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Synthesis, structure and properties of complex transition metal salts containing various ligands and oxidizing anions

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The aim of our work was – continuing our previous efforts [1-5] – the preparation and structural characterization of various transition metal complexes having different kinds of central metal ions (Fe^{III}, Co^{III}, Ag^{I}), reducing ligands (NH_3, pyridine or urea) and oxidizing anions (NO_3^-, MnO_4^-, ReO_4^-, ClO_4^- etc.). Structural characterization has been performed by single crystal X-ray diffraction and various spectroscopic (Mössbauer, IR, Raman) methods. In the case of all studied complexes there are extended hydrogen bond networks formed by N-H and X-O (X=N, Mn, Re, Cl) functionalities verified by single crystal X-ray diffraction. The hydrogen bonding can act as a reaction centre during the thermal treatment and facilitates a redox reaction between the reducing ligands and the oxidizing anions. This heat-induced solid phase reaction ensures a convenient way to prepare simple or mixed transition metal oxides with nanometer sized particles from transition metal complexes ($[ML_n](XO_4)_m$ where M = Fe, Ag, Co; L = Urea, NH_3 or pyridine, and n= 2–6; X = Mn, Re, N, Cl, and m= 0.5–3. These nanosized oxides are excellent catalysts in various industrial processes, like the Fischer-Tropsch synthesis [6].

Studies on thermally treated hexaurea-iron(III)-permanganate had been done. Amorphous, micro sized, mixed iron-manganese oxides could be observed even at low temperature (~ 100 °C) due to a solid phase redox reaction between the oxidizing anion and reducing ligand. Catalytic activity tests of these mixed oxides had been done. It was proved that these mixed oxides are catalytically active with various selectivity and conversion rates in the hydrogenation of CO_2 producing CO, CH_4, C_2H_6 and C_3H_8 [3].

In the case of urea and urea derivatives (N-methylurea, N,N-dimethylurea, N,N'-dimethylurea) containing Fe^{III} it is important to explore how the methyl substitution can change the hydrogen bond system (through the electron density change inside the urea molecule). Mössbauer spectroscopic results show that the substitution of the H atoms by methyl groups causes drastic decrease in the Debye-temperature (Θ_D). The explanation for this change may be related to the additional molecular/lattice vibrations due to the presence of the methyl groups and to a less extended H-bonding network.

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Absolute determination of uranium enrichment calibration curve by high-resolution gamma spectrometry of a measured gamma-ray spectrum of an unknown sample using a non-destructive measurement approach for nuclear safeguards purposes

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Illicit traffic of nuclear materials has increased tremendously over the past years and has become imperative to apply fast and reliable measures or methods for uranium enrichment determination to ascertain its enrichment level. As a regulatory authority in charge of overseeing all nuclear materials subject to safeguards within the State, methodology or techniques in the determination of uranium enrichment have become something challenging as a competent State regulatory authority because there is a lack of expertise in the area. This work seeks the address how regulatory safeguards inspectors can perform uranium enrichment determination of known and unknown spectra using mass spectroscopy. This is to improve the competency of inspectors in the area and to have an expert to address some of the challenges when it comes to uranium enrichment determination using gamma-ray detectors. In order to determine the uranium enrichment the activity ratios of 234U/235U and 238U/235U were measured. Uranium isotopic abundance can be determined by alpha spectrometry and mass- spectrometry methods, which are destructive methods. This work presents the non-destructive gamma–spectroscopic method for uranium enrichment. First, a gamma-ray detector was used to measure a 207Bi calibration source to check the proper functioning of the detector. This measurement allows the energy calibration of the detector. The gamma-ray detector was then used to measure a set of 4 CBNM U3O8 sources with known enrichment. This set of measurements allows the development of a calibration curve for the Uranium enrichment determination. Finally, the gamma-ray detector was used to measure an additional CBNM U3O8 source with unknown enrichment with the aim of determining the Uranium enrichment. The measurements of the CBNM sources were carried out in the so-called infinite thickness conditions. According to this approach, the collimated detector is sensitive only to a portion of the measured sample due to the size of the measured sample and the self-attenuation of the 185.7 keV gamma-ray emitted by 235U. The method is applicable to the material of any physical form and geometrical shape. Two methods called 3-ROI and 2-ROI were used for the data analysis of the CBNM sources, and finally, to make a comparison of the results. The obtained result agreed as expected.

Scattering Studies on the Bioflocculation Process

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Solid and liquid separation is a crucial step in wastewater treatment plants (WTPs). In a WTP flocculation is performed in the secondary clarifier on the lean mixture of activated sludge and during flocculation, microorganisms spontaneously form large, dense, and quick settling flocs. These bioflocs contain porous fractal-like aggregates. Therefore, it is important to know the fractal dimension of the bioflocs, which is very critical to understand the dewatering process from the activated sludge. The aggregates' fractal dimension governs how quickly the activated sludge in the lean mixture can be dewatered, and the literature lacks in predicting the exact nature of the complex aggregates formed in the actual treatment process, their structure and morphology. Here, I will present the results of the scattering and aggregation kinetic studies carried out on a on a model system containing an industrial flocculating agent and microspheres.

