

Construction of coordination polymers based on mixed-ligand synthetic strategy

Huda K. Alkhalaf

Department of Chemistry - University of Houston

1-Abstract

At this stage, the rational construction of structurally-defined CPs is quite successful and a variety of typical examples for such crystalline materials with attractive network architectures and potential applications have been reported. In this regard, their possible applications arising from the remarkable physico-chemical properties of CPs are widely recognized in gas adsorption, molecular/ionic separation, optics, electricity, magnetism, chirality, catalysis, and drug delivery, etc. As a rule, the optimal implementation for such applications requires good phase purity, framework stability, and in some cases the available porosity of the targeted materials. While their absolute structural control still remains a long-term goal and a worthwhile endeavor.

Much work has been devoted to the synthesis, structural characterization, and properties of CPs. In this process, the accumulation of sufficient experimental data allows chemists to proceed beyond the random studies and to derive some useful laws of assembly. Towards this end, several effective synthetic strategies such as ‘node-and-spacer’ and ‘secondary building units (SBUs)’ have been successfully established and developed, based on the notion that molecular precursors (metal ions and organic ligands) can be conveniently conceptualized as objects such as points, lines, polygons, and polyhedra, with CPs as periodic and complementary assemblies of these geometric motifs. Nevertheless, there are still many challenges in practice to perfectly project and regulate the specific crystal packing of such materials, because structural control will be readily thwarted by the intricate and noncovalent nature of the secondary interactions such as H-bonding, aromatic stacking, and van der Waals force, as well as some external physical or chemical factors including counterion, template, temperature, pressure, solvent, and pH value, etc. .

To date, it is well known that multi-topic bridging ligands with two or more nitrogen, sulfur, phosphorus, and/or oxygen-involving functional groups can show different binding abilities to metal ions and thus can be applied as effective tectons for constructing diverse coordination networks . However, in the scope of mixed-ligand CPs, ligands such as disulfoxide , dithioether ,diphosphine , and hybrid P,N-type compounds have seldom been used in practice, probably owing to the lack of competent counterparts as the co-ligands. In contrast, intense research activity towards the rational design and construction of mixed-ligand CPs has shown that the bipyridine, polycarboxyl , and amino compounds represent the most reliable and typical building blocks which can be jointly applied to synthesize a wide range of desired coordination networks. A choice of such connectors in coordination assembly can be rationalized based on the following considerations. On the one hand, the neutral bipyridine ligands normally bind to the metal ions as the rod-like bidentate tectons. On the other hand, polycarboxyl compounds may take the anionic or protonated form to provide various coordination modes upon binding to

metals and anionic form can obviate small counteranion incorporation, resulting in neutral frameworks with enhanced porosity. While amino acids are well known as excellent building blocks for hydrogen bonded network. Thus, the underlying structures of CPs obtained from a given bipyridine spacer can usually be ascribed to known geometrical networks, at least with hindsight, while assembly of a polycarboxyl bridging ligand with the metal ion under different conditions will lead to considerable structural complexity and diversity of CPs. As a result, by combining the advantages of two such types of ligands, the so-called mixed-ligand synthetic strategy can be rationally proposed. In the broad domain of CP study, coordination frameworks based on mixed ligands have been heavily developed. However, no specific structural prediction and design scheme is available at this stage.

SO from the mixed-ligand synthetic point of view, I have proposed to synthesis Monometallic and Heterometallic coordination polymers .

-According to the different compositions of mixed-ligand CP systems, they can be roughly classified into three categories:

- 1-Acid–acid mixed-ligand CPs.
 - 2- Base–base mixed-ligand CPs.
 - 3- Acid–base mixed-ligand CPs
- (the concept of acid or base in this review only indicates the Lewis acid/base ligands).

* Acid–base mixed-ligand CPs

The acid–base system is the most important and flourishing branch of mixed-ligand CPs. Naturally, acid and base ligands are perfect partners that can compensate charge balance, coordination deficiency, repulsive vacuum, and weakly interaction all at once. In this context, it is valuable to propose the rational synthetic strategy to regulate the network structures of mixed-ligand CPs by ligand design or selection, considering the aspects of spacer effect, positional isomeric effect, and substituent effect of the organic building blocks.

Keywords : Coordination polymers - Crystal engineering - Mixed-ligand synthetic strategy - heterometallic system - Lanthanide

2- Materials

The construction of molecular architecture depends on the combination of several factors, such as the solvent system, template, temperature, counter ion, ratio of ligands to metal ions, coordination geometry of central metals and organic ligands.

Choice of the working conditions

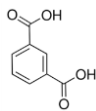
The most important factors during the construction of coordination polymers are the coordination preferences of the metal center and the ligand functionality.

