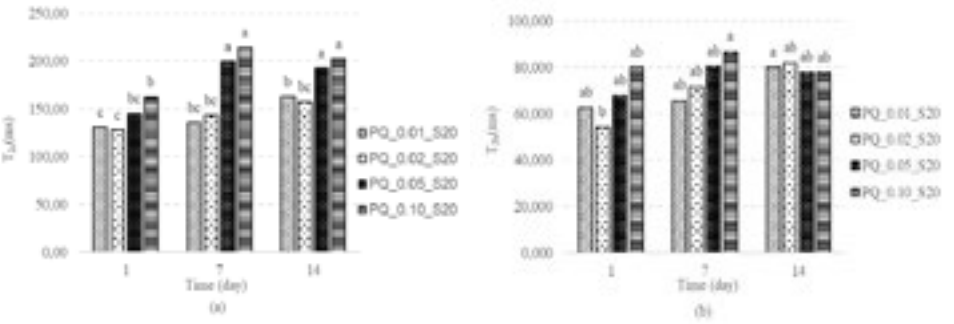

FIGURE 1. T_{2a} (a) and T_{2b} (b) spin-spin relaxation times of pea protein isolate(P) quince seed mucilage(Q) and emulsions containing 20% oil (S20) according to the biexponential model. Different lowercase letters indicate significant difference for each sample ($p < 0.05$).

Acknowledgements

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METHODS OF FUNCTIONALIZATION OF SMART HYDROGEL SURFACES BASED ON HYALURONIC ACID.

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Intelligent hydrogels have the potential for application in therapeutic systems due to their mechanical strength, physicochemical properties, and biocompatibility. Intelligent hydrogels can regulate their hydrophilicity, swelling capacity, and permeability to molecules under the influence of external physical and chemical stimuli such as pH, temperature, electric and magnetic fields, light, ionic strength, and the presence of specific biomolecules. As a result, hydrogels can reduce the frequency of dosing, maintain the desired therapeutic concentration in a single dose, and minimize the side effects of drugs by preventing their accumulation in target tissues. Additionally, they are also used in wound dressings, biosensors, and medical implants [1,2].

The main aim of our study was to design and develop surface-functionalized hydrogels based on hyaluronic acid for post-myocardial infarction therapy. Hyaluronic acid is a natural anionic polysaccharide with a wide range of physicochemical and functional properties, making it highly useful in biomedical applications such as tissue engineering and drug delivery systems [3]. It is characterized by biodegradability, non-toxicity, and the ability to modify its properties through chemical conjugation of functional groups [4].

To prepare potential therapeutic systems characterized by drug loading capability, long lifespan, increased active surface area, selectivity, and good membrane permeability and ease of transport in the body, we synthesized three types of hyaluronan-based nanoparticles. Using an ion gelation method, we obtained a polycationic nanocomplex - pure sodium hyaluronate (SH-NP). As a second hydrogel type, an amphiphilic hyaluronans modified with lithocholic acid (HA-LCA) were obtained. Subsequently, using amine coupling, hyaluronic acid complexes functionalized with β-cyclodextrin with hydrophobic cavities (HA-CD). The structure and composition of the obtained nanomaterials were confirmed using nuclear magnetic resonance spectroscopy, moreover, fourier-transform infrared spectroscopy was employed to examine the diverse surface of the hydrogels and evaluate the network properties of the polymer layers. The obtained systems can be characterized with physical properties similar to living tissues, making them suitable for further investigations as conjugates of Bis-MPA PEG pseudodendrimers functionalized with Apelin-13 polypeptide.

Acknowledgements
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EVALUATION OF MIXING EFFICIENCY IN BUBBLE REACTORS FOR CARBON DIOXIDE CAPTURE USING MRI

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CO_2 absorption is a potential solution to hamper further climate changes. A promising alternative to amine absorbents are alcohol solvents with sodium hydroxide [1]. The efficiency of the CO_2 capture depends heavily on the mixing of the liquid and the gas. Here we evaluated the mixing efficiency of our 3D-printed bubble reactor without forced mixing over time using fast MRI techniques. The absorbent solutions were prepared with 96% ethanol, 4% water and NaOH at concentrations of 2-10 g/L. The gas was a mixture of 30% CO_2 and 70% N_2. Experiments were conducted in a 3D-printed bubble column reactor at ambient pressure and temperature. The reactor was placed inside an MRI (Bruker Avance III 300MHz with a H^1 transmit/receive probe of 66 mm diameter) and the gas was sparged continuously into the reactor. Figure 1(a) depicts the experimental setup. One-shot RARE images and flow maps were recorded throughout the reaction. During CO_2 absorption, a gel is formed containing most likely a mixture of inorganic and organic carbonates. This gelation hinders further absorption as it inhibits good mixing and further dissolution of CO_2. One-shot RARE images (Figure 1(b) and (c)) enabled monitoring of the liquid which became gradually more viscous. High signal intensity (bright yellow) corresponds to the liquid, while low intensity corresponds to regions where the gas is creating turbulence. Good mixing regions could be identified based on the intensity of the signal. For the 2 g/L NaOH solution, better mixing was observed due to less gel formation. We also found that the positioning of the sparger is crucial to minimize regions with bad mixing. MRI provided an understanding of the fluid dynamics inside a bubble column. In cases where the liquid phase is opaque, or physicochemical changes affect the process, conventional methods like high-speed cameras tend to fail. Fast MRI can enable the optimization of bubble reactor designs [2].