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Tailor-designed Metal-Organic Framework@Polymer composites for water remediation

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Metal-Organic Frameworks (MOFs) have emerged as highly promising porous and crystalline materials to address heavy metal pollution, overcoming the adsorbing capacities and kinetics of classic adsorbents [1]. MOFs are formed by easily tunable inorganic and organic building blocks arranged in a porous and ordered fashion, creating a crystalline structure with large surface areas. Among all the broad variety of MOFs, these ones assembled from Zr(IV) and C4-dicarboxylic acids as linkers are highly robust and versatile materials. When adding a -SH, NH3, -COOH and -SO3 as pedant groups of the C4-linker, we can tune the adsorption capacities of the framework towards the capture of heavy, intermediate and soft metal-ions.

By modifying the synthesis conditions, and specially the concentration and addition of acid modulators, we have obtained the cubic (fcu) and hexagonal (hcp) polymorphs of the materials, which show different arrangements of the -SH, -NH3, -COOH and -SO3 functional groups in these polymorphic structures.

Indeed, this arrangement affects the adsorption properties of the materials towards metal ions. Giving a step further, we have combined in a synergic-manner the different functional groups into cubic and hexagonal MTV-MOF frameworks. Finally, after the incorporation of the MOF-materials into polysaccharide based polymer matrixes, we have obtained composite-filtering systems that can be used as chromatographic metal colums. Depending of the functional groups installed in the MOF, we are able to tune the specificity of the composite filters towards the capture of (i) soft heavy metals as Hg(II), Cd(II), Pb(II); or (ii) intermediate acidity metals ions as Cu(II), Co(II) or Ni(II), or (iii) hard metal ions as Cr(III) and Rare Earth Elements. . Information about nanoscale structure of MOF@polymer composites is important for understanding and further improvement of composites properties for water remediation. Thus, in future we are planning to use small-angle scattering and inelastic neutron scattering methods for the understanding structure and dynamics in our nanocomposites at different MOFs amount and modifications.

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Spinel structured high entropy oxides as advanced anode active materials for lithium-ion batteries

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Nowadays, energy storage technologies are in focus of public interest. Amongst the various types of batteries, lithium-ion batteries show the highest interest because of their promising properties such as high energy density, low self-discharge rate and long cycle life. They are used in various devices such as smartphones, laptops, electric vehicles, and even grid-scale energy storage systems. While the cathode material is a key factor in determining the performance of lithium-ion batteries, improving the anode material is also important for several reasons, such as improving the energy density, stability, safety and performance. Recently, it has been demonstrated that high entropy oxides (HEOs) as anode active materials possess promising and unexpected electrochemical properties, such as remarkable reversible capacity and cycle stability due to the high entropy of the system. HEOs are relatively new inorganic materials composed of at least five different cations and have a single-phase structure. Among various types of high entropy oxides, spinel-structured HEOs are the most studied because they ensure the three-dimensional transport of lithium ions leading to high-rate capability. However, in most of the cases it has not been proven that the improved electrochemical properties are the consequence of the entropy stabilization effect. In HEOs the constituent elements need to be homogenously and randomly dispersed over a particular sublattice, resulting in a true solid solution with no evidence of local order or clustering. The determination of the local structure can provide the opportunity for tailoring the properties of HEOs.

We have prepared 3 different high entropy oxides in a two-step process. First, high entropy alloys were synthetized by arc melting of pure elements. Then, the powder particles of the alloys were oxidized in a pure oxygen atmosphere at 1000 °C for several hours. The phase analysis revealed that the CrMnFeCoNi oxide has a single phase spinel structure belonging to the Fd-3m space group with a lattice parameter of a=8.3485(7) Å. The AlCrFeCoNi oxide has a dual phase structure composed of a spinel (a=8.2607(5) Å, 98.7 wt.%) and rocksalt structure (a=4.1969(9) Å, 1.3 wt.%). The CrMnFeCoCu oxide was composed of two phases: the major phase has a spinel

structure (a=8.3584(6) Å, 99.1 wt.%), while the minor phase was based on CuO with a monoclinic structure of C2/c space group (a=4.6877(9) Å, b=3.4942(0) Å, c=5.0701(9) Å, β=100.954 °, 1.3 wt.%).

Further analysis, such as XANES/EXAFS and neutron diffraction/total scattering experiments, are needed to reveal the crystal structure of the synthetized materials. XANES and EXAFS measurements would reveal the oxidation state of the elements and bond lengths, respectively. Neutron diffraction could clarify the oxygen positions and site occupancy. Reverse Monte Carlo simulations of these high entropy oxides based on synchrotron and neutron scattering data would reveal the three-dimensional arrangement of atoms within the materials to understand the structural properties.

Characterization studies using Small Angle Neutron Scattering for Carbon dots and Graphene oxide based Optical Biosensor