A)-Metal ions used

The transition metals such as Cu, Co, Ni etc. are capable of adopting various geometries like tetrahedral, octahedral, square planar, square pyramidal etc and thus 1D, 2D and 3D architectures are expected with interesting topologies. The aim is to develop compounds that exhibit properties suitable for present and future electrical and optical applications, though they may be used in magnetism and catalysis as well.

Lanthanide coordination polymers have attracted much attention over the past decades because of their intriguing topological structures, their possible high framework stability, and their interesting luminescent and magnetic properties which have potential applications in biology and optical, electronic, and magnetic functional materials. Although the varied coordination requirements of the lanthanide ions make the rational design and assembly of the lanthanide coordination polymers quite difficult, the inherently high and variable coordination numbers and flexible coordination environment of lanthanide ions provide unique opportunities for synthesizing coordination polymers with various structures and properties have been obtained polymers with unusual architectures and topologies. By rational selection of organic ligand, a variety of lanthanide.

The current increasing interest in designing and synthesizing heterometallic coordination polymers (HCPs) has been significantly provoked not only by their impressive structural diversity but also by the potential applications as functional materials in magnetism, molecular adsorption, photoluminescence, catalysis and chemical separation. From the synthetic point of view, the assembly of extended structures with high-dimensionality lanthanide- transition metal heterometallic coordination frameworks is a formidable task, because there are several complicated factors: (i) the choice of organic ligands; (ii) the control of the second ligand and (iii) the competitive reaction between lanthanide and transition metal ions, etc. Despite of these factors, the selective strategy for main organic ligand containing appropriate coordination sites is crucial to construct target compounds. Therefore, the system composed of ligands containing N, O-donor atoms can easily form heterometallic coordination polymers. Transition metal and lanthanide ions have different affinities for N or O donors. The lanthanide ions prefer to O-donors, whilst transition metal ions have a tendency to both N- and O-donors. Therefore, ligands containing both N- and O-donors, such as imidazolecarboxylic acid, pyridinecarboxylic acid, triazolecarboxylic acid and pyrazinecarboxylic acid have the ability to give rise to the formation of HCPs.\

B) Ligands used**1) Isophthalic acid (MW - 166.13 g/mol)**

We selected isophthalic acid as a ligand based on the following considerations: (a) it has multiple coordination sites that may generate structures of higher dimensions; (b) it has two carboxyl groups which may be completely or only partly deprotonated and thus result in acidity-dependent coordination modes; (c) it can act not only as a hydrogen-bond acceptor but also as a hydrogen-bond donor because of the presence of the protonated carboxyl group; (d) it can form short bridges via one carboxylato end or long bridges via the benzene ring. We believe that coordination functionality of isophthalate could lead to interesting structures.

The dicarboxylates, as important organic anions, have attracted intense interest from chemists due to its versatile coordination modes in the assembly of polymeric structures

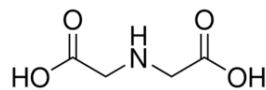
2) Bidentate 4,4'-bipyridine ligand (Mw156.18 g/mol)

The studies of bipyridine based coordination polymers started with the complexation of 4,4'-bipyridine ligands with a large variety of metal centres changing also the experimental conditions. This ligand was widely and is still extensively studied, giving a lot of new materials (more than 300 articles relate coordination polymers built up with 4,4'-bipyridine among other building blocks). The 4,4'-bipyridine ligand can bridge a lot of metal centres in a numerous number of structural motifs.

Research on bridging bidentate bipyridyl based complexes is also of interest. Indeed the introduction of linkers between the two N-donors groups allows a larger diversity, *i.e.* a larger potentiality in the resulting coordination polymer topologies. The bridging part of the ligands confers to the ligand some specificity: linearity, determined shape, rigidity/flexibility, specific functionality, increasing the ligand adaptability and the number of possible interactions. Due to the attraction towards these systems, bipyridine based coordination polymers are widely reviewed.

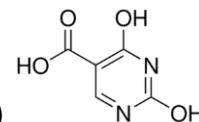


3) Iminodiacetic acid (M_w 133.10 g/mol)



iminodiacetic acid is a ligand with multiple coordination sites containing both O and N atoms, and a ligand with both rigidity and flexibility, which makes it a potentially good candidate for constructing stable high-dimensional metal–organic coordination polymers.