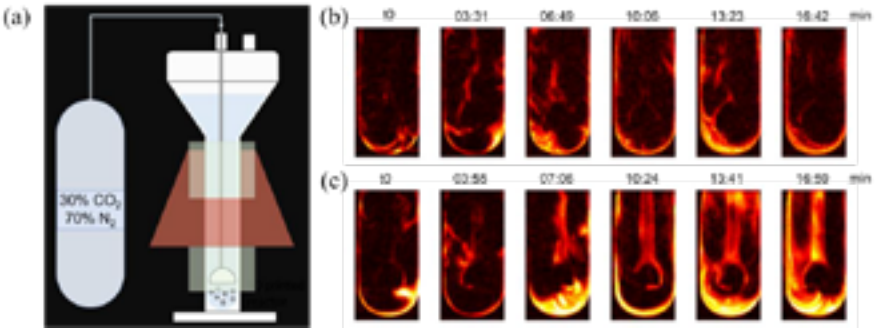

FIGURE 1. (a) Experimental setup and sagittal RARE images over time during sparging gas into a solution of (b) 2 g/L of NaOH in ethanol and (c) 10 g/L of NaOH in ethanol.

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In-House Fabrication of Microwave Waveguides and Resonators for Efficient Dynamic Nuclear Polarization

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Dynamic nuclear polarization (DNP) is an NMR hyperpolarization technique that facilitates the transfer of polarization from unpaired electron spins to nuclear spins. To achieve efficient DNP performances, low-loss corrugated microwave waveguides and tunable high-Q dual resonance (RF and microwave) TE_{011} resonator is required [1, 2]. We will present the methods to fabricate the 9.4 T/ 263 GHz microwave components using additive (3D printing) or subtractive (CNC machining) manufacturing processes. For the additive approach, we will first 3D print the components with plastic (ABS or similar) materials, followed by either electro or electroless plating with copper. For the subtractive method, we will machine a manual tap using a combined 5-axis CNC machine and a manual lathe fabrication process. The tap will then be heat treated (via tempering and/or annealing) for more favorable mechanical properties. These microwave accessories are expected to yield better DNP and EPR performances.

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Novel 1D ZnO Nanomaterials with Promising Photoelectrochemical Properties for Biosensing and Water Splitting Applications

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Antigen rapid detection tests are more frequently used to identify the SARS-CoV-2 viral proteins. Despite being less sensitive than molecular tests, antigen rapid detection tests have the advantages of being inexpensive and providing results quickly at the point of care. Additionally, they can detect target analytes with low concentrations which makes them advantageous for early diagnosis of the disease [1]. To reduce the spread of the virus, it is necessary to develop diagnostic tests that can rapidly identify the infected individual. ZnO possesses good physical and chemical stability, sustainability, high energy conversion efficiency, sensitivity, and abundance, which make it promising for photoelectrochemical applications, such as SARS-CoV-2 antibodies detection [2] and water splitting [3]

In this work, we present $ZnFe_2O_4$/ZnO core-shell electrospun fibers as working electrode in standard three-electrode configuration, the studied nanostructures revealed catalytic activity without applied solar-light radiation and up to 45% incremental photocurrent growth with solar-light radiation. And 1D ZnO nanorods used as photoelectrochemical immunosensor. In comparison with traditional methods of antibody detection, the present study offers several advantages such as low cost, portability, and high accuracy. With continuous research and development, the 1D ZnO based electrochemical sensor will play an important role in rapid and sensitive diagnostic tools for the monitoring of infectious diseases.

Acknowledgments

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*Both authors contributed equally to this research.

STRUCTURE AND MOLECULAR INTERACTIONS IN AMMONIUM IONIC LIQUIDS

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Ionic liquids, organic/inorganic salts with melting points below 100 ºC, are typically characterized by a variety of characteristics, from which common to great group of ILs are low vapor pressure, a wide range of liquids, high thermal, electrochemical and chemical stability, and the ability to dissolve many organic and inorganic compounds, including some polymers. The construction of the anion and cation determines their unique properties, which can be changed to create an ionic liquid with the required physical and chemical properties. The key advantage that sets ILs apart from conventional solvents is that they may be projected, and for this reason, ILs might be referred to as green solvents utilized in industrial and laboratory applications.