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Today, 'Hormonal imbalance' is a significant concern worldwide that lead to various types of complications in human body. For instance, abnormal levels of steroid hormones, such as progesterone are associated with irregular or absent menstrual periods, reproductive disorders, infertility, cardiovascular diseases, and cancer. According to a European Society of Endocrinology report, the present COVID-19 pandemic amply demonstrates how fragile and susceptible many patients with these common disorders are. Nowadays, progesterone is being excessively consumed as oral contraceptives, menopausal hormone therapy and treating infertility, resulting into the deleterious effects due to its endocrine disrupting activity. Furthermore, its presence as a potent endocrine disruptor in surface or environmental waters and various daily use products is another concern. Therefore, monitoring progesterone hormone is vital in environmental samples because of the adverse effects on reproductive health and causing hormonal imbalance. Herein, we have developed optical photoluminescence (PL) method implementing carbon dots (CDs) and graphene oxide (GO) based fluorescence resonance energy transfer (FRET) mechanism for progesterone hormone sensing. In this method, firstly progesterone-specific antibodies are covalently functionalized to CDs whose fluorescence has been quenched (turn off) by adding GO. However, the presence of progesterone increases the distance between donor and acceptor moieties and turns-on the fluorescence by displacing GO off the surface of CDs which in turn substantially reduces the FRET efficiency. Various optimization steps have been performed under control experiments to tune the fluorescence of CDs and the concentration of graphene oxide for maximum efficiency. In conclusion, a linear relationship is achieved in between PL intensity and different progesterone concentrations (10-900 nM) with 13.8 nM detection limits (R^2=0.974). The developed method and used materials demonstrated high selectivity and sensitivity, but we still do not have the solid characterization results to support the modification steps which is very crucial to replicate the method for other biomolecules sensing. Although the structural and morphological analysis (XPS, and FT-IR) revealed the structural and surface functionalities of CDs. However, the structural changes resulting due to antigen-antibody interactions cannot be revealed by UV-Vis/PL spectral data and are still need to be interpreted. Therefore, the small angle neutron scattering (SANS) experiment is essential to provide better understanding of complementary structural

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changes during quenching process, binding of antibody with CDs and binding of progesterone molecules to antibody-functionalized CDs in the presence of GO. Finally, the potential of the developed biosensor can be explored in complex matrices with well-defined phenomenon by neutron scattering techniques.

Polysaccharide-based nanogels for the food sector

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Polysaccharides are the major natural originated components finding extensive investigation and utilization in diverse fields including the nutritional manufacturing sector. Due to their advantageous properties such as safety, stability, biocompatibility, biodegradability and nontoxicity, polysaccharide-based complex systems have a significant potential in the fields of cosmetics, pharmaceutics and food engineering. Microencapsulation of active ingredients such as, flavours, antioxidants, vitamins, lipids, offers greater bioavailability, effectiveness, lower toxicity and more lasting stability than conventional formulations. Understanding the physicochemical properties of these micro- and nanogels and their encapsulating and release properties in different conditions is therefore crucial for their optimization and use in the food sector.

In this research we investigated k-carrageenan-based nanogels obtained as a result of coassembly with a-lactalbumin as macro-ionic crosslinking agent. Such systems are of interest as carriers for bioactive ingredients therefore we compared the microstructure and VD3 encapsulation capacity of nanogels as a function of preparation protocol and various environment conditions (pH, T). Thus, FTIR spectroscopy revealed that nanogels prepared with thermal treatment show stabilization of α-helical and intermolecular β-sheet secondary structures of protein in contrast to nanogels prepared by direct assembly without thermal treatment where the random coil and β-turn structures dominated. The redshift and fluorescence quenching of tryptophan residues upon vitamin D3 addition to nanogels suggest VD3 encapsulation due to the binding with a-lactalbumin and changes in protein structure. Light scattering experiments showed formation of more compact particles along the vitamin D3 encapsulation. The microstructure characteristics of the system and relationship between nanoscopic/mesoscopic structural characteristics and macroscopic functional properties should be further examined by the contrast variation small-angle neutron scattering (CV-SANS) in a wide range of scales.

HDL remodelling by SARS CoV-2 Spike protein

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The SARS-CoV-2 pandemic struck hard and had a vast impact on human health and lifestyle. It was shown that SARS-CoV-2 Spike protein binds cholesterol as well as HDL particles [1], thus changing the HDL's capability to exchange lipids from model membranes [2]. It is likely that this will lead to a misbalance in the lipid metabolism and the regulation of serum lipid and lipoprotein concentrations. Neutron Reflection (NR) experiments performed in our lab showed that the HDL's capacity to deposit and exchange lipids with a model membrane is affected by both the membrane composition and the presence of the S protein (results submitted recently for publication [3]). We found that S protein takes up some lipids in presence of cholesterol (DMPC, PLPC), and others not (POPC). We assume that the selective removal of lipids from HDL influences the function of HDL [3] . We are aiming to verify our assumption by examining the interaction of HDL with different lipid compositions with a model membrane when incubated with S protein.

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Neutron imaging of plant rehydration

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Due to climate change and global warming periods of drought are constantly increasing and becoming a great challenge for plant production. Revealing the mechanisms that enable some plants to survive with limited amount of water available may potentially enhance the drought tolerance of agricultural crops using biotechnological approaches. Haberlea rhodopensis is a unique resurrection plant, colonizing both shady habitats and sun-exposed rock clefts, and known to survive desiccation to an air-dry state and recover fully upon rehydration [1]. Although the rehydration of tissues is a fast process upon rewatering, the mechanism of water uptake, i.e. the contribution of root and leaf tissues is not fully understood.

Neutron beam imaging is a perfect non-invasive method to visualize the water dynamics in real time. Several desiccated plants of H. rhodopensis were placed in aluminium containers supporting their position and serving as water reservoir during the rehydration. Two plant orientations were used to compare water uptake through roots or leaves ("leaves-up/root-down" and "leaves-down/root-up"). During the rehydration period time-lapse neutron radiography images were taken every minute revealing the temporal changes in the spatial distribution of water in the plant with high precision. The obtained results of water dynamics were linked to the physiological parameters of the plant on different stages of rehydration.

