A New Potassium-Based Coordination Polymer with Hydrogen Bonding and Zigzag Metallophilic Interactions

Zarin Moghadam¹, Kamran Akhbari^{1,*}, Jonathan White², and Anukorn Phuruangrat³

¹School of Chemistry, College of Science, University of Tehran, Tehran, Iran.

Tel.: +98 21 61113734; fax: +98 21 66495291. E-mail address:

akhbari.k@khayam.ut.ac.ir (K. Akhbari)

²School of Chemistry and Bio21 Institute, The University of Melbourne, VIC 3010, Australia

³Department of Materials Science and Technology, Faculty of Science, Prince of Songkla University, Hat Yai, Songkhla, Thailand.

Abstract

A new potassium-based coordination polymer $[K(\mu_7\text{-HTar})]_n$ (**1**), (H_2Tar = (+)-tartaric acid), has been synthesized and structurally characterized by single crystal X-ray crystallography. It has a three-dimensional structure with zigzag $\text{K}\cdots\text{K}$ metallophilic interactions. Also widespread hydrogen bonding network in three-dimensional structure of **1** was observed. In order to evaluation the effects of concentration, ultrasonic irradiation and type of the solvents on morphology and thermal behavior of **1**, some experiments were designed and six samples of **1** were synthesized under different conditions. These samples were characterized by IR spectroscopy, thermo gravimetric and differential thermal analyses (TG-DTA), X-ray powder diffraction (XRD) and Scanning Electron Microscopy (SEM).

Keywords: Coordination polymer; Potassium; metallophilic; Nanostructure; Ultrasonic; Solvent type.

This is the author manuscript accepted for publication and has undergone full peer review but has not been through the copyediting, typesetting, pagination and proofreading process, which may lead to differences between this version and the Version of Record. Please cite this article as doi: [10.1002/aoc.4613](https://doi.org/10.1002/aoc.4613)

1. Introduction.

Self-assembly of supramolecular polymers is a powerful tool for producing functional materials that combine several properties and may respond to external conditions.¹⁻⁷ Recently, the construction of coordination supramolecular or coordination polymers has emerged as a powerful and versatile strategy for engineering metallo-polymers.⁸⁻¹² A wide branch of supramolecular compounds is “Coordination polymer” which is achieved from coordination bonds between metal centers and appropriate ligands. Infinite coordination polymer particles represent an area of growing interest in chemistry and materials science due to their unique and profitable properties.¹³ Coordination polymers (CPs), including metal ions linked together by organic bridging ligands,^{1,2} are new class of polymeric materials that dwell between the interface of molecular coordination chemistry and material science.¹⁰ Synthesizing of these types of compound is frequently used in several applications such as electronics, catalysis, luminescence, drug delivery and separation.¹⁴⁻²³ The process of self-assembly in coordination polymers can frequently be modified by various external factors, such as metal source, ligands, solvents, templates, pH value, temperature, different concentrations and so on, the same reactants can usually result in completely different structures because of the extreme sensitivity of self-assembly to the reaction conditions.²⁴⁻³¹ A variety of methods such as Sol-Gel, microwave and sonochemical synthesis now exist for making numerous coordination polymers, with modest control over particle size and shape.^{13,17,26-30} Ultrasonic irradiation dramatically influences on the reactivity of different kind of inorganic solids. The use of ultrasound to accelerate chemical reactivity is becoming a common and widely applicable laboratory technique. The physical origins of the chemical effects of ultrasound lie principally in acoustic cavitation and other non-linear phenomena.^{31,32} The creation, expansion and collapse of bubbles in ultrasonically irradiated liquids generate localized hot spots from acoustic cavitation which are responsible for most homogeneous

sonochemistry. Ultrasonic method, with benefit such as cost effectively and being environmentally friendly, is suitable for synthesizing of nanoscale coordination supramolecular compounds.^{31,32} Herein, we wish to report a potassium-based coordination polymer, $[K(\mu_7\text{-HTar})]_n$ (**1**) with zigzag chain $K\cdots K$ metallophilic interactions, which was characterized by IR spectroscopy and single-crystal X-ray diffraction. Prior to this study, some coordination polymers with metallophilic interaction such as $[Na(\mu_2\text{-Hdcpa})(\mu_3\text{-dcpa})]_n$, (Hdcpa = 2,4-dichlorophenoxyacetic acid)³³, $[Tl(\mu_3\text{-HTA})]_n$, (H_2TA = terephthalic acid)³⁴ and $\{[Ag(1\text{-methylhydantoin})]\cdot 0.5H_2O\}_n$ ³⁵ were reported. Moreover, the effects of ultrasonic irradiation, concentration of initial reagents and types of solvents on formation of compound **1** nanostructures were investigated.