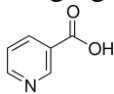
4) 2,4-Dihydroxypyrimidine-5-carboxylic acid (M_w 156.10 g/mol)



pyrimidine and its derivatives, in spite of their simplicity, are very versatile systems. Their ability for giving specific H-bonding patterns as a key step in the storage and transmission of genetic information is a well-known process. Pyrimidine-carboxylate adopts diverse coordination mode coupling with metal atoms and pyrimidine ring can exchange magnetic interactions. Model calculation and analyses of available experimental data indicated that the higher metal atom deviations from the mean plane of the pyrimidine ring, the weaker antiferromagnetic exchange.

By the way, 4-(2-pyridyl)pyrimidine (pprd) is an attractive N-donor ligand with a bidentate site for chelating and an exo N-donorsite for bridging.

5) Isonicotinic acid (MW 123.11 g/mol)



In order to create a poly-dimension in searching for functional materials that have desired structures and properties led to the development of a rich field of chemistry owing to the potential applications of these materials in catalysis, separation, gas storage and molecular recognition. Research efforts in metal-organic networks have so far been mostly focused on coordination polymers with neutral organic rod-like rigid group ligands containing either N-donor or O-donor ones. While the nicotinic acid (nic) ligand is just a combination of these nature, it's consider multifunctional organic linker molecules as it can act as a multidentate ligand with versatile binding and coordination modes. Furthermore, it has recently shown interesting properties.

3-. Experimental

All chemicals were obtained from Sigma-Aldrich, and used without purification. The syntheses were carried out in home-built teflon-lined cylindrical stainless steel pressure bombs with maximum capacity of 120 ml.

First : Synthesis Monometallic Coordination Polymers

The following reactions has been done using various metal salts with mixed-ligands:



Ligand L1- Isophthalic acid

FW- 166.13 g/mol

Ligand L2- 4,4''dipyridyl

FW-156.19 g/mol

S.NO	COMPOUNDS	RATIO	MOLAR RATIO	WEIGHT RATIO IN GRAMS	TEMPERATURE	TIME
1	M:L1:L2	1:1:1	0.001:0.001:0.001	0.2416:0.1661:0.156	140 °C	3DAYS



2-Metal M – (CF₃SO₃)₂Cu

FW- 361.68 g/mol

Ligand L1- Isophthalic acid

FW- 166.13 g/mol

Ligand L2- 4,4''dipyridyl

FW-156.19 g/mol

S.NO	COMPOUNDS	RATIO	MOLAR RATIO	WEIGHT RATIO IN GRAMS	TEMPERATURE	TIME
1	M:L1:L2	1:1:1	0.001:0.001:0.001	0.361:0.166:0.156	140 °C	3 DAYS
2	M:L1:L2	2:1:1	0.002:0.001:0.001	0.722:0.166:0.156	140 °C	3 DAYS
3	M:L1:L2	1:1:2	0.001:0.001:0.002	0.361:0.166:0.312	140 °C	3 DAYS

3) Metal M – Nd(NO₃)₃ · 6H₂O

FW- 438.35 g/mol

Ligand L1- Isophthalic acid

FW- 166.13 g/mol

Ligand L2- 4,4''dipyridyl

FW-156.19 g/mol

S.NO	COMPOUNDS	RATIO	MOLAR RATIO	WEIGHT RATIO IN GRAMS	TEMPERATURE	TIME
1	M:L1:L2	1:1:1	0.001:0.001:0.001	0.438:0.1661:0.156		

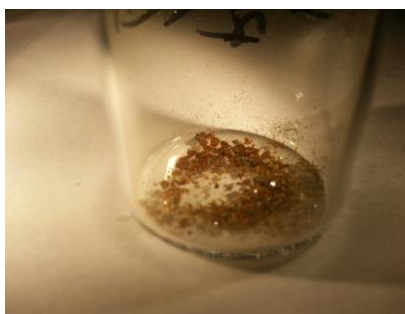


4) Metal M – $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ FW- 241.60 g/mol

Ligand L1- Iminodiacetic acid FW- 133.10 g/mol

Ligand L2- Isonicotinic acid FW-123.11 g/mol

S.NO	COMPOUNDS	RATIO	MOLAR RATIO	WEIGHT RATIO IN GRAMS	TEMPERATURE	TIME
1	M:L1:L2	1:1:1	0.001:0.001:0.001	0.241:0.133:0.123		



Second : Synthesis Heterometallic coordination polymers (HCPs)

1) Metal 1 M_1 – ZnCl_2 FW- 136.31 g/mol

Metal 2 M_2 = $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ FW- 451.36 g/mol

