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Synthesis And Characterization Of New Materials Like Perovskite $[NH_3-(CH_2)_n-NH_3]$ $ZnCl_4$ avec n=8 et 10

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Abstract

During this work, we have prepared new semiconductors $[NH_3-(CH_2)_n-NH_3]ZnCl_4$ (n=8 ; 10) (Short notation $2C_nZnCl_4$) which are self assembled organic-inorganic hybrid materials by solution chemistry technique using the HCl in evaporating phase. The grown crystals have been studied by infrared absorption spectroscopy, X-ray diffraction, scanning electron microscopy; the electrical properties of the hybrid perovskites synthesised were also studied by UV-visible measurements.

Introduction:

The organic–inorganic pervskites are on of the most extensively studied crystalline families of nanohybrids which offers an important opportunity to combine useful properties from two chemical realms, organic and inorganic compounds, within a single molecular scale composite. Especially, the design and generation of the organic–inorganic self-organized quantum well structure represent an approach to synthesis that offers new horizons in the context of synthetic chemistry and its possible impact on nanotechnology [**K. Kikuchi**].

Organic compounds offer a number of useful properties, including structural diversity, plastic mechanical, and ease of processing. Inorganic materials have a distinct set of advantages, including good electric mobility, tuneable band gap property, magnetic property, and thermal stability **[D.B. Mitzi(1996),].** Organic-inorganic hybrids offer an important opportunity to combine useful properties from these two chemical realms within a single molecular scale composite **[David B. Mitzi(2001)].**

They are characterized by strong intralayer covalent ionic bonding in an inorganic frame and weak interlayer interaction such as van der Waals force between organic molecules [**Young-Duk Huh**].

The organic–inorganic perovskites are consisting of inorganic anions, each comprised of an extended network of corner-sharing metal halide octahedra, alternating with a variety of different organic cations [**K. Kikuchi**].This class of materials, generally expressed as $(R\text{-}NH_3)_2MX_4$ or $(NH_3\text{-}R\text{-}NH_3)MX_4$ (R = organic group, M = Cu, Zn, Sn, Pb and X = Cl, Br, I), consists of an extended network of corner-sharing metal halide octahedrons, alternating with a bilayer or monolayer organic moiety. $R\text{-}NH_3^+$ is a primary amine cation [**Z.Y. Cheng**].

This kind of materials can be synthesized by divers methods as solution chemistry addition [**Ying-Ying Zheng**], by solid state interaction into inert atmosphere [**Etienne Worthama**], with spray pyrolyse [**Z.Y. Cheng,**]... A number of techniques have been described for depositing hybrid films, including multiple-source thermal evaporation, single-source thermal ablation, Langmuir-Blodgett, two-step dip processing, spin coating, and stamping. The solution-based processes are particularly attractive because they enable the quick and inexpensive deposition of the hybrids on a diverse array of substrates [**David B. Mitzi(2001)**].

Generally, the single crystals and powder samples of organic-inorganic perovskite hybrids can be obtained in solution by evaporation of the solvent or cooling the saturated solution slowly [**A. Vecht**].

I- Sample preparation:

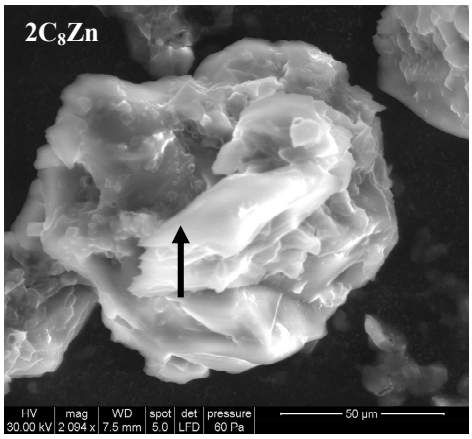
First of all, instead of using HCl in aqueous solution, we are going to synthesize gaseous HCl: Concentrated H_2SO_4 is poured; drop with drop; on solid NaCl, gaseous HCl frees is dissolved in solution. Next, we prepare a powder finely crushed of diamine $NH_2\text{-}(CH_2)_n\text{-}NH_2$ (with n = 8, 10) solid, at the end to ameliorate its solubility. 0.5 mg of every one is weighted and dissolved in a small quantity of distilled water. The solution is made under regular and weak agitation during at least half an hour. We point out that the solubility of the diamine is slow in distilled water, but it improves by agitating. After complete dissolution of the diamine, we use a reduced quantity of HCl, only to saturate solution with H^+ ions. The diamino-hydrochloride is formed.