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Small-Angle Scattering Estimation of Pore Sizes in Mesoporous Silica

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The properties of porous materials often depend critically on their pore size. It is therefore extremely important to develop reliable methods to determine pore size distributions. For mesoporous materials (with pore size between 2 and 50 nm) a classical method consists in using nitrogen adsorption-desorption experiments. The physical models used to convert these data into pore size distributions generally assume that the adsorption branch is metastable and that desorption is an equilibrium process. Evidence, however, has also been reported that the opposite is sometimes true. The exact nature of adsorption and desorption branches in nitrogen sorption experiments is therefore still unclear.

Ordered mesoporous silica SBA-15 is an ideal material to investigate these questions because its pore size can be estimated independently of N2 adsorption through small-angle scattering. These materials are synthesized through liquid-crystal templating, and their structure is a hexagonal array of cylindrical pores. This leads to scattering patterns with well-defined peaks. In this work, we synthesized a variety of SBA-15 materials and characterized them by nitrogen sorption and small-angle x-ray scattering (SAXS). We systematically compare the pore sizes derived from nitrogen sorption to those estimated from a quantitative analysis of the peak intensities. This notably enables us to test various assumptions concerning the metastability of the nitrogen adsorption and desorption branches.

Here we report specifically on data-analysis aspects concerning the determination of pore sizes from scattering patterns. This is a two-step process consisting (i) in the determination of the peak intensities (ii) followed by their modeling with suitable form factors. We consider various procedures for modeling the background and the peaks (with Gaussian, Lorentizan, and pseudo-Voigt shapes) to extract the peaks intensities, as well as different form factors for modeling the intensities (mono- or poly-dispersed pores, with or without Debye-Waller disorder). The results are critically analyzed in the light also of nitrogen sorption data.

Structure of Lithium Tellurite and Vanadium Lithium Tellurite Glasses by High Energy X-ray and Neutron Diffraction

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The mixed ionic and electronic conduction in glasses has the potential to be used as a cathode in Li-ion batteries. The yLi_2O-$(100$-$y)TeO_2$ and xV_2O_5-$(25$-$x)Li_2O$-$(100$-x-$y)TeO_2$ glasses, (where x= 1,2,3,4, and 5 mol% and y= 20 and 25 mol%) were prepared by melt quenching, and their thermal, vibrational, and structural parameters are being calculated by differential scanning colorimetry, Raman spectroscopy, high-energy X-ray and neutron diffraction, and Reverse Monte Carlo (RMC) simulations. It was found that the glass transition temperature increased steadily with varying contents of 1–5 mol% of V_2O_5 in the lithium tellurite glass network due to an enhancement in the average single bond enthalpy. The combined datasets of X-ray and neutron diffraction were modelled by the RMC technique, and the Te-O, V-O, Li-O, and O-O distributions show their first peaks in the range of: 1.85–1.90 Å, 1.75–1.95 Å, 1.85-2.15 Å, and 2.70–2.80 Å, respectively. The average Te-O coordination number decreases with an increase in Li_2O mol% in lithium tellurite glasses, whereas the V-O coordination number significantly decreases from 5.12 to 3.81 with an addition of V_2O_5 mol% in lithium tellurite glasses. The bond angle distribution of O-Te-O, O-V-O, O-Li-O, and O-O-O linkages has maxima in the following ranges: 86o-89o, 82o-87o, 80o-85o, and 59o, respectively. Moreover, the glass network also revealed the wide range of existence of Te-O and Te-Te distance over short- and medium-range disorders in the first coordination shell.

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Evaluation of the Use of Functional Silica Coatings in Energy-Storage and Energy-Saving Applications

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Sol-gel coating technologies provide advanced properties for many industrial applications. More specifically, silica-based coatings can be well utilized in anti-corrosive, anti-icing, thermal insulation, opacify, and ion/electron separations applications. Among them, energy applications require significant attend regarding the increasing world population and worldwide energy-demand, recently. It has become essential to protect energy as well as to seek ways to meet energy needs. As major heat losses usually arise from buildings, developing insulative surfaces for building applications is therefore crucial in many aspects. For this, having ultra-low thermal conductivity, silica coatings can be an effective way to reduce the thermal conductivity of the surfaces. Apart from thermal insulation, another important energy application is the efficient and safe storage of energy. Having solid-state electrolytes, Li-ion polymer batteries (LiPoBs) have become increasingly important energy storage systems. In this way, severe drawbacks regarding the safety issues of liquid electrolytes and practical applications of Li-ion batteries (LiBs) have been overcome. However, the poor ionic conductivity resulting from the thickness of solid-state electrolytes is still a drawback. Silica coatings with rich oxygen-content can be used as a separator over anode for LiPoBs. Thus, the ion conductivity of the solid-electrolyte will be increased by greatly reducing the polymer thickness. In this study, different silica coating strategies were developed under two parts considering both energy-saving and energy-storage targets.