2. Experimental

2.1. Materials and Physical Techniques

All reagents and solvents for the synthesis and analysis were commercially available and were used as received. The molecular structure plots were prepared using Mercury. PARSONIC 15S ultrasonic bath (with the frequency 28 KHz) was used for the ultrasonic irradiation. Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. IR spectra were recorded using an Equinox 55 FT-IR spectrometer (Bruker, Bremen, Germany) in ATR form, in the range of 400-4000 cm^{-1} with 4.0 cm^{-1} resolution and the 16 scan's numbers. Intensity data for $[K(\mu_7\text{-HTar})]_n$ (**1**) was collected with an Oxford Diffraction SuperNova CCD diffractometer using Mo-K_α radiation, the temperature during all data collections was maintained at 100.0(2) K using an Oxford Cryosystems cooling device. The structures were solved by direct methods and difference Fourier synthesis.³⁶ Thermal ellipsoid plots were generated using the program ORTEP-3.³⁷ within the WINGX suite of programs.³⁸ The thermal behavior was measured with a PL-STA 1500 apparatus between 20 and 800 $^\circ\text{C}$, with a heating rate of 5 $^\circ\text{C}\cdot\text{min}^{-1}$, under a nitrogen atmosphere. X-ray powder diffraction (XRD) measurements were performed

using an X'pert diffractometer manufactured by Philips with monochromatized $\text{CuK}\alpha$ radiation ($\lambda = 1.54056 \text{ \AA}$) with step-size of 0.01671 (degree). The X-ray source was operated under voltage and current of 40 kV and 30 mA, respectively. Bragg-Brentano was used as a source – detector geometry with scintillation detector. Additional attachments or peripheral equipment such as anti-scatter slit (1°), divergence slit (1°), monochromator and soller slit (0.04 rad) were also used in this diffractometer. The samples were prepared as fine powders on silicon based material. Simulated XRD powder patterns based on single crystal data were prepared using the Mercury software. The samples were characterized with a scanning electron microscope (Philips XL 30) with gold coating.

2.2. Synthesis of $[\text{K}(\mu_7\text{-HTar})]_n$ (1) and preparation of its single crystals.

1.5 mmol (0.225 gr) of (+)-tartaric acid (H_2Tar) was dissolved in 20 mL H_2O and was mixed and stirred with solution of 3.0 mmol (0.171 gr) KOH in 5 mL H_2O at 100°C for an hour. After filtering, it was allowed to evaporate for several days and then suitable colorless crystals were obtained. d.p. = 275°C , yield: 0.116 g, 41.2 % based on final product.

2.3. Synthesis of $[\text{K}(\mu_7\text{-HTar})]_n$ (1) with altering concentrations of initial reagents as the bulk samples and by sonochemical process.

In order to study the effect of various concentrations on synthesis of compound **1** as a bulk sample, first, 4 mmol (0.600 gr) of H_2Tar in 20 mL H_2O and 8 mmol (0.456 gr) of KOH in 5 mL H_2O were mixed, heated and stirred at 100°C for 3 hours. Then, it was allowed to evaporate for several days. Consequently, the white precipitate was formed (**1LWB**). d.p. for **1LWB** = 270°C , yield: 0.580 g, 77.0 % based on final product. The second reaction condition was similar to previous one except 16 mmol KOH (0.912 gr) which was solved in 5 mL H_2O , was added to 8 mmol of H_2Tar (1.200 gr) in 20 mL

water. White precipitate was formed which was dried at room temperature (**1HWB**). d.p. for **1HWB** = 271 °C, yield: 0.731 g, 48.4 % based on final product. To consider the effect of ultrasonic irradiation on formation of compound **1**, 8 mmol (1.200 gr) of H₂Tar in 20 mL H₂O and 16 mmol (0.912 gr) of KOH in 5 mL H₂O were mixed under the ultrasonic irradiation. The obtained precipitates were filtered and dried at room temperature (**1HWS**). d.p. for **1HWS** = 250 °C, yield: 0.870 g, 57.6 % based on final product.

