

out in order to check the conditions for total polymer removal. Thermo-gravimetric analysis and nitrogen adsorption isotherms completed the study.

Keywords: small-angle diffraction, *in-situ* experiments, temperature

### P11.09.70

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#### Crystal structures and mesomorphism of metal-salen complexes with 4-substituted long alkoxy chains

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Liquid crystals with transition-metal core groups, called as metallomesogens, have attracted increasing attention because of the possibility of combining their physico-chemical properties of the metal with those of organic framework. Since the metallomesogens are achieved through changes of molecular conformation, shape, and structure, their physico-chemical properties can be turned by the choice of metal ions, substituents, and position of substituents on core moieties. Schiff base ligands provide a wide range of ways to modify liquid crystal compounds. However, to date there has been no systematic investigation concerning molecular assemblies and metallomesogens of metal-salen (salen=*N,N'*-ethylenebis(salicylideneiminato)) complexes substituted by two long alkoxy chains at the 4-positions though the metal-salen complexes with 5-substituted alkoxy or alkyl chains usually show smectic A (S<sub>A</sub>) mesophases at higher temperature. Recently, we prepared three series of the VO(IV), Ni(II), and Cu(II) salen complexes with two long alkoxy chains introduced at the 4-positions, [M((4-C<sub>n</sub>H<sub>2n+1</sub>O)<sub>2</sub>salen)] (M=VO(IV), Ni(II), Cu(II), *n*=3-20) and found an unprecedented bilayer mesophase having symmetry Pa2<sub>1</sub> in the layers for the VO(IV) salen complexes with *n*=16-20.<sup>1</sup> On the other hand, the Ni(II) and Cu(II) salen complexes with longer alkoxy chains showed an unusual metallomesogen of a lamello-columnar (Col<sub>l</sub>) mesophase.<sup>2,3</sup> There have been few reports on the metallomesogen of the Col<sub>l</sub> mesophase. These liquid crystalline phase structures have been affiliated with the molecular assemblies in the crystalline states.

1) Y. Abe, *et al.*, *Inorg. Chim. Acta*, **359**, 3934 (2006). 2) Y. Abe, *et al.*, *Inorg. Chim. Acta*, **359**, 3157 (2006). 3) Y. Abe, *et al.*, *Mol. Cryst. Liq. Cryst.*, **466**, 129 (2007).

Keywords: metal-salen complexes, 4-substituted long alkoxy chains, metallomesogen

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#### Profile analysis of chemically prepared emeraldine-salt and emeraldine-base forms of polyaniline

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ES-PANI form powder was prepared by chemical oxydation using aniline previously distilled added to HCl aqueous solution. ammonium peroxydisulphate was dissolved in HCl aqueous solution and then added to the above solution slowly at room temperature.

ES-PANI was obtained and kept in NH<sub>4</sub>OH yielding EB-PANI. XRD patterns were obtained with a Rigaku RotaFlex, CuK $\alpha$ , 50 kV, 100 mA, graphite monochromator. Data collected from 2 $\theta$  = 5 - 60°, step 0.02° and 5 sec. Profile Matching fit was performed using FULLPROF. Initial cell parameters from Evain *et al.*, 2002. Anisotropic size broadening was modeled. Refined structural parameter and agreement factors for ES-PANI are *a*=5.61214; *b*=17.78087; *c*=23.04183;  $\alpha$ =83.25719;  $\beta$ =84.71980;  $\gamma$ =88.08441; *V*=2273.126Å<sup>3</sup>; *R*<sub>p</sub>=1.08; *R*<sub>w</sub>*p*=1.38;  $\chi^2$ =1.40 and for EB-PANI are *a*=5.71920; *b*=17.77932; *c*=23.52416Å;  $\alpha$ =84.13010;  $\beta$ =85.22524;  $\gamma$ =87.30433; *V*=2369.428Å<sup>3</sup>; *R*<sub>p</sub>=1.77; *R*<sub>w</sub>*p*=1.39;  $\chi^2$ =1.14. Obtained values are in agreement with literature, except cell parameter *b*, which was almost doubled. EB-PANI form average crystallite size (25.42Å) obtained from the refinement confirmed its lowest percentage of crystallinity compared with doped ES-PANI form (36.45Å).

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#### Coordination polymers towards applications: Chirality by design

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Crystal engineering involves the design and subsequent synthesis of crystals with the specific aim of developing new crystalline materials. The main focus of this field lies in the ability to predictably synthesize supramolecular structures from smaller, well designed building blocks.[1] The formation of self-assembled coordination polymers has attracted much interest in recent years. Here crystal engineering takes a vital role in the construction of porous frameworks that may show functionality in various applications. There are two main components to any coordination polymer, namely the connectors (metal ions) and the linkers (organic ligands). Because there are a number of tunable parameters (for example: metal centre coordination environments, ligand size and flexibility, ligand functional group type and position, ligand charge, choice of counter ions and solvents) it is possible to generate a diverse number of framework architectures.[2] In this study we have selected dipyriddy type linkers and a number of divalent transition metals [Cd(II), Cu(II), Co(II) and Ni(II)] as building blocks. The organic linkers have been carefully chosen to coordinate with the metals to form chiral crystals. This is most effectively done by selecting chiral moieties, removing the possibility of creating centrosymmetry in the crystal structure. We have also used flexible achiral moieties to create chiral crystals. This is significant, as only about 17% of structures in the CSD (Nov 2007) are chiral, with only a small fraction of these arising from achiral molecules. Selected results will be presented.

[1] L. Brammer, *Chem. Soc. Rev.*, 2004, 33, 476. and citations therein.

[2] S. Kitagawa, R. Kitaura, S. Noro, *Angew. Chem. Int. Ed.*, 2004, 43, 2334. and citations therein.

Keywords: coordination polymers, crystal engineering, chirality