In the first part, it was aimed to reduce the thermal conductivity of glass surfaces by applying two layers (first hydrophilic and then hydrophobic) of silica coatings via sol-gel method. In the coating solution, Tetraethylorthosilicate(TEOS) and VTMS(vinyltrimethoxysilane) as silica-precursors, ethanol and water as solvents, and 0.01 M HCl acid as catalyst. Coating solutions were applied to glass substrates treated with acetone, ethanol, and distilled water, subsequently. Then, the coated surfaces were dried overnight under ambient. The formation of 3-D nanoporous network on the glass complicates the transfer of heat from the surface. Therefore, the thermal conductivity of the glass was decreased from 0.80 to 0.125 W/mK.

In the second part, methylated silica functionalized with imidazole-based ionic liquid (IL) was synthesized as a thin-film electrolyte for LiPoBs. Prepared electrolyte solution was implemented on the anode surface by casting under ambient condition. TEOS and MTMS (methyltrimethoxysilane) as silica-precursors, IL(1-Butyl-3-

methylimidazolium-bis(trifluoromethylsulfonyl)imide–[BMIM][TFSI]) and Li-salt(Lithium bis(trifluoromethanesulfonyl)imide–[Li][TFSI]) as the electrolyte components were used. The resultant electrolyte with an application thickness of 300-μm having 75% MTMS content and 0.2 mL ionic liquid exhibited 0.08 Li+ transfer number and 4.1x10-1 S/m ion conductivity.

Since the efficiency of the coatings is directly related to the pore hierarchy and the proportion of the pore widths existing in their structure, establishment of a well-defined surface and pore-network are vital criteria to improve the performance in both applications. Therefore, in future studies, performing advanced pore characterization techniques like Small Angle Neutron Scattering (SANS) will be beneficial for us to fully characterize the existing microstructures.

Structure-property-function relationships of complex (soft) materials using Rheo-SANS

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Complex (soft) materials are ubiquitous in nature, with vivid applications ranging from biomaterials, food systems to personal care products. They are usually multicomponent, and have hierarchical structures at different lengthscales by virtue of several interactions. It is essential to understand the exact relationships between processes unfolding at micron/sub-micron structural levels and properties/functionalities of such systems. Neutrons are powerful in accessing structures up to mesoscopic length scales and dynamics over timescale relevant to polymer flow. Rheology is known to effectively perturb the structures at these length scales, in terms of deformation and flow. Therefore, the goal of my project is to explore scattering, and combined Rheo-scattering techniques to investigate the structural, functional, and dynamic behaviour of soft materials.

An interesting class of soft materials are the biomacromolecules Mucins, that are essential soft wet barriers to chemical transport and shear stress in physiological milieu [1]. They are characterized by a charged and highly glycosylated linked peptide backbone and a propensity to form cross-links by a range of mechanisms. The fundamental structural unit is a bottle-brush-like morphology organized into higher level aggregates, or gel-forming networks [1,2]. The aggregation and disaggregation can thus be modulated by perturbation. Scattering studies, and complementary techniques to understand such structures/processes will be discussed, for potential collaboration.

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Development of titania and silver nanohybrids for antibacterial applications

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Titania and silver nanoparticles (TiO2NPs, AgNPs) have attracted considerable attention in recent years for their interesting biological applications [1,2]. Due to their known antibacterial properties, the aim of this work is to develop nanohybrids by direct TiO2NPs-AgNPs conjugation to achieve synergistic antibacterial activity. For this conjugation process, the synthesis in aqueous environment of TiO2NPs-AgNPs nanohybrid platform was carried out by two main steps: 1) a bifunctional alkoxysilane linker, (3-mercaptopropyl)trimethoxysilane (MPTMS), was selected to functionalize commercial TiO2NPs with a size range of 30-50 nm, containing both -SH group (for the Ag attachment) and oxygen atoms (for the TiO2NPs conjugation) in its structure, resulting in TiO2NPs-MPTMS; 2) hydrophilic nanohybrids were prepared by decoration of TiO2NPs surface with AgNPs-3MPS (3MPS = 3-mercapto-1-propanesulfonate) via an in situ synthesis procedure in the presence of sodium borohydride as reducing agent to obtain TiO2NPs-MPTMS-AgNPs-3MPS hybrid systems. In detail, MPTMS silane linker was chosen to increase the biocompatibility and stability of TiO2NPs, whereas 3MPS was used as stabilizing agent for AgNPs in order to prevent the aggregation of nanoparticles during their in situ wet chemical reduction from Ag(I) salt to Ag(0). A spectroscopic method was used to quantify the amount of free -SH groups on titania surface by using rhodamine-based dye. The stability, size, morphology, and chemical composition of the as-synthesized nanohybrids TiO2NPs-MPTMS-AgNPs-3MPS were evaluated by extensive characterizations including UV-Vis, ATR, XPS, ICP-OES, FESEM-EDX, SAXS, DLS and ζ-potential. These results demonstrated the successful silanization of commercial TiO2NPs and subsequent in situ AgNPs-3MPS decoration. Furthermore, in vitro antibacterial studies on nanohybrids were done on E. coli (gram-negative) and S. aureus (gram-positive) bacteria and the results showed the synergistic antibacterial effects of TiO2NPs and AgNPs in the nanohybrid, compared to unconjugated TiO2NPs and control groups.

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Neutron scattering investigations of exotic quantum phases in geometrically frustrated rare-earth chalcohalides and natural minerals

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Low-dimensional quantum spin systems such as geometrically frustrated 2D layered materials, realised in rare-earth chalcohalides (REChX) and natural minerals are of current interest due to their novel properties arising from interplay of quantum fluctuations and reduced dimensionality.