2.4. Synthesis of [K(μ_7 -HTar)]_n (**1**) nanostructure in various solvent by sonochemical process.

To consider the effect of different solvents on formation of compound **1**, three types of reaction were designed. First, we used ultrasonic bath with 4:8 mmol concentrations of initial reagents (H₂Tar:KOH) in a solution mixture of 3 mL H₂O and 7 mL acetonitrile. The precipitates were obtained after 3 hours and then filtered, subsequently washed with water and then dried (**1LAS**), d.p. = 251 °C, yield: 0.620 g, 82.0 % based on final product. Second and third reactions were performed under the same conditions, however, with different solvents, ethanol, (**1LES**, d.p. = 272 °C, yield: 0.681 g, 90.2 % based on final product) and methanol, (**1LMS**, d.p. = 271 °C, yield: 0.640 g, 84.8 % based on final product).

3. Result and discussion.

The reaction between (+)-tartaric acid (H₂Tar) and KOH provided a crystalline material of the general formula [K(μ_7 -HTar)]_n (**1**). Determination of the structure of compound **1** by X-ray crystallography (Tables S1 and S2 in the SI) showed that it has one type of K atom with **KO₉** coordination sphere (Figure 1a). H₂Tar was not deprotonated completely and HTar⁻ is bridging between seven K ions and can be considered as heptadentate ligand (Figure 1b). Each of the -COOH and -COO⁻ groups of HTar⁻ bridges between three K ions (Figure 1b). The two -OH group of HTar⁻ showed different coordination behavior.

One of them is coordinated a K ion which has coordination bond with -COO^- group and another -OH is coordinated to infinite K ion (Figure 1b). Compound **1** is a three-dimensional coordination polymer (Figure 1c) and has zigzag chain $\text{K}\cdots\text{K}$ metallophilic interactions with distance of $4.0479(7)$ Å (Table S2) which is lower than the sum of the vander Waals radii of two K atoms³⁹ (5.50 Å, Figure 2a). The neighbor three K ions have the bond angle of $139.939(19)^\circ$ (Table S2 in the SI). HTar^- with variety of hydrogen bonding donors and acceptors formed widespread hydrogen bonding network in three-dimensional structure of **1** (Table S3 in the SI and Figure 2b). Thus the broad absorption band around $3000\text{-}3700\text{ cm}^{-1}$ in its IR spectrum (Figure 3a) is due to some reasons: i. deprotonation of the carboxylic acid groups in H_2Tar were not occurred completely, ii. presence of two -OH groups on H_2Tar and iii. widespread hydrogen bonding network in the structure of **1**. The absorption bands in the frequency of 1100 and 1580 cm^{-1} attributed to C ~~-O-C-O bonds in~~ -O-C-O bonds in -COOH group. The appearance of C=O bond in -COOH group of HTar^- in the frequency of 1580 cm^{-1} is as a result of its coordination to K ion. Also the absorption bands in the frequency of 1300 and 1400 cm^{-1} attributed to ν_{sym} and ν_{asym} of -COO^- group in **1**.

In order to evaluation the effects of concentration, ultrasonic irradiation and types of the solvent on morphology of compound **1**, we designed some experiments and synthesized six samples of **1** under different conditions (Table 1).

First, for considering the effect of concentration of initial reagents we performed two reactions for synthesizing bulk sample of **1** with $\text{H}_2\text{Tar}:\text{KOH}$ molar ratios of $4:8$ (**1LWB**) and $8:16$ (**1HWB**). The FT-IR spectra of them (Figure 3b,c) showed that **1LWB** and **1HWB** have the same structure as the crystal sample (Figure 3a). Figure 4a shows the simulated XRD pattern from single crystal X-ray data of **1**. Figure 4b,c show the XRD patterns of the typical bulk samples of **1LWB** and **1HWB**. Acceptable matches with slight differences in 2θ , were observed between the simulated from single-crystal X-ray data pattern (Figure 4a) and that from the experimental powder X-ray diffraction pattern

of **1LWB** and **1HWP** (Figure 4b,c). The SEM image of **1LWB** indicates the formation of compound **1** spongy microstructure with nanostructural surface (Figure 5a). By concentration increase, SEM image (Figure 5b) did not show the spongy structure in **1HWP** but existence of nanoparticles in its surface is approved. Thus with concentration increase, the hollow spaces was filled. In order to study the effect of ultrasonic irradiation on forming compound **1**, another reaction with 8:16 molar ratio of $\text{H}_2\text{Tar}:\text{KOH}$ was performed by sonochemical process. The FT-IR spectrum (Figure 3d) approved that this sample also has the same structure as the crystal sample (Figure 3a). Figure 4d shows the XRD patterns of the typical sonochemical prepared sample of **1HWS**. Acceptable matches were observed between the simulated from single-crystal X-ray data pattern (Figure 4a) and that from the experimental powder X-ray diffraction pattern of **1HWS** (Figure 4d). The SEM image of **1HWS** (Figure 5c) did not show any difference in comparison with **1HWP** (Figure 5b) but its surface composed from nanocubes of **1** instead of nanoparticles in **1HWP**. Thus ultrasonic irradiation has led to directional growth of nanostructures on surface of compound **1** microstructure.