Ligand L1- 2,4-Dihydroxypyrimidine-5-carboxylic acid FW- 156.10 g/mol

Ligand L2- Tartaric acid FW- 150.09 g/mol

S.NO	COMPOUNDS	RATIO	MOLAR RATIO	WEIGHT RATIO IN GRAMS	TEMPERATURE	TIME
1	M1:M2:L1:L2	1:1:1:1	0.001:0.001:0.001:0.001	0.136:0.451:0.156 0.150	140 °C	3DAYS



2)Metal 1 M₁ – ZnCl₂

FW- 136.31 g\mol

Metal 2 M₂= Nd(NO₃)₃ · 6H₂O

FW- 438.34g\mol

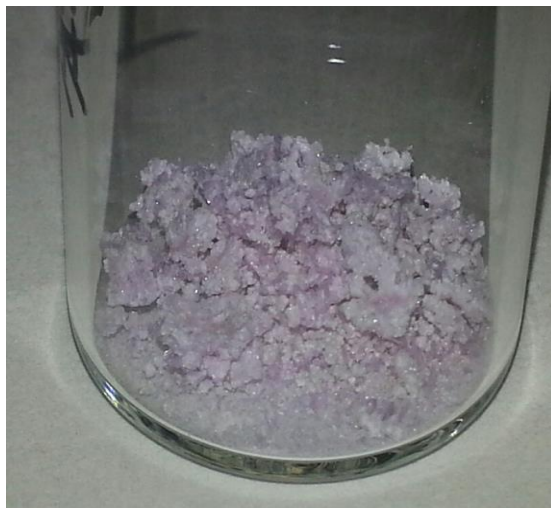
Ligand L1- 2,4-Dihydroxypyrimidine-5-carboxylic acid

FW- 156.10 g\mol

Ligand L2- Tartaric acid

FW- 150.09 g\mol

S.NO	COMPOUNDS	RATIO	MOLAR RATIO	WEIGHT RATIO IN GRAMS	TEMPERATURE	TIME
1	M1:M2:L1:L2	1:1:1:1	0.001:0.001:0.001:0.001	0.136:0.438:0.156 0.150	140 °C	3DAYS



- 3) Metal 1 $M_1 - ZnCl_2$ FW- 136.31 g/mol
 Metal 2 $M_2 = Tb(NO_3)_3 \cdot 5H_2O$ FW- 435.06 g/mol
 Ligand L1- 2,4-Dihydroxypyrimidine-5-carboxylic acid FW- 156.10 g/mol
 Ligand L2- Tartaric acid FW- 150.09 g/mol

S.NO	COMPOUNDS	RATIO	MOLAR RATIO	WEIGHT RATIO IN GRAMS	TEMPERATURE	TIME
1	M1:M2:L1:L2	1:1:1:1	0.001:0.001:0.001:0.001	0.136:0.435:0.156 0.150	140 °C	3DAYS

- 4) Metal 1 $M_1 - Cu(NO_3)_2 \cdot 3H_2O$ FW- 241.60 g/mol
 Metal 2 $M_2 = Gd(NO_3)_3 \cdot 6H_2O$ FW- 451.36 g/mol
 Ligand L1- 2,4-Dihydroxypyrimidine-5-carboxylic acid FW- 156.10 g/mol
 Ligand L2- Tartaric acid FW- 150.09 g/mol

S.NO	COMPOUNDS	RATIO	MOLAR RATIO	WEIGHT RATIO IN GRAMS	TEMPERATURE	TIME
1	M1:M2:L1:L2	1:1:1:1	0.001:0.001:0.001:0.001	0.241:0.451:0.156 0.150	140 °C	3DAYS

- 5) Metal 1 $M_1 - Cu(NO_3)_2 \cdot 3H_2O$ FW- 241.60 g/mol
 Metal 2 $M_2 = Nd(NO_3)_3 \cdot 6H_2O$ FW- 438.34 g/mol
 Ligand L1- 2,4-Dihydroxypyrimidine-5-carboxylic acid FW- 156.10 g/mol
 Ligand L2- Tartaric acid FW- 150.09 g/mol

S.NO	COMPOUNDS	RATIO	MOLAR RATIO	WEIGHT RATIO IN GRAMS	TEMPERATURE	TIME
1	M1:M2:L1:L2	1:1:1:1	0.001:0.001:0.001:0.001	0.241:0.438:0.156 0.150	140 °C	3DAYS

- 6) Metal 1 $M_1 - Cu(NO_3)_2 \cdot 3H_2O$ FW- 241.60 g/mol
 Metal 2 $M_2 = Tb(NO_3)_3 \cdot 5H_2O$ FW- 435.06 g/mol
 Ligand L1- 2,4-Dihydroxypyrimidine-5-carboxylic acid FW- 156.10 g/mol
 Ligand L2- Tartaric acid FW- 150.09 g/mol

S.NO	COMPOUNDS	RATIO	MOLAR RATIO	WEIGHT RATIO IN GRAMS	TEMPERATURE	TIME
1	M1:M2:L1:L2	1:1:1:1	0.001:0.001:0.001:0.001	0.241:0.435:0.156 0.150	140 °C	3DAYS

(4) Discussion

The samples will be sent to analyze the metal-ligand and packing geometry through single crystal X-ray structural investigations.

