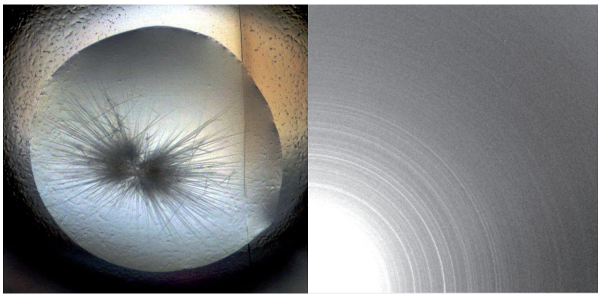
d'Aix-Marseille I et II, UMR 6098, ESIL Case 925, 13288 Marseille, FRANCE, [c]Department of Biology, Section of Genetics, Cell Biology and Development, University of Patras, GRECE E-mail: yves.watier@esrf.fr

Measuring on a single urchin like crystal at room temperature used to be unrealistic due to the absence of a reliable available setup. In air, the samples were dehydrated, and measuring a small amount of protein powder sample in a capillary is also difficult. Also, for fragile samples, the centrifuging of microcrystals in a capillary may be fatal for diffraction. We have seen that the design of the humidity control device available at the ESRF allowed us to collect high quality powder diffraction pattern on a PX beamline on a single urchin like crystal. The possibility to preserve small protein powder samples at room temperature without losing diffraction properties is a new step for protein powder diffraction. This allows several possibilities, first, to screen very easily and routinely crystalline precipitates in order to determine their diffraction quality, on any PX beamline; second, to get reliable intensities at low to medium resolution, suitable to solve a structure by molecular replacement; third, to build and refine a preliminary model using these extracted intensities or via a Rietveld refinement.

Data acquisition and preliminary analysis on the non structural protein 3 macro domain of the Mayaro virus will be presented, following previous studies[1] done on a larger amount of sample.



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Keywords: protein, virus, powder

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Solid state reactivity and solvent mobility in crystals of a cubane polymer

Elena Forcén-Vázquez,[a] Javier Campo,[a] Larry R. Falvello,[a,b] Fernando Palacio,[a] Milagros Tomás,[c] [a]*Aragón Materials Science Institute (ICMA), CSIC, University of Zaragoza (Spain).* [b]*Department of Inorganic Chemistry, University of Zaragoza (Spain).* [c]*Instituto de Síntesis Química y Catálisis Homogénea (ISQCH), CSIC, Universidad de Zaragoza (Spain).* E-mail: elenafv@unizar.es

Solids that accommodate small molecules in structural voids are of interest for applications as diverse as catalysis or gas storage and purification. Most systems studied to date in this area are 2- and 3-D polymers with frameworks that maintain open voids. In contrast, 1-D polymers give structures without rigidity in three dimensions, so removal of small molecules that might be present leads to the collapse of the crystal structure. Moreover, solids formed by 1-D polymers usually have a lesser amount of void space to house solvent and other small molecules.

To understand the storage of small molecules in solids, it is necessary to characterize the geometries of the pores or voids involved, and also their chemical nature. The study of solid-state reactions provides insight into both the chemical and physical natures of the substances and processes involved. [2]

Quadruply deprotonated citrate forms transition metal complexes with a topologically cubic M_4O_4 core and 12 peripheral oxygen atoms that can bind transition metals or form non-covalent interactions with interstitial small molecules. The cubane units are structural building blocks for polymers of varying dimensionality; their hydrophilic periphery confers exceptional qualities on the solids, including structural variability, water solubility and solid-state chemical reactivity. [4,5]

We have previously reported an unprecedented solid-state crosslinking in which a 1-D polymer of cobalt citrate cubanes fuses under mild conditions to produce a 2D polymer. [5] This quasi-topotactic process demonstrates the structural flexibility and solid-state reactivity typical of these compounds and suggests the possibility of studying the reaction mechanisms of the solid state transformations.