Second for studies the types of the solvent effect on morphology of compound **1**, three reactions in the presence of acetonitrile (**1LAS**), ethanol (**1LES**) and methanol (**1LMS**) with 4:8 ratios of initial reagents were performed under ultrasonic irradiation (Table 1). The FT-IR spectra of them (Figure 3e-g) approved the formation of them successfully. Acceptable matches were observed between the simulated from single-crystal X-ray data pattern (Figure 4a) and that from the experimental powder X-ray diffraction patterns of **1LAS**, **1LES** and **1LMS** samples (Figure 4e-g). Figure 6a,b show the SEM images of **1LAS** and **1LES**. A comparison between them indicated that both of them have similar surface composed from small nanoparticles and the solvents only affected on microstructure morphology of **1**. On the other hand with methanol (**1LMS**) instead of acetonitrile (**1LAS**) and ethanol (**1LES**) solvents, spongy microstructure with regular

nanostructure surface was obtained (Figure 6c). It seems that the two factors of concentration and solvent can affect on formation of spongy microstructures of **1**.

TG-DTA data (Figure 7a) indicated that compound **1LWB** is stable up to 255 °C. The endothermic mass loss of 3.6% between 160-180 °C may be attributed to adsorption species on the surface of **1LWB**. Further mass loss of it up to 500 °C attributed to removal of other species such as CO₂ gas molecules (Figure 7a). **1HWB** showed similar thermal behavior to **1LWB** (Figure 7b). This similarity is expected because these two samples were synthesized in the same conditions with different concentration of initial reagents. **1HWS** which was synthesized by sonochemical process is stable up to 221 °C (Figure 7c). No mass loss was observed between 160-180 °C. Thus it seems that ultrasonic waves prevented the adsorption of external species on the surface of **1HWS**. Thermal behavior of **1HWS** after 221 °C is similar to the two previous samples (Figure 7c). A comparison between the thermal behaviors of **1LAS**, **1LES** and **1LMS** (Figure 7d-f, which were synthesized in acetonitrile, ethanol and methanol, respectively) indicated that **1LAS** (Figure 7d) and **1LES** (Figure 7e) have similar behavior and the strange thing in their TG curves is that both of them have continues gradual mass loss up to 220 °C. After 220 °C similar behavior (Figure 7d,e) to previous samples (Figure 7a-c) were observed. Finally **1LMS** sample has similar thermal behavior (Figure 7f) to other previous samples (Figure 7a-c). It seems that the type of solvent that used in synthesis of compound **1** has some effects on thermal stability of them and acetonitrile and ethanol are not good solvents for this proposes.

4. Conclusion.

In summary, a new three-dimensional potassium-based coordination polymer [K(μ -HTar)]_n (**1**) with zigzag chain K...K metallophilic interactions has been synthesized and structurally characterized by single crystal X-ray crystallography. Also widespread

hydrogen bonding network in three-dimensional structure of **1** was observed. K atom has KO_9 coordination sphere in **1**. H_2Tar was not deprotonated completely and HTar^- is bridging between seven K ions and can be considered as heptadentate ligand. The two $-\text{OH}$ group of HTar^- showed different coordination behavior. One of them is coordinated a K ion which has coordination bond with $-\text{COO}^-$ group and another $-\text{OH}$ is coordinated to infinite K ion. The broad absorption band around $3000\text{--}3700\text{ cm}^{-1}$ in its IR spectrum of **1** is due to partial deprotonation of the carboxylic acid groups in H_2Tar , presence of two $-\text{OH}$ groups on H_2Tar and widespread hydrogen bonding network in the structure of it. In order to evaluation the effects of concentration, ultrasonic irradiation and types of the solvent on morphology and thermal behavior of **1**, six samples of it were synthesized under different conditions. Concentration of initial reagents can change the morphology of the final product and with low concentration of initial reagents, spongy microstructures was formed. Ultrasonic irradiation was led to directional growth of nanostructures on surface of compound **1** microstructure. Also types of the solvent can affect on morphology of **1**. With methanol instead of acetonitrile and ethanol, spongy microstructure with regular nanostructure surface was obtained. The solvent used in synthesis of **1** has some effects on thermal stability of them and acetonitrile and ethanol are not good solvents for this proposes.

Supplementary material.