This study chose cations of different structure (aromatic, cyclic, and aliphatic), different alkyl chain length attached to the quaternary nitrogen, and bis(trifluoromethanesufonyl)imide anion, which results in three homologous series of ionic liquids with the cation of different geometry, with different alkyl chain lengths attached to the quaternary nitrogen. The main aim was to evaluate the macro- and microscopic structure of the synthesized ILs.

After structure confirmation by means of NMR and FTIR (collected with a FT/IR-4700, JASCO), phase transition temperatures were determined by means of differential scanning calorimetry technique (with a DSC 8000, Perkin Elmer unit). Moreover, Small-angle X-ray scattering studies were performed using the Xeuss 2.0 SAXS/WAXS system. Finally, self-diffusion coefficients were determined for series of the prepared ILs measurements at 14,4 T Agilent NMR spectrometer techniques (DOTY DSI-1372, gz=28 T/m, VnmrJ 4.2; Pulse sequence: PGSE (Pulsed Gradient Spin-Echo).

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POSTER N^{O} 23

WATER DYNAMICS IN ARTIFICIAL TISSUES BY MEANS OF NMR RELAXOMETRY

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^{1}H NMR relaxometry experiments have been performed for examples of materials used in regenerative medicine ("artificial tissues'). The experiments have been performed in the frequency range from 10kHz to 20MHz at 25˚C and 38˚C. The purpose of the studies is to reveal dynamical and structural properties of selected materials used in regenerative medicine in connection to their macroscopic properties (such as viscoelasticity) and get insight into the mechanism of water motion and interactions of water molecules with the macromolecular matrix. In Fig.1 (left) examples of the ^{1}H spin-lattice relaxation data are shown in comparison with solid collagen, considered as a model system. In Fig.1 (right) one sees examples of ^{1}H magnetization for the hydrated material – the relaxation process has turned out to be single-exponential.

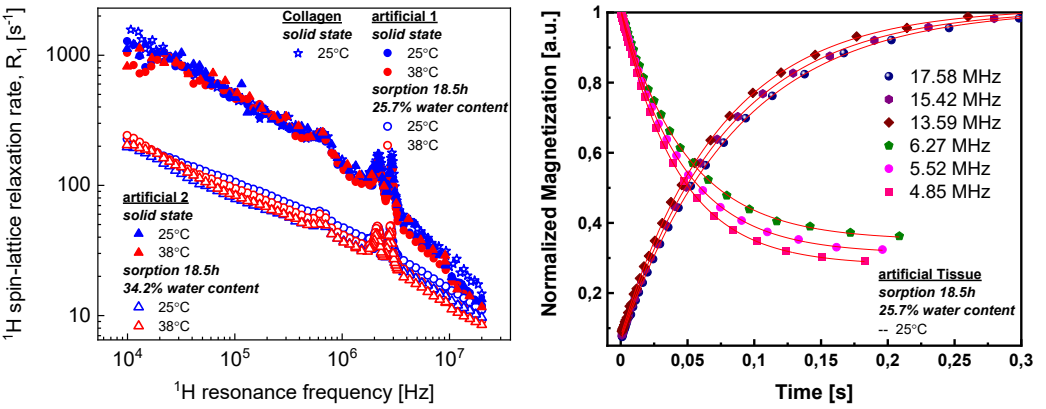

FIGURE 1. (left) ^{1}H spin-lattice relaxation data for artificial tissues compared with analogous data for solid collagen; (right) examples of ^{1}H magnetization versus time for hydrated arificial tissues.

The data have been thoroughly analyzed in terms of advanced models of relaxation processes, including Quadrupole Relaxation Enhancement effects [1]. The analysis has led to a series of parameters characterizing the dynamics of different moelcular fractions present in the materials and revealed the mechanism of water diffusion in the vicinity of the macromolecular surfaces.

Acknowledgements

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Analysis molecular dynamic processes in the complex of human S100B protein with Aβ peptide on base ^{15}N relaxation data

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S100B proteins are a type of calcium-binding protein that play a crucial role in the regulation of various processes associated with Alzheimer's disease (AD). In AD patients, neuroinflammation leads to an increase in the levels of S100B proteins in the brain. This heightened presence of S100B proteins plays a significant role in the processing of the amyloid precursor protein (APP), the regulation of amyloid beta peptides (Aβ), and the phosphorylation of Tau. The interactions between S100B and Aβ amyloid suggest that S100B has a regulatory role, which can vary depending on the stage of AD and the level of Aβ presented in cell [1].

In our studies we explore structural alterations observed under interaction of the uniformly ^{15}N-labeled human S100B in Ca-loaded form with A$\beta_{1\text{-}40}$ peptide utilizing heteronuclear NMR spectroscopy. Analysis of chemical shift perturbations reveals the α-helices I – I' together with the α-helices IV – IV' as a structural motif involved in binding of the A$\beta_{1\text{-}40}$ peptide (Figure 1), as previously reported by other group for A$\beta_{1\text{-}42}$ [2].