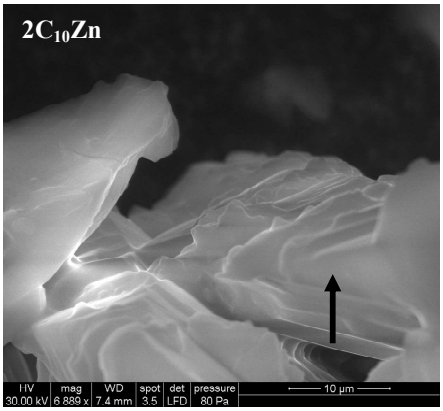
After stage of protonation, we add stœchiometric quantities of $ZnCl_2$ salt in a minimum of distilled water. The acquired solution is clear, homogeneous and uncoloured. It is covered with some paper and left at room temperature up to complete disappearing of solvent.

II- Results and discussion

1- Scanning electron microscopy

Micrographs recorded for the synthesized composite materials, show a foliated structure of particles, result of an alternation of the organic and inorganic leaves, and the growth of crystals is perfectly orientated to procreate a molecular bidimensionnelle structure orientated also.





2- XR-Diffraction:

Acquired preliminary results revelled that the new crystals of NH_3-$(CH_2)_nNH_3]ZnCl_4$ (n = 8; 10) belongs to the P-1 space group, Z =2 with a=6.998(1), b=10.769(3), c=11.020(4), α = 83.26(3), β =82.12(2) and γ =73.37(2) for $2C_8ZnCl_4$ and a=7.294(1), b=10.058(9), c=12.812(1), α = 90.89(2), β =101.21(2) and γ =92.40(3) for the $2C_{10}ZnCl_4$.

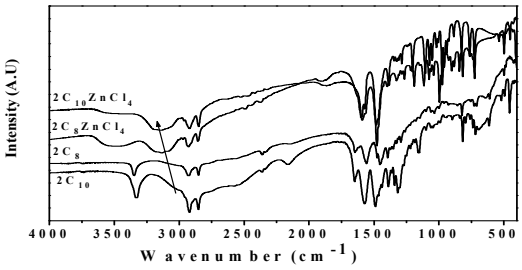
We point out that both materials have similar characteristics; the weak difference is due to the organic chain length [**Liling Guo**]

3- FTIR spectroscopy:

The infrared spectroscopy is an attractive method to characterize alkyl chain conformation in these hybrids because of its effectiveness in determining low concentrations of disorder (gauche conformers) in an other wise ordered systems. It is well known that the vibrational modes in infrared spectra of ordered alkylchains may be described as coupled oscillators, witch produce band progressions. Structural applications of these modes have been reported for the characterisation of lipid bilayers, gels an triglyserols [**N.V. Venkataraman**].

Pellets were prepared by dispersing the powder of the composite material in KCl. Then they were compacted under a pressure of 10 kbar, before being put under 100°C, during one day. Samples were analysed by infrared spectroscopy (IRTF) into transmission mode. The used apparatus is of model Jasco FT / IR – 4100 A type, spectra were recorded between 4000 and 400 cm^{-1} with a resolution about 4 cm^{-1}.