Complete bond lengths and angles, co-ordinates and displacement parameters have been deposited at Cambridge Crystallography Data Center. Supplementary data are available from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK on request, quoting the deposition number 1845659 for compound **1**.

Acknowledgements

The authors would like to acknowledge the financial support of University of Tehran for this research under grant number 01/1/389845.

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Table 1. A summary of reaction conditions for synthesis six samples of $[K(\mu_7\text{-HTar})]_n$ (1).

Compound Label	H ₂ Tar:KOH ratio (mmol)	Ultrasonic Irradiation	Solvent	Thermal behavior
1LWB	4:8	No	Water	It is stable up to 255 °C. Mass loss of it up to 500 °C attributed to removal of other species such as CO ₂ gas molecules.
1HWB	8:16	No	Water	It showed similar thermal behavior to 1LWB.
1HWS	8:16	Yes	Water	It is stable up to 221 °C. Thermal behavior of 1HWS after 221 °C is

				similar to the two previous samples.
1LAS	4:8	Yes	Acetonitrile	TG curve of 1LAS have continues gradual mass loss up to 220 °C.
1LES	4:8	Yes	Ethanol	It showed similar thermal behavior to 1LAS
1LMS	4:8	Yes	Methanol	It has similar thermal behavior to other previous samples (1LWB , 1HWP , 1HWS)

Figure 1. Primary structural building unit in $[K(\mu_7\text{-HTar})]_n$ (**1**) indicating a) coordination sphere around K ion and b) coordination behavior of HTar[−] and c) a fragment of three-dimensional coordination polymer in **1**, (K = violet, O = red, C = gray and H = white, in a and c hydrogen atoms have been omitted for clarity).

Figure 2. Illustrating a) zigzag K...K interactions in $[K(\mu_7\text{-HTar})]_n$ (**1**) with pink dashed line along the crystallographic b axis and b) three-dimensional hydrogen bonding network in **1** with green dashed line, (K = violet, O = red, C = gray and H = white, in a hydrogen atoms have been omitted for clarity).

Figure 3. IR spectra of a) single crystal of compound $[K(\mu_7\text{-HTar})]_n$ (**1**), compound **1** (as bulk sample) prepared with b) 4:8 mmol of H_2Tar and KOH (**1LWB**), c) 8:16 mmol of H_2Tar and KOH (**1HWP**), d) compound **1** synthesized by sonochemical process with 8:16 mmol of H_2Tar and KOH (**1HWS**), compound **1** prepared by sonochemical process with 4:8 mmol of H_2Tar and KOH in 3:7 mixture of e) Water:MeCN (**1LAS**), f) Water:Ethanol (**1LES**) and g) Water:Methanol (**1LMS**).

Figure 4. XRD patterns; a) simulated pattern based on single crystal data of compound $[K(\mu_7\text{-HTar})]_n$ (**1**), compound **1** (as bulk sample) prepared with b) 4:8 mmol of H_2Tar and KOH (**1LWB**), c) 8:16 mmol of H_2Tar and KOH (**1HWP**), d) compound **1** synthesized by sonochemical process with 8:16 mmol of H_2Tar and KOH (**1HWS**), compound **1** prepared by sonochemical process with 4:8 mmol of H_2Tar and KOH in 3:7 mixture of e) Water:MeCN (**1LAS**), f) Water:Ethanol (**1LES**) and g) Water:Methanol (**1LMS**)

Figure 5. SEM images of compound **1** (as bulk sample) prepared with a) 4:8 mmol of H_2Tar and KOH (**1LWB**), b) 8:16 mmol of H_2Tar and KOH (**1HWP**) and c) compound **1** synthesized by sonochemical process with 8:16 mmol of H_2Tar and KOH (**1HWS**).

Figure 6. SEM images of compound **1** prepared by sonochemical process with 4:8 mmol of H_2Tar and KOH in 3:7 mixture of e) Water:MeCN (**1LAS**), f) Water:Ethanol (**1LES**) and g) Water:Methanol (**1LMS**).

Figure 7. Thermal behavior of compound **1** (as bulk sample) prepared with a) 4:8 mmol of H_2Tar and KOH (**1LWB**), b) 8:16 mmol of H_2Tar and KOH (**1HWP**), c) compound **1** synthesized by sonochemical process with 8:16 mmol of H_2Tar and KOH (**1HWS**), compound **1** prepared by sonochemical process with 4:8 mmol of H_2Tar and KOH in 3:7 mixture of d) Water:MeCN (**1LAS**), e) Water:Ethanol (**1LES**) and f) Water:Methanol (**1LMS**).

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Zigzag K-K metallophilic interactions

3D hydrogen bonding network

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