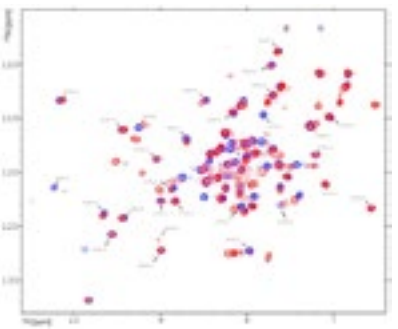

Figure 1. Overlay of ^{1}H-^{15}N HSQC spectra collected for ^{15}N-labeled S100B (blue) and after addition A$\beta_{1\text{-}40}$ peptide (red).

The ^{15}N relaxation experiments, including R_1, R_2, and ^{1}H-^{15}N *NOE*, were conducted at a temperature of 298K and a magnetic field strength of 11.7 T for the S100B protein both in the absence and presence of A$\beta_{1\text{-}40}$. The analysis of relaxation data was performed using the Spectral Density Mapping (SDM) approach. The obtained values of spectral densities at frequencies of 0, ω_N, and ω_H demonstrated changes in the dynamics of the protein backbone upon interaction with the A$\beta_{1\text{-}40}$ peptide. Specifically, a decrease in the $J(\omega_N)$ function was observed for the majority of the ^{15}N, indicating a reduction in molecular dynamic processes in the S100B backbone within the μs – ns time-frame. Simultaneously, increased mobility was observed for the residues Ser41 and His42, which are located in the loop between α-helices II and III.

Acknowledgments
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FREQUENCY OPTIMIZATION IN MULTIDIMENSIONAL DIFFUSION-RELAXATION CORRELATION MRI ON A FIXED MOUSE BRAIN

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Multidimensional diffusion-relaxation correlation magnetic resonance imaging (mdMRI) is a recently-developed technique that simultaneously measures and correlates diffusion and relaxation times to overcome the limitations of traditional measurements. At the core of our mdMRI technique is the tensor-valued encoding of the diffusion measurements[1], which provides control over the range of frequencies probed in the diffusion spectra. Having a wide range is useful for probing restricted diffusion[2]. However, due to hardware constraints, achieving higher frequencies at constant b-values requires increasing echo time (TE), sacrificing signal-to-noise ratio (SNR). How large of a range is achievable without losing too much SNR is a particularly important problem for samples with short T_2, like our mouse brain sample fixed in 2% formaldehyde, T_2 approximately 25 ms.

To find the optimal experimental settings for our sample, we designed several acquisition protocols covering different frequency ranges at each b-value and correspondingly different minimum TEs. At the max b-value of $6.7*10^9$ s/m^{-2}, the protocol with the smallest range of 45-111 Hz has minimum TE 46 ms. The protocol with the largest range of 30 – 228 Hz has minimum TE 63 ms. Fitting was done with a custom Monte-Carlo inversion algorithm[3], producing distributions for each voxel in the solution space from which we extract statistical descriptors for the solution distribution.

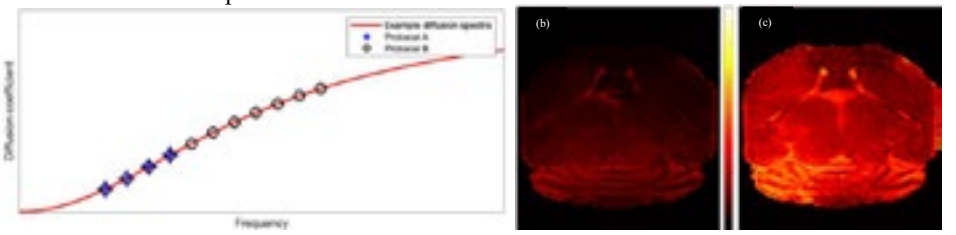

FIGURE 1. (a) Illustrative plot of experimental design and result. Plotted on a generic diffusion spectra, protocol A represents one with a small frequency range, while protocol B represents one with a large frequency range. Plots (b) and (c) show the change in D_{iso} per unit frequency, obtained from a protocol with the smallest and largest frequency range respectively. Color bar range: 0-6e-12 m^2.

We observed the strongest sensitivity to frequency for both mean isotropic diffusivity (D_{iso}) and squared normalized anisotropy (D_Δ^2) values for the protocol having an intermediate frequency range of 33-215 Hz, minimum TE 54 ms at max b-value. While in general larger ranges should result in higher sensitivities, the associated decrease in SNR counteracts that effect.