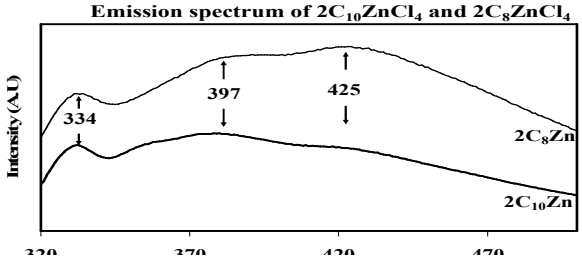
The studies based on some compared compounds revelled that the number of progression bands is induced by the number of CH_2 groups in tans registry and that the structure of the inorganic slabs in these compounds are invariant with respect to the value of n, so the difference between the FTIR spectra of $2C_8ZnCl_4$ and the $2C_{10}ZnCl_4$ is presumably depend on the length of organic chain.



The FTIR spectra of $2C_8ZnCl_4$ and the $2C_{10}ZnCl_4$ revelled that the stretching vibration peak of NH_3^+ head groups shift to higher wave numbers; this can be attributed to the chemical environment change of NH_3^+ groups after they self assemble into the layered perovskite structure.
The location and their assignments of internal modes of the organic cations, respectively for the $2C_8ZnCl_4$ and $2C_{10}ZnCl_4$, observed in the infrared spectra, is based on the comparison with the well documented spectra of homologous compounds [**Young-Duk Huh, N.V. Venkataraman, Ze-Long Xiao, Abdellah KAIBA (2004), T. Dammak** and **KHECHOUBI El Mostafa(2001)**].

- Band situated around 3500 cm^{-1} is attributed to the stretching mode of OH raised respectively for the $2C_8ZnCl_4$ and $2C_{10}ZnCl_4$ at 3483 $cm^{-1}$and 3556 cm^{-1}.
- Bands between 2500 cm^{-1} and 3500 cm^{-1} are owed to stretching of modes NH^{3+} group and absorption in this region is characterized by a fine structure in the region of low frequencies, they are groups of peaks solved well between 1200 cm^{-1} and 1650 cm^{-1}.
- The occurrence of bands (2928; 2857) cm^{-1}, (2923; 2856) cm^{-1} corresponding to the symmetric and asymmetric stretching mode of CH_2.
- Bands at (1477; 1600) $cm^{-1}$and (1482; 1590) cm^{-1} are attributed to the asymmetric and symmetric deformation in the plane of CH_2 group.
- Bands observed at 1120 cm^{-1} and 1112cm^{-1} is attributed to the roking and twisting mode of NH^{3+} group. The 1105 cm^{-1} frequency is assigned to the stretching mode of C-N.
- The bands located at 497 cm^{-1} and 446 $cm^{-1}$are attributed to the deformation of NCC angle in «gauche » conformation.

4- UV Visible:



The room temperature UV-Vis emission spectra showed three similar clear exciton peaks, for both of materials; this is in perfect agreement with papers which says that crystal feature is practically independent of the length of the alkylammonium chains; and are not observed for the crystal in the absence of metallic cation and not observed for the metal halide either [**Ze-Long Xiao**] So this strong and sharp emission is characteristic of the perovskit-type hybrid based on two-dimensional structure.

On the other hand; the emission spectra of this compounds is independent of the excitation wavelength and the optical properties of the systems can be adjusted by replacement halogen within the inorganic layer.

As a result; we can suggests the potential use of the perovskite film for UV detection and as non-linear optical materials [**Ying-Ying Zheng**].

Conclusion

During this study, we have synthesize two composite materials based on zinc $2C_nZ_nCl_4$, (n = 8; 10) in solution, using gaseous HCl in protonation stage.

Micrographs acquired by electronic microscope, show that the synthesized composite materials have a foliated, bidimensionnelle and orientated structure, which is in agreement with RDX results.

By spectroscopy IR, we could put in an obvious place the most part of the characteristic bands revealed by the different internal movements of the radical $[NH_3\text{-}(CH_2)_n\text{-}NH_3]^{2+}$, within the composite $2C_nZnCl_4$.

The optical behaviour of these materials is due to the inorganic party within the composite and it is not influenced by the length of the organic chain.

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Appendix: At the end of the paper

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