



Fig. 1

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**Synthesis and Characterization of Polyoxometalate to Functional Groups (MIL-101) by Phosphotungstic Acid**

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A new strategy has been developed for the direct encapsulation of polyoxometalates (POMs) in to MIL-101(Cr). The addition of phosphotungstic acid (PW<sub>12</sub>) to the synthesis mixture of MIL-101 yields the direct encapsulation of chromium-containing POMs inside the metal organic framework (MOF) structure, with a good distribution over the MIL-101 crystals. It was characterized by FT-IR spectroscopy and XRD, in contrast to the poor or absent activity of the catalysts prepared via the impregnation of the polyoxometalate in MIL-101, where the strong interaction between POM and support deteriorates the catalytic performance. The medium-sized cavities of MIL-101 are occupied by POM units bigger than their pentagonal windows when this one-pot approach is followed, and no leaching is observed.

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**Synthesis and characterization of nanoflower-like Bi<sub>2</sub>S<sub>3</sub> via simple microwave approach**

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The present investigation reports the novel method to synthesis of nanoflower-like Bi<sub>2</sub>S<sub>3</sub> using microwave irradiation and its physicochemical characterization. The Bi<sub>2</sub>S<sub>3</sub> nanostructure powder was prepared directly by a microwave route using Bi(NO<sub>3</sub>)<sub>3</sub>·5H<sub>2</sub>O and thioglycolic acid (TGA) as starting materials in ethyleneglycol at 750W for 5min. Bismuth sulfide is a photoconductive, direct bandgap layer semiconductor with numerous potential applications including photovoltaics, IR spectroscopy and thermoelectrics. It also belongs to a family of solid-state materials with applications in thermo-electric cooling technologies based on the Peltier effect. The influence of several parameters on the morphology and particle sizes of Bi<sub>2</sub>S<sub>3</sub> nanostructures has been investigated. X-ray diffraction measurements (XRD) showed the formation of high pure Bi<sub>2</sub>S<sub>3</sub> with Orthorhombic phase (JCPDS; 17-0320). The average particle size obtained from the Debye–Scherrer equation was 9.7 nm. SEM analysis showed that the Bi:S mole ratio, solvent, microwave power and heating time play a key role in Bi<sub>2</sub>S<sub>3</sub> size and morphology. It was determined that the best Bi:S mole ratio and solvent for preparation of Bi<sub>2</sub>S<sub>3</sub> nanostructure are 1:5 and ethylene glycol, respectively. Also the optimum power and heating time for this experiment was 750W and 5 min, respectively. The quality of the product was analyzed by the FT-IR spectroscopy and it was found that there is not any organic compound on the Bi<sub>2</sub>S<sub>3</sub> surface and synthesized product has high purity. Optical properties of product were characterized with ultraviolet–visible (UV–vis) and photoluminescence (PL) spectroscopy. The calculated bandgap from PL was 2.24eV that has a large blue shift in comparison with bulk sample with 1.3 eV. This different value is due to quantum size effect.

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**Synthesis and Characterization of a Series of d<sup>10</sup> Metal Complexes with a New Flexible Pyridyl-carboxylic acid Ligand**

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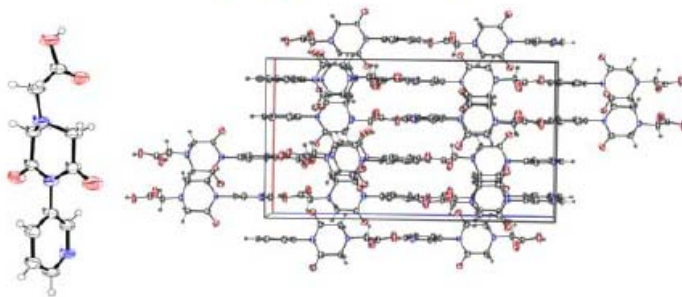
Self-assembly processes involving metal centers and designed organic ligands have attracted considerable attention in the field of crystal engineering and coordination chemistry. Basically, the design of ligands with different coordination sites together with coordination preferences of the metal centers can produce the new materials with unique electronic, magnetic, catalytic and optical properties [1–4]. In this study, we have designed and synthesized the flexible multifunctional ligand of 2-(3,5-dioxo-4-(3,5,9-triazine-3-yl)piperazin-1-yl)acetic acid, as a pyridyl-carboxylic acid (Hpyca) ligand (I). The crystal and molecular structure of I and its OH...N hydrogen bond interaction are illustrated in Figure 1. Also the d<sup>10</sup> metal complexes of designated ligand have been investigated. These complexes have been characterized by FT-IR and NMR spectra.