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LUNG CANCER RADIOMICS INVESTIGATION: CT-MRI FEATURES COMPARISON AND SOFTWARE CONSISTENCY

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Radiomics is a quantitative method that allows to extract mineable data from medical images. The aim of Radiomics is to improve clinical decision-making effectiveness defining novel imaging biomarkers, labelled *radiomic features*, able to reflect tissue properties. Currently, Radiomics has not yet been introduced in clinical routine mainly because of its poor generalizability and reproducibility [1]. Within this framework, our purpose is to preliminary investigate the statistical reliability of radiomics features extracted from Computed Tomography (CT) and Magnetic Resonance Imaging (MRI) lung cancer images, and the consistency between two open-source radiomics platforms, LIFEx [2] and Pyradiomics [3].
26 lung cancer patients were retrospectively included in the considered cohort. For each patient, both CT and MRI images have been manually segmented using the ITK SNAP tool. Three different images pre-processing methods were considered, acting on voxels resampling and grey-level discretization. From the pre-processed images, 66 features *IBSI-compliant* [4] common to the two radiomics software were extracted and organized in datasets. At this point, the correlation between Pyradiomics and LIFEx features, for each imaging technique, and the correlation between CT and MRI features, for each radiomics platform, were investigated. The correlation was analysed both graphically, through graphs, and quantitatively, by the *Spearman test* and the *Intraclass correlation coefficient* (ICC).
Among LIFEx and PyRadiomics software, statistically significant correlations were found for 88% of CT-derived features and 91% of MR-derived features. Comparing CT and MRI images, significant correspondences were found for 15% of LIFEx features and 12% of PyRadiomics features. The obtained results suggest that the IBSI standardization is quite effective even when two radiomic software are considered, and that CT and MRI images contain different, non-redundant information, paving the way for a future multi-modal radiomics analysis.

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Insight into Mass Transfer within a Superabsorbent Polymer Using MRI

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Absorption-based technologies are widely used for many applications, particularly in hygiene products for the absorption of body fluids. Designing efficient absorbents with high capacity requires better insight into the mechanism of the absorption. It means an attempt to unravel the role of intermolecular interactions and swelling on the behavior of absorbing fluids. Although it is a commonly accepted fact that the absorption performance of hygiene pads can be affected by operational conditions, such as the ratio of used materials, determining the extent to which parameters modify the behavior of absorbed fluid has proven to be challenging and contentious [1,2]. To further elucidate the water and salt behavior at a molecular scale in these environments, the magnetic resonance imaging (MRI) technique was implemented to evaluate water absorbed in the super absorbent polymers (SAPs). Some principal parameters, including the T_2 relaxation time, were noticed to be significantly dependent on the ratio of the fluid/polymer, as well as the particle size of the absorbents. The outcome elucidated that the absorption inside the SAP samples is not homogeneous and the salt ions enhance the observed heterogeneity. In this regard, the intermolecular forces between ions and the polymer chains seem to make the polymer act like a filter. It means that sites on the polymer chains are occupied by sodium ions and, consequently, the number of possible interactions between water molecules and polymer chains seems to drop [1]. Moreover, T_2 relaxation time showed a descending trend as the fluid/polymer ratio and particle size were reduced expressing more interaction or lower mobility due to higher chain density per volume.

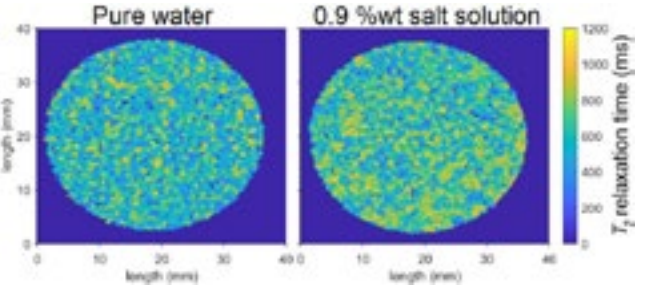

FIGURE 1. Axial maps of long T_2 relaxation times of fluids within the SAP sample with a particle size 300-600 μm.

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Investigation of the interaction of sodium ions with ZIF-8 in sodium-ion battery electrolytes using ^{23}Na and ^{1}H NMR.

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Sodium-ion batteries (SIBs) are attracting a lot of investigation and commercial interest at present. They are considered the most promising alternative to lithium-ion batteries (LIBs) due to their similar chemical properties, far greater availability of natural resources and resultant low cost, reduced carbon footprint, and, above all, increased safety [1].

Because sodium and lithium batteries have comparable structures and operating mechanisms, many advances in lithium-ion technologu can be leveraged to increase the performance of SIBs through optimization of electrode materials and electrolytes. In this context, ^{23}Na NMR and ^{1}H NMR techniques are used to explore the incorporation of sodium electrolytes with a porous MOF (Metal Organic Framework) material, such as the highly porous ZIF-8 (Zeolitic Imidazolate Frameworks) [2]. These methods were chosen because of their capacity to precisely follow sodium ion and proton dynamics, which is critical for understanding the interaction between the electrolyte and the porous ZIF-8 material. The findings of these investigations can help evaluate whether sodium ions interact with MOFs and flow inside the structure or are maintained on the surface, and how this impacts ionic conductivity. This understanding can help researchers grasp how MOFs can be utilized to improve the performance of sodium-ion batteries, particularly those used to store energy. The combined use of electrolytes with MOFs is an intriguing area of research for future advancements in battery technology.