Currently, I aim to design, synthesize, and characterize single crystals of synthetic minerals & YbOX, [X = Cl, Br, F and I]. All these materials show strong spin-orbit coupling due to anisotropic spin interactions. Large single crystals for neutron spectroscopy experiments are grown by employing flux and hydrothermal methods. I intend to use temperature dependent neutron diffraction to determine the nuclear and magnetic structure accompanied by the complex excitation spectrum. Analyses of the diffraction patterns will help me investigate the crystalline structure, local order/disorders and determine structural parameters. Determination of magnetic ground state will be achieved by performing low temperature neutron diffraction. Later, I plan on using inelastic neutron scattering to investigate the spin Hamiltonian of the magnetic systems. The spatial directional dependent spin excitations measurements will provide the understanding of the origin of the novel magnetic properties. It is planned to investigate structure-magnetism interactions and understand the intrinsic interplay of electronic, spin, and orbital degrees of freedom arising from a complex balance of electron-electron correlations, spin-orbit coupling, and crystal-field splitting in tuning the magnetic ground states. Ultimately, magnetic phase diagram in the temperature and magnetic field plane will be determined. Through my Ph.D., I aspire to gain fundamental knowledge of geometrical-spin-frustrations, competing interactions, and anisotropic-exchanges in these systems.

Thiol functionalized mesoporous silica sorbent for selective sorption of aqueous Ag(I)

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In the last decades, the concentration of silver compounds in surface waters has increased significantly due to their wide range of technological applications. The high concentration of silver compounds may cause toxic effects on the human body: irritate eye, skin, breathe, digestive system and altering blood cells. Due to its unique, advantageous properties silver is an invaluable substance in many industrial applications. The recovery of silver ions from aqueous solutions containing other metal compounds is an important task of environmental technology. One solution is the development of selective sorbents by the appropriate design of surface functionality. For this purpose, a mesoporous, mercaptopropyl functionalized silica xerogel was prepared by the sol-gel method. It is characterized by low voltage scanning electron microscopy (LV-SEM), N2-sorption porosimetry, small angle neutron scattering (SANS), EDX analysis and X-ray photoelectron spectroscopy (XPS). Its aqueous phase Zeta potential was investigated as a function of pH. The xerogel has excellent selectivity for binding Ag(I) in the simultaneous presence of seven other metal ions at pH = 5.0, with a very high sorption capacity of 238 mg g-1. The sorption equilibrium establishes extremely fast, after 15 min which is a major advantage regarding the practical functionality of the present xerogel sorbents. The quantitative recovery of Ag(I) and the regeneration of the sorbent is possible using 10.0 mM $Na_2S_2O_3$ solution. The mechanism of binding can be explained by the formation of Ag(I) clusters on the surface, which is usual for silver ions in the presence of thiol groups. This explains the excellent selectivity of the sorbent for silver ions.

Acknowledgements

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Structure and properties of Metal Matrix Composites reinforced by inclusion of nanoparticles: a new feedstock material for Metal Additive Manufacturing

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Aluminium alloys are one of the most widely applied materials in industrial engineerings, such as in aeronautical, aerospace, marine and automotive industries, mainly due to their high strength-to-weight ratio. Although several casting techniques are used for the production of aluminium alloys, new technological challenges arise when the complexity of novel geometries requires the implementation of advanced additive manufacturing techniques, such as directed energy deposition (DED) 3D-printing. However, most of these materials cannot be used directly in DED due to the enormous thermal shock that occurred during printing, causing various reactions in the molten metal and resulting in a lower performing printed sample.

The inclusion of high-hardness ceramic nanoparticles in aluminium results in the enhancement of its mechanical properties, showing values four times higher than those typical of aluminium alloys. The latter, combined with the reduction of material content through the use of additive manufacturing, will be the basis for the development of lighter structures, and consequently, will reduce the fuel consumption and gas emissions of traditional means of mobility. For this reason, Aluminium Metal Matrix Composites (AMMCs) are the most attractive option due to their stiffness and damage resistance as well as their high thermal and wear resistance.

The focus of this research is on the development of such kind of Metal Matrix Composites as new feedstock material for various technologies like Metal Additive Manufacturing (MAM), more specifically the called Wire Arc Additive Manufacturing (WAAM) technology. We have used nanofillers of TiC and TiB2 as reinforced nanoparticles in different concentrations for the study of their mechanical property. The dispersion of TiC and TiB2 (size 50 nm) nanoceramics in the AMMCs has a key role to achieve the promised mechanical properties of these materials. Among the wide range of available characterization tools, neutron scattering techniques, especially small-angle neutron scattering (SANS), stand out in acquiring advanced knowledge about the structure of these AMMCs. Evaluation of the dispersion and homogeneity of nanofiller embedded in the pure Al metallic matrix, as well as the

formation of micro- or nano-segregations, or intermetallic phases are derived from SANS data. Detailed information about the structure and properties of the materials helps us to describe the synthesis process and improve the technology procedure fully.