# پنجمین کنفرانس علمی ایران

۱۳-۱۵ شهریور ۱۳۸۰ دانشگاه بوعلی سینا



(A)

Fig.1

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### Synthesis and determination of $[Ni(phen)_2(Ph_2phen)](PF_6)_2$ complex

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Polypyridyl complexes are compounds in which a polypyridyl ligands such as 2,2':6',2''-terpyridine, 2,3,5,6-tetrakis(2-pyridyl)pyrazine, 1,10-phenanthroline-5,6-dione, 2,2'-dipyridylamine, 1,10-phenanthroline or 2,2'-bipyridine, coordinates to a metal center. These complexes have peculiar electrochemical, spectroelectrochemical, magnetic, medicinal, and optical properties. Some polypyridyl complexes exhibit a strong absorption band in the visible region, which is called metal-to-ligand charge transfer (MLCT) or ligand-to-metal charge transfer (LMCT). The properties of the complexes can be tuned easily by introducing substituents, for example, electron donating groups (EDG), electron withdrawing groups (EWG), and  $\pi$ -conjugating groups, to pyridyl moiety [1-5]. In this study we report synthesized  $[Ni(phen)_2(Ph_2phen)](PF_6)_2$  complex, where  $Ph_2phen$  is 4,7-diphenyl-1,10-phenanthroline and phen is 1,10-phenanthroline, and characterized by electrochemical method and spectral techniques FT-IR, UV-vis and <sup>1</sup>H-NMR. Electronic spectral shows two absorption bands at 505 and 787 nm originate from the spin-allowed, <sup>3</sup>A<sub>2g</sub> → <sup>3</sup>T<sub>1g(F)</sub> and <sup>3</sup>A<sub>2g</sub> → <sup>3</sup>T<sub>2g</sub> transitions, whereas the third spin-allowed transition, <sup>3</sup>A<sub>2g</sub> → <sup>3</sup>T<sub>1g(P)</sub>, could not be distinguished due to the strong ligand specific and charge-transfer metal to ligand transitions. The absorption bands seen in the UV ( $\lambda = 220-360$  nm) region are assigned to ligand-centered  $\pi \rightarrow \pi^*$  transitions. IR spectroscopy of complex show the typical pyridinic C=C and C=N stretching vibrations give rise to strong absorption at around 1450-1480 cm<sup>-1</sup> and 1600-1630 cm<sup>-1</sup>, respectively. The IR spectrum of the complex also exhibit the strong absorption band around 840 cm<sup>-1</sup> which is assigned to  $\nu(P-F)$  and demonstrate the existence of PF<sub>6</sub><sup>-</sup> as a counter ion. <sup>1</sup>H-NMR spectra of complex with broad resonance indicate paramagnetic behavior. Magnetic susceptibility data revealed effective magnetic moment 3.1 BM at room temperature.

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### Synthesis and characterization Copper (II) complex with polypyridyl ligands

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Transition metal complexes with potential biological activity are the focus of extensive investigation. Copper(II) is the most studied metal ion among all the transition metal ions. Copper(II) complexes are known to play a significant role either in naturally occurring biological systems or as pharmacological agents. A large number of mixed ligand copper(II) complexes have been shown to exhibit super oxide dismutase activity. This activity depends on the Cu(II)/Cu(I) redox process, which is related to flexibility of the geometric transformation around the metal centres. Complexation with copper enhances the biological activity of a wide variety of organic ligands. Copper(II) complexes of polypyridyl ligands may be considered as models for SOD [1-5]. In the present, we report new copper(II) complex