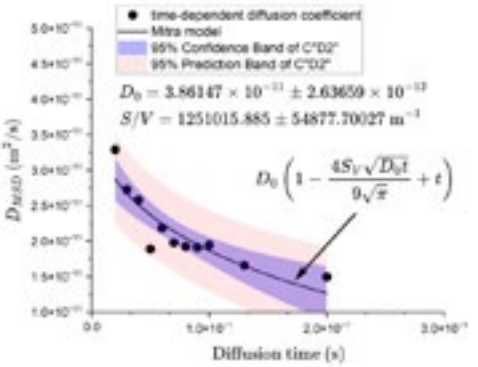

FIGURE 1. Time-dependent diffusion coefficients of PC as obtained in ZIF-8-PC. [3]

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^{13}C SOLID-STATE NMR PROVIDING CRUCIAL INSIGHTS INTO PULPING OF NON-WOODEN CELLULOSE RESOURCES

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The demand for textile fibres continues to increase due to the steady growth of the world's population. Cotton being one of the main resources for textile fibre production cannot meet the market needs due to a limited production capacity. During the last century wood was the primary resource for cellulose for both paper and textile industry. Its pulp is preferred due to its long fibres and all-year availability.

Albeit containing cellulose and enabling a circular cellulose economy, very little is know about pulping of non-wooden resources such as straw, bamboo, bagasse and reeds, corn straw, reed, grass, jute, flax, sisal etc. In comparison to wood, the hierarchical organization differs severely due to different purposes e.g. oat husks' purpose is to protect the grain.

Apart from this, cellulose consists not only of amorphous and cyrstalline fractions—the crystalline fraction has many allomorphs. The most common ones being native cellulose—cellulose I and regenerated cellulose II. The production of textile fibres includes dissolving the cellulose pulp and coagulation in an anti-solvent during the spinning process. One sustainable dissolution system is cold alkali. It was however observerd that the dissolution of the cellulose II allomorph in cold alkali is impared. Pulping is a multiple step process—prehydrolysis, cooking and post-treatments such as bleaching. Each of these steps might if not optimized cause partial transformation of cellulose from native cellulose to cellulose II—preventing the pulp from dissolution.

^{13}C solid-state NMR is a crucial and common technique to analyse cellulose and their excisting allomorphs (see Fig. 1). Here we applied ^{13}C solid-state NMR to follow the cellulose pulp from oat husks and wheat straw during the different pulping steps. We aim to optimize the pulping process to enable dissolution in cold alkali for the production of sustainable textile fibres.

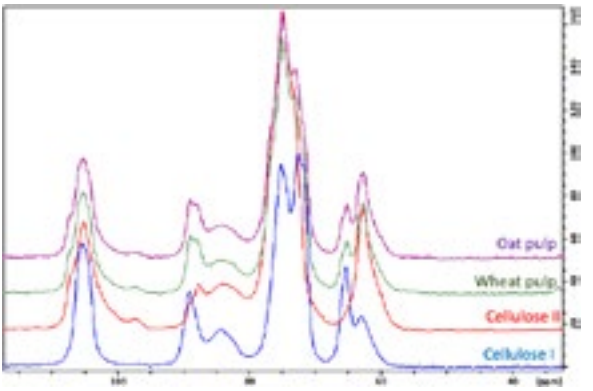

FIGURE 1. Solid state NMR spectra of various cellulose samples

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THE POSITIVE INTERPLAY BETWEEN IMAGING AND THERAPEUTIC AGENTS ENCAPSULATED IN THERANOSTIC LIPOSOMES TARGETING ORAL CANCER

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The theranostic liposomes are a multifunctional approach that combines diagnosis and treatment [1]. We reported an improved relaxation (resulting from the presence of zinc phthalocyanine, ZnPc), possibly improving MRI contrast and potentially allowing a reduction of the dose of co-administered lipid analog of Magnevist® (PE-DTPAGd), keeping the performance unmodified [2, 3]. The aim of the study was to replace ZnPc with a marketed photosensitizer, verteporfin (VRP), and verify our previous observations. Additionally, the theranostic liposomes were conjugated with an anti-EGFR antibody and its efficacy was evaluated in a cellular model of head and neck squamous cell carcinoma (HNSCC).

Liposomes were prepared by the microfluidic method (MF). Loading efficacy was determined using HPLC (VRP, ZnPc) or NMR (PE-DTPAGd). Vesicle size and zeta potential were characterized by dynamic light scattering. Relaxometric measurements were performed at different magnetic fields at 23°C and 37 °C. *In vitro* cytotoxicity and photosensitizing activity were evaluated in two HNSCC cell lines in 2D and 3D cell culture models.