We present here a new family of non-porous 1-D and 2-D manganese citrate cubane polymers whose flexible structural natures permit the reversible desorption of water in SCSC transformations at room temperature. Both species have mobile interstitial water molecules. There are no clearly defined voids or channels for water mobility, so the structural framework must yield in order for water egress and reuptake to take place. In addition, if the dehydrated derivative of the 1-D polymer is exposed to a methanol atmosphere, one molecule of methanol substitutes an aqua ligand on a non-cubane manganese(II) unit present in the structure, through a possible associative mechanism. This reversible transformation preserves the crystallinity of the polymer. There is evidence that one more molecule of methanol enters the polymer through a physisorption process.

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Keywords: crystalline, polymer, chemisorption

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Flexibility found in silica-like metal-organic frameworks

I.E. Collings,[a] A.L. Goodwin,[a] A.L. Thompson,[a] M. Dove,[b] L. Rimmer,[b] [a]*Department of Inorganic Chemistry, University of Oxford, Oxford (UK.)* [b]*Department of Earth Sciences, University of Cambridge, Cambridge (UK).* E-mail: ines.collings@chem.ox.ac.uk

Throughout the last decade, the study of metal-organic frameworks (MOFs) has remained one of the most topical fields in solid-state chemistry, due to the extensive and unique range of structural and functional properties they exhibit [1].

In recent years, studies on MOFs have revealed unusual mechanical properties, such as negative thermal expansion in MOF-5 [2], and amorphisation of zeolitic imidazolate frameworks at high temperature [3].

This poster will be focused on mechanical properties of some MOFs whose structures resemble those of silicate frameworks. For example, cadmium and mercury imidazolates (with a topological resemblance to α-cristobalite), have shown unusual thermal expansion coefficients as determined from variable temperature x-ray diffraction. Molecular dynamics is used to fully characterise ligand movement, and understand the extend to which framework flexibility determines mechanical behaviour.

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Keywords: metal-organic frameworks, flexibility, molecular dynamics

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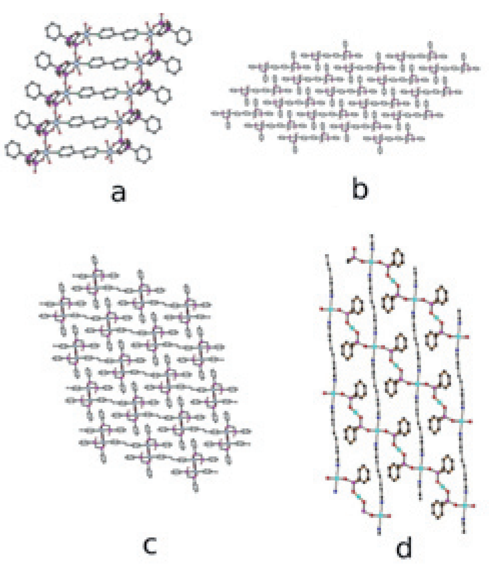
Polymer dimensionality lead by supramolecular interactions in cobalt system

Annalisa Guerri,[a] Ferdinando Costantino,[b] Andrea Ienco,[c] Stefano Midollini,[c] [a]*Departament of Chemistry, University of Florence (Italy).* [b]*Department of Chemistry, University of Perugia (Italy),*[c]*CNR-ICCOM, (Italy).* E-mail: annalisa.guerri@unifi.it

Supramolecular forces are involved in the building of the three-dimensional structure in the solid state [1]. Few years ago, it has shown that in metal inorganic organic hybrids the metal coordination environment can be forced by non-covalent interactions. The close packing of 2,2'-bipyridine ligands was responsible for the uncommon trigonal prismatic coordination of Mn(II). The *normal* octahedral Mn(II) coordination was restored when the intercalation of the bipyridine was prevented using substituted bipyridines [2].



We report here a series of hybrid inorganic organic polymers $[Co(pcp)(4,4'\text{-bipy})_{0.5}2H_2O]_n$ (**1**), $[Co(pcp)(bpye)_{0.5}H_2O]_n$ (**2**), $[Co(pcp)(bpyet)_{0.5}H_2O]_n$ (**3**) and $[Co(pcp)(bpyet)_{0.5}]_n$ (**4**) where pcp = P,P'-diphenyl-methylene-diphosphinato, 4,4'-bipy = 4,4'-bipyridine, bpye =1,2-bis(4-pyridyl)ethane, bpyet =1,2-bis(4-pyridyl)ethene).

