Microwave-assisted synthesis of $C_{10}H_{16}CoO_{14}$ and $C_{10}H_{18}Na_2NiO_{16}$

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Abstract: 1,2,4,5-Benzenetetracarboxylic acid (H4btec) with transition metal ions Co(II) and Ni (II), give two coordination complexes, $C_{10}H_{16}CoO_{14}$ (**I**) and $C_{10}H_{18}Na_2NiO_{16}$ (**II**). These complexes have been synthesized by a fast and efficient method under microwave irradiation. These compounds have been characterized by FT-IR spectroscopy and elemental analysis.

Keywords: 1, 2, 4, 5-Benzenetetracarboxylic acid; Microwave; Cobalt(II) nitrate hexahydrate; Nickel(II) nitrate hexahydrate.

Introduction

Aromatic polycarboxylate ligands have proved to be good building blocks for the construction of coordination polymers [1, 2]. Among the various aromatic polycarboxylate ligands, 1, 2, 4, 5-benzenetetracarboxylic acid (H_4btec) has been proved to be a good candidate due to its various bridging abilities and strong coordination tendency with transition metals to form 2- and 3D networks. It has four COOH groups which can be fully or partially deprotonated, resulting in versatile coordination behavior to metal ions. H4btec has been used as a ligand to create 1D polymeric chain, 2D polymeric sheets, and 3D polymeric networks with a variety of metal ions reported in the literature [3-5]. The various methods have been used for preparation of 2D and 3D polymeric networks such as hydrothermal/solvothermal, branched tube, diffusion and other conventional processes. Bo and co-workers have been reported the synthesis of $[Co(H_2O)_6(H_2btec)]$ [6]. Also, Sun and co-workers have been presented preparation of $[Na_2Ni(btec)(H_2O)_8]_n$ by hydrothermal method[7]. We now report the synthesis of these complexes base on H_4btec using microwave irradiation for the first time. Microwave irradiation is well known as environmentally benign method, which offers several advantages including shorter reaction times, cleaner reaction profiles and simple experimental/product isolation procedures [8]. Microwave irradiation presents a powerful tool toward chemical reactions.

Results and Discussion

The microwave irradiation was employed to obtain two complexes in high yield and short time.