Methotrexate loaded mixed thiols functionalised gold nanoparticles: Synthesis and characterisation

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Nanomaterials find several applications in different fields, one of the most promising is nanomedicine. Here, gold nanoparticles (AuNPs) have been functionalised with two hydrophilic thiols – 3-mercapto-1-propanesulfonate (3MPS) and 2-(diethylamino)ethanethiol (DEA) – following a single-phase wet chemical reduction in water where sodium borohydride is used as the reducing agent. This allowed to obtain ultra-fine nanoparticles with a controlled diameter (<10 nm), suitable for their use as drug delivery probes. This AuNPs system was loaded with methotrexate (MTX), an anti-folate drug. The loading, calculated as drug molar encapsulation efficiency, was optimised and found to be ca. 70%. Loaded and free AuNPs have been extensively characterised, both spectroscopically and from a morpho-structural point of view: UV-visible and FT-IR spectroscopy were performed, together with Dynamic Light Scattering and ζ-potential measurements to evaluate size, stability, and functionalisation of the nanoparticles, before and after the interaction with the drug. Synchrotron Radiation Grazing Incidence Small and Wide Angle X-Ray Scattering (GISAXS, GIWAXS), as well as SAXS, X-Ray Photoelectron Spectroscopy (XPS), and 1D, 2D NMR experiments were carried out to study more closely the structure of the nanosystems, i.e. the ratio between the two thiols and their spatial arrangement on the surface of the AuNP, as well as the interaction with MTX. Field Emission Scanning Electron Microscopy (FE-SEM) and Transmission Electron Microscopy (TEM) were performed as means to determine the morpho-structural features of the nano-systems on both pristine AuNPs and the drug loaded nanoconjugate (AuNPs@MTX).

Facile Synthesis and Performance Evaluation of Functionalized Silica Aerogel-Like Materials in Diverse Applications

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Silica aerogels are sol-gel derived nanoporous materials with unique properties such as very low density, high porosity, high surface area, and low thermal conductivity. They are traditionally synthesized via sol-gel method followed by supercritical drying. By following proper strategies such as silylation and co-gelation, on the other hand, it is entirely possible to synthesize ambient pressure dried aerogels with a morphology indistinguishable from that of supercritical dried analogs.

In this study, various organically functionalized silica aerogels were prepared under ambient conditions by incorporating functional alkoxysilanes such as trimethylchlorosilane (TMCS), methyltrimethoxysilane (MTMS), methyltriethoxysilane (MTES), vinyltrimethoxysilane (VTMS), 3-Glycidyloxypropyltrimethoxysilane (GLYMO), 3-methacryloxypropyltrimethoxysilane (MEMO) into sol-gel reactions both as silylating agents and as a co-precursors to be used in combination with classically used tetraalkoxysilanes (tetraethylorthosilicate, TEOS). Depending on the gelation behavior and steric effects of the selected silanes, each sample exhibited completely different morphologies and physicochemical properties. According to characterization results, the obtained density, porosity, apparent surface area, and thermal conductivity varied between 0.092-0.71g/cm3, 68-96%, 223-964m2/g, 42-47mW/mK, respectively. They were also thermally stable up to 600°C. As MTMS, MTES, VTMS possess hydrophobic organic radicals in their structure, they also improved the hydrophobic behavior (θ=120-172°)[1,2]. Owing to these properties, they seem as ideal candidates to be employed as thermal insulators and as adsorbents. Their adsorptive performance was tested by batch sorption studies to remove oils and organic solvents from wastewater (capacity up to 12.5g/g). Results show that they are promising materials both in energy-saving applications and in environmental clean-up practices[3].

Nevertheless, the complete definition of the microstructures of these complex nanosystems is a must to further improve their performances in such applications. The efficiency of the materials is directly related to the pore hierarchy and the proportion of the micro/mesopores to macropores existing in their structure. The presence of micro/mesopores imparts high surface areas and pore volumes that offer high adsorption potential and complicate the heat transport phenomenon which yields even lower thermal conductivity. The macropores can ease the way to achieve

functional active sites in the material during sorption applications and offers ease of use by maintaining monolithic form in insulation applications. Therefore, in future studies, it is important to employ some advanced characterization techniques like Small Angle X-Ray Scattering (SAXS) and Small Angle Neutron Scattering (SANS) to fully characterize their intricate nanostructures for a better design of the material.

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Metal Organic Framework derived Titanium dioxide/Carbon structures covered with Molybdenum disulfide nanosheets for solid-state symmetrical supercapacitors

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With the advancement of the electrification of devices, the demand for efficient energy storage devices has also risen exponentially. Various EU programs such as Battery 2030+ and ERA net+ energy is running to address and improve the current state of renewable energy storage devices. Compared to batteries, supercapacitors can charge/discharge very quickly (i.e., have high power density), which is the forefront requirement for electric vehicles and various other devices. Basically, supercapacitors store charge through the fast adsorption of electrolytes instead of slow intercalation/deintercalation of ions like batteries. However, instead of having high power density, the energy density of the supercapacitor is much lower than batteries which restricts its application in various energy devices. To push its limits, researchers are exploring new materials with a hybrid design that can show fast surface redox reactions with a high surface area to adsorb electrolyte ions. MOF is a class of crystalline porous material with high surface area and tunable pore size distribution. Herein, we have produced metal-organic framework (MOF) derived TiO_2/carbon structures whose surface is further modified with MoS_2 nanosheets to get the synergistic effect of redox-active MoS_2/TiO_2 and the high surface area carbon structure. Upon pyrolysis of MOF, TiO_2/Carbon has been produced at three different pyrolysis temperatures (600, 800, 1000 ℃). TiO_2/carbon produced at 1000 ℃ attained a high surface area and so has been chosen to modify with MoS_2 nanosheets (TiO_2/carbon/MoS_2). When used as electrode material, the tri-composite delivered the specific capacitance of 470 F/g with 70% capacitance retention even after 4000 charging-discharging cycles. The tri-composite outperformed the bulk MoS_2 (288 F/g) and bare TiO_2/carbon (382 F/g). The symmetrical supercapacitor has also been assembled with PVA gel electrolyte, which delivered 49.2 Wh/kg of energy density at the power density of 1.3 kW/kg. The higher performance of the tri-composite can be attributed to the porous and conductive core (TiO_2/carbon) with a redox-active MoS_2 structure at the surface. However, when the calculation of surface and diffusion-controlled charge contribution was done, the tri-composite