In Figure 1 are shown 1D ribbon in **1** (a) and the packing view of **1** (b) and of **2** (c) parallel to the ribbon, finally the packing view of **4** (d).

In the 1D polymer of **1**, the ribbons of $[Co_2(pcp)_2bipy]$ are connected by hydrogen bonds (Figure 1a and 1b) while **2** and **3** are built by 2D plane formed by the same kind of 1D ribbons that are covalent bonded by water molecules. The view of the packing parallel to the ribbons shows the same type of arrangement of the phenyl rings of pcp and of the bi-pyridines. For **4**, a totally different 2D network was found with 1D columns of [Co(bpyet)] connected by square planar $Co(pcp)_2$ units (Figure 1d). The role of the bipyridine and its lenght in the formation of the network will be discussed.

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Keywords: metallorganic polymers, diphosphinate, supramolecular chemistry

MS38.P04

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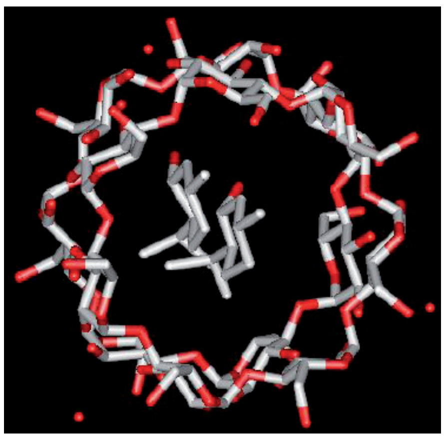
Inclusion Complex of β-Cyclodextrin and l-Menthol

Yasushi Kai,[a] Masaki Yamamoto,[a] Nobuko Kanehisa,[b] Tsuyoshi Inoue[b] [a]*Department of Environmental and Biological Chemistry, Fukui University of Technology, Fukui.* [b]Department of Applied Chemistry, Graduate School of Engineering, Osaka University, Suita, *(Japan).* E-mail: kai@fukui-ut.ac.jp

Cyclodextrins are the cyclic oligosaccharides composed of the glucopyranoses linked by α-1,4-glucoside bond. There are three common cyclodextrins with 6, 7 or 8 D-glucopyranosyl residues, α-, β- and γ-cyclodextrin respectively. Cyclodextrin has the truncated corn shape with hydrophilic OH substituents outside and hydrophobic cavity inside the molecule. The secondary alcohols (2-OH and 3-OH) make bigger and primary alcohols (6-OH) make smaller rims of truncated corn structure. Therefore, cyclodextrin can include the water insoluble molecules inside its cavity. This property is applied for various reagents to solubilize in water, stabilize from oxidation, recognize a molecule, or transfer a drug. Menthol has local anesthetic and counterirritant qualities, and it is widely used to relieve minor throat irritation. Menthol is included in many products such as toothpaste, chewing gum, cigarette etc. To keep the function of menthol, cyclodextrins are often included in these products. In order to understand the molecular function of cyclodextrin in these products, the crystal structure of β-cyclodextrin and l-menthol was determined by the X-ray diffraction method.

The inclusion complex of β-cyclodextrin and l-menthol crystallized in monoclinic space group $P2_1$ with cell parameters of a=15.2487(7), b=32.487(1), c=15.3835(5)Å, β=101.712(1)°, V=7462.2(5)Å^3, Z=2, F.W.=2582.52. Two β-cyclodextrins and two l-menthols are in the asymmetric unit. The X-ray diffraction data were collected on Rigaku RAPID diffractometer. The crystals include many crystalline waters and were very unstable in air. Thus, the data measurements were carried out at -150 deg. using flash cooling method. In total, 33,583 independent reflections were observed (R_{int}=0.0293). Crystal structure was solved by direct method (SHELXD) and refined by full-matrix least-squares on F^2 (SHELXL-97). The present R1 is 0.1100.



The crystal structure is shown in the figure. Two The detailed molecular interactions between β-cyclodextrin and l-menthol will be discussed.