The size of theranostic liposomes prepared by MF was 30-50 nm, while a slight increase in diameter was noted for antibody-conjugated liposomes (*ca.* 60 nm). The presence of VRP in the liposomal bilayer led to increased relaxivity of Gd(III) ions in theranostic liposomes compared to paramagnetic vesicles (without VRP but with PE-DTPAGd), which is in agreement with results obtained for ZnPc-loaded liposomes. The VRP encapsulated in targeting theranostic liposomes effectively inhibited cancer cell growth in a 2D model of SCC-25 and FaDU with IC_{50} of 16 nM and 45.3 nm, respectively, as well as in a 3D model with corresponding IC_{50} values of 61.5 nM (SCC-25) and 108.3 nM (FaDu).

Changes inside the liposomal bilayer upon dye incorporation among phospholipids` molecules result in increased water permeability across the liposomal membrane and relaxivity of incorporated MRI CA. Theranostic liposomes allow the delivery of higher doses of VRP than can be achieved for the free drug allowing the effective photodynamic treatment of HNSCC spheroids.

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POSTER N° 31

NMR DEPTH PROFILING OF PAINTED WALLS: OSTIA ANTICA

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The mortar-layer stratigraphy of Roman wall paintings at Ostia Antica, the historic port of ancient Rome, was studied at two occasions with nuclear magnetic resonance (NMR) depth profiling [1] to explore manufacturing details in the context of the use and history of the buildings. In the first campaign in 2019 the NMR signature of covered wall paintings was studied [2,3], while in the second campaign in 2022, depth profiles were compared to answer the question if different painted walls were prepared for painting at the same time or by the same workshop [4]. The experience gained in handling the portable NMR hardware for onsite measurements has stimulated the development of an improved NMR depth-profiling instrument [5,6]. The information provided by NMR depth profiling is illustrated with experimental data, and progress in hardware development is reported.

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Contrast Enhancement in MRI Using Combined Double Action Contrast Agents and Image Post-Processing in the Breast Cancer Model

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MRI provides the best soft tissue contrast when comparing with other diagnostic modalities. Relaxation times T_1 and T_2 of normal tissue and tumor create the contrast in MRI. Contrast agents shortening T_1 and T_2 relaxation times further improve detection of small pathologies such as early stages breast or brain cancers [1]. Furthermore, images can be post-processed to improve contrast. We studied changes in tumor contrast before and after non-targeted $NaDyF_4/NaGdF_4$ contrast injection. Then to further increase the contrast we applied an image subtraction and addition. Therefore, we performed theoretical calculations of MR signal in a tumor model using T_1-weighted, T_2-weighted, and combined images for T_1-, T_2-, and T_1/T_2-targeted contrast agents. We imaged a mouse with triple negative breast cancer (TNB) before and after an injection of a nontargeted $NaDyF_4/NaGdF_4$ nanoparticles (NP). We applied the RARE (T_1-weighted), and MSME (T_2-weighted) pulse sequences, using a 9.4 T MRI system. The results show that subtraction of T_2-weighted from T_1-weighted MR images provides additional increase in the tumor contrast: over two-fold in the tumor model and 12% in the in vivo experiment. We concluded that an image subtraction improved tumor contrast hence it may improve the accuracy of cancer detection [2].

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EXPLORING TEMPERATURE RELATED PROTON DYNAMICS IN HYDRATED STARCH BY TIME DOMAIN NMR

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The use of various dry thermal treatment techniques to modify starch properties have gained increasing attention in recent years [1]. However, its effect on the hydrothermal transformation upon heating is not fully understood. Wheat starch isolated from roasted (115ºC for 165 s) and unroasted wheat as well as maize starch were used to investigate the temperature-dependent changes in starch-water mixtures by Time Domain-Nuclear Magnetic Resonance (TD-NMR). T_2 relaxation spectra are appropriate for analysis of heterogeneous food samples to investigate its multi-component behaviour [2]

Molecular properties of starch-water systems at 50% moisture content were characterised from 20 to 90ºC by Rhim Kessemeier-Radiofrequency Optimised Solid Echo (RK-ROSE) and Carr-Purcell-Meiboom-Gill (CPMG) pulse sequence for detection of the solid and liquid phases, respectively. Six proton populations were identified. For the component of shortest relaxation time (T_{2r}*), the contribution of the amorphous and crystalline part within the semicrystalline structure were detected by comparing the second moment values to the relaxation times. Water absorption by the different starches and the associated granule swelling were observed up to 50ºC by the lowered relaxation times (T_{2i}*, $T_{2(2)}$ and $T_{2(3)}$). The displacement of the proton relaxation time distribution ($T_{2(3)}$) towards shorter times suggests a reduction in proton mobility. This is due to water migration from the outer to the inner region of the granule. On the other hand, the reduction in the fraction of the shortest component (T_{2r}*) suggests the loss of structured semicrystalline regions due to the increase in proton mobility upon heating.