showed a higher fraction of diffusion-controlled charge storage mechanism compared to bulk MoS2 and TiO2/carbon which is still unclear and very important to resolve to design the efficient electrode material for high-performance supercapacitor. Small angle neutron scattering (SANS) is an advanced toolbox for in-situ characterization, which can allow studying the structures at the molecular level. With the use of SANS, it is expected to resolve the role of material nanostructure for surface and diffusion-controlled charge transportation.

Micellization of Triton X100 and Brij 58 in aqueous solution of Poloxamer 188 at the range of temperature T = (273.15–323.15) K

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The micellization of the binary mixture of surfactants Triton X100 – Brij 58 in an aqueous solution with Poloxamer 188 was investigated. The critical micelle concentrations of pure surfactants and their binary mixtures in aqueous solutions with Poloxamer 188 were determined by an invasive spectrofluorimetric method, using pyrene as probe molecule. Measurements were done with different molar ratios (α) of surfactants in their binary mixtures (α = 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9), in the temperature range of T = (273.15–323.15) K, with 5 K increments. All solutions of surfactants were prepared using pyrene-saturated aqueous solutions containing 0.01 w/w of Poloxamer 188. Regular Solution Theory was used for calculation of the surfactants' molar fractions in the binary mixed micelle, the coefficients of the activity of surfactants in the micellar pseudophase and the interaction coefficient (β). The results show that the mean values of the interaction coefficient in the temperature interval T = (273.15–313.15) K are β 0. In the presence of Poloxamer 188, the thermodynamic destabilization of the binary mixed micelle Triton X100 – Brij 58 increases with increasing temperature. Above a temperature of 313.15 K, the binary mixed micelle Triton X100 – Brij 58 in an aqueous solution with 0.01 w/w Poloxamer 188 is thermodynamically less stable than the hypothetical ideal binary mixed micelle Triton X100 – Brij 58 with the same molar content, implying the presence of antagonistic interactions between different types of micellar building units. With increasing temperature, the conformation of Poloxamer 188 changes, and since hydrogen bonds probably bind this polymer to the terminal OH groups of polyoxyethylene chains of Brij 58 surfactants, the domains of binary mixed micelles where Poloxamer 188 is bound become more diffuse, making it challenging to form hydrogen bonds between Brij 58 and Triton X100. The size, shape and aggregation number of the examined mixed micelles should be investigated by small angle neutron scattering, in order to further explain the obtained results.
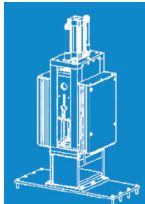
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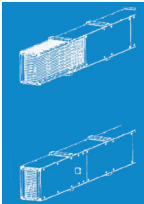
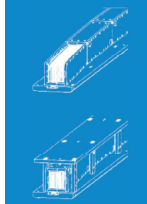## OPTICS AND INSTRUMENTS FOR NEUTRON RESEARCH
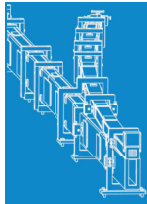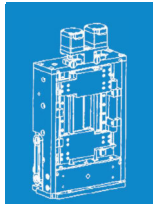
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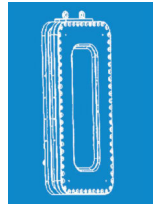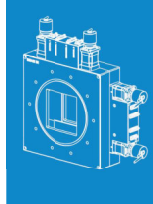
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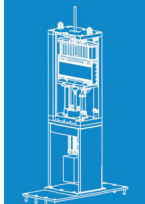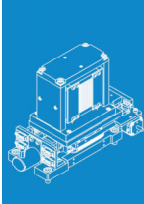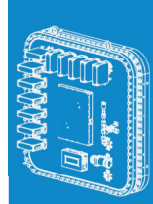
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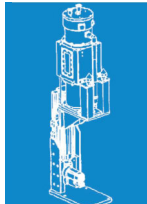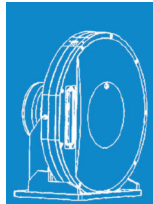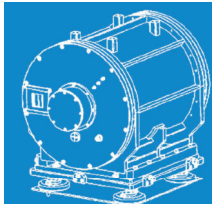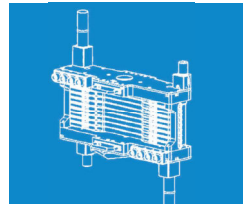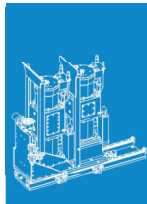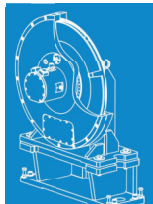

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