The above reactions and pictures prove that coordination polymers based on mixed-ligand synthetic strategy can be effectively produced and an accurate prediction of the overall crystal structure is usually not possible due to non-covalent interactions and their poor directionality involving the ligands and non-covalently bound anions, cations as well as solvent (guest) molecules. Obviously, this work presented here demonstrates that utilizing the mixed ligand to coordinate with the transition metals, Lanthanide, and heterometallic system may lead to even more novel materials with some promising structural features and properties.

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بناء مركبات تناسقية باستراتيجية الليجاند المختلطة

في هذه المرحلة ، يعد البناء العقلاني المعتمد على أسس علمية و تصنيع شبكات المركبات التناسقية نموذجاً ناجحاً تماماً في للحصول على بلورات ذات بنية شبكية لتطبيقات و استعمالات محتملة. وفي هذا الصدد ، فإن تطبيقاتها المحتملة التي تنشأ عن الخصائص الفيزيائية الكيميائية الرائعة للبلورات معروفة على نطاق واسع في امتصاص الغاز ، والفصل الجزيئي / الأيوني ، والبصريات ، والكهرباء ، والمغناطيسية ، والأدوية ، والحفز ، وتوصيل الأدوية ، إلخ.

وكقاعدة ، يتطلب التنفيذ الأمثل لمثل هذه التطبيقات نقاء الطور الجيد واستقرار الإطار وفي بعض الحالات المسامية المتاحة للمواد المستهدفة. في حين لا تزال السيطرة على التركيب و الحصول على تركيب محدد لهذه المركبات هدفاً بعيداً المدى ومسعى جديراً بالاهتمام.

تمت كريس الكثير من الابحاث لتصنيع ، و التفسير الهيكلي ، ودراسة خصائص المركبات التناسقية ، فإن تراكم البيانات التجريبية و نتائج التجارب العملية يسمح للكيميائيين بالانتقال الى حد أبعد من الدراسات العشوائية و استخلاص بعض القوانيم لبناء و تركيب المركبات التناسقية.

لتحقيق هذه الغاية ، تموضع وتطوير العديد من الاستراتيجيات الاصطناعية الفعالة مثل "العقدة والمباعدة" و "وحدات البناء ومعدلك ، لا تزال هنا كالعديد من التحديات في الممارسة العملية لتحضير المركبات التناسقية على نحو مثالي بتركيب محدد بسبب امكانية التداخل و حدوث تفاعلات ثانوية بواسطة الروابط التساهمية و الهيدروجينية

قوة der Waal ، بالإضافة إلى بعض العوامل الفيزيائية أو الكيميائية الخارجية درجة الحرارة ، والضغط ، والمذيب ، وقيمة الأسال هيدروجيني ، إلخ

حتى الآن ، مثبت علمياً أنا ليجاند متعددة الاسنان بوجود اثنين أو أكثر من المجموعات الوظيفية للنيتروجين والكبريت والفوسفور و / أو الأكسجين يمكن أن تظهر قدرات ربط مختلفة على أيونات المعادن وبالتالي يمكن تطبيقها لتحضير المركبات التناسقية. و نتيجة لذلك في هذا البحث ، من خلال الجمع بين مزايا نوعين من هذه الليجاند ، يمكن اقتراح استراتيجية يطلق عليها "الليجاند المختلطة" لتحضير عدد من المركبات التناسقية.

تثبت التفاعلات والصور المذكورة في هذا البحث أن البوليمرات التناسقية المستندة إلى إستراتيجية الليجاند المختلطة يمكن إنتاجها بشكل فعال و لكن يبقى التنبؤ الدقيق للهيكل البلوري الكلي غير ممكن في الغالب بسبب التفاعلات غير التساهمية الذي يشتمل على بروابط وأخرى غير تساهمية الأنيونات ملزمة ، والكاتيونات ، فضلاً عن جزيئات المذيب.

من الواضح أن هذا العمل المعروض هنا يوضح أن استخدام الليجاند المختلط للتنسيق معاً لمعادن الانتقالية ، اللانثانيد ، و مركبات ذات فلزات متعددة قد يؤدي تكوين مركبات تناسقية بلورية والخصائص الهيكلية الواعدة..