The second, and most abundant component detected by CPMG provides information on the onset of gelatinisation. A stabilisation in the reduction of $T_{2(2)}$ occurred at similar temperatures as the onset of gelatinisation detected by differential scanning calorimetry and intial structural deformation detected by the complex modulus (G*). This study proved that TD-NMR can be used to accurately demonstrate the fast changes in water mobility during heating of starch-water systems; as observed for dry heat treated wheat and maize starches.

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AMPERE NMR School

June 18th - June 24th 2023, Zakopane, Poland

PROGRAMME

SUNDAY, June 18th		MONDAY, June 19th	TUESDAY, June 20th	WEDNESDAY, June 21st	THURSDAY, June 22nd	FRIDAY, June 23rd	SATURDAY, June 24th
Afternoon Arrivals & Accommodation	8:00-9:00	*Breakfast*	*Breakfast*	*Breakfast*	*Breakfast*	*Breakfast*	*Breakfast*
	9:00-9:45	**Anja Böckmann** Solid-state NMR of the Multidrug ABC Transporter BmrA	**Adolfo Botana** Practical aspects of 13C qNMR	**Pedro José Sebastião** Observing short-range orientational order: from liquid crystals to small molecule liquids	**Olivier Lafon** NMR of quadrupolar nuclei at high magnetic fields	**Leonid Grunin** Analysis of Strongly Dipolar Coupled Solids by Time-Domain NMR	*Departure*
	9:45-10:30	**Siegfried Stapf** Bicomponent fluids in porous media: what NMR can do for you	**Mecit Oztop** TD-NMR & MRI Applications in Food Systems	**Esteban Anoardo** Low field MRI solutions: fixed field, pre-polarized or field-cycled	**Magdalena Wencka** 69Ga and 71Ga as probes of a local symmetry in diamagnetic complex metallic phases	**Janez Stepišnik** Chemical exchange rate studied by NMR CPMG method	
	10:30-11:00	*Coffee Break*	*Coffee Break*	*Coffee Break*	*Coffee Break*	*Coffee Break*	
	11:00-11:45	**Beat Meier** Protein fast MAS experiments at high field	**David Lurie** Basics of MRI & Field-Cycling Imaging	**Bernhard Blümich** Asymmetry in Three-Site Relaxation-Exchange NMR	**Lucia Calucci** Looking into porous materials with solid-state NMR and relaxometry	**Maria Beira** Assesing the Molecular Dynamics in paramagnetic liquid systems	
	11:45-12:30	**Adolfo Botana** The magical world of pure shift NMR	**Ville-Veiko Telkki** Effect of surfactant aggregation on cloud formation	**Daniel Topgaard** Translational motion and magnetic field gradients	**Giacomo Parrigi** Field-cycling NMR relaxometry of paramagnetic-labeled proteins	**Claudia Schmidt** Ionic-liquid based hybrid gel polymer electrolytes as seen by NMR	
	12:30-13:15	**Wiktor Koźmiński** High dimensionality and high resolution NMR experiments for biomolecules	**Felice D'Alia/ Ahmed Dhifaoui** JEOL - The Luminous NMR on cloud formation	**Arkadiusz Leniak** Exploring NMR Laboratory Operations in Medicinal Chemistry: Insights from Everyday Challenges at Celon Pharma SA	**Danuta Kruk** NMR relaxation processes in solutions of superparamagnetic nanoparticles	**Adriane Auccaise** Challenges of non-exponential NMR relaxation processes	
	13:15-14:00	*Lunch*	*Lunch*	*Lunch*	*Lunch*	*Lunch*	
	14:00-16:15	*Free Time*	*Free Time*	*Excursion*	*Free Time*	*Free Time*	
	16:15-16:30	*Coffee Break*	*Coffee Break*		*Coffee Break*	*Coffee Break*	
	16:30-17:15	*On-line laboratory training* **Adolfo Botana** JASON workshop: practical aspects *Group 1* **Jacek Jenczyk** Magic Angle *Group 2*	*Poster session*		*On-line laboratory training* **Tomasz Zalewski Marek Kempka** MRI: basic principles and applications *Group 1* **Elżbieta Masiewicz** FFC NMR *Group 2*	*On-line laboratory training* **Michał Bielejewski Kosma Szutkowski** NMR diffusometry	
	17:15-18:00	*groups rotation* **Jacek Jenczyk** Magic Angle *Group 1* **Adolfo Botana** JASON workshop: practical aspects *Group 2*			*groups rotation* **Elżbieta Masiewicz** FFC NMR *Group 1* **Tomasz Zalewski Marek Kempka** MRI: basic principles and applications *Group 2*	*17:45* **Closing ceremony**	
			18:45 - walk to ...				
19:45 *Opening dinner*	19:00	*Dinner*	*Dinner at the Regional Restaurant 19:15*	*Dinner*	*Dinner*	*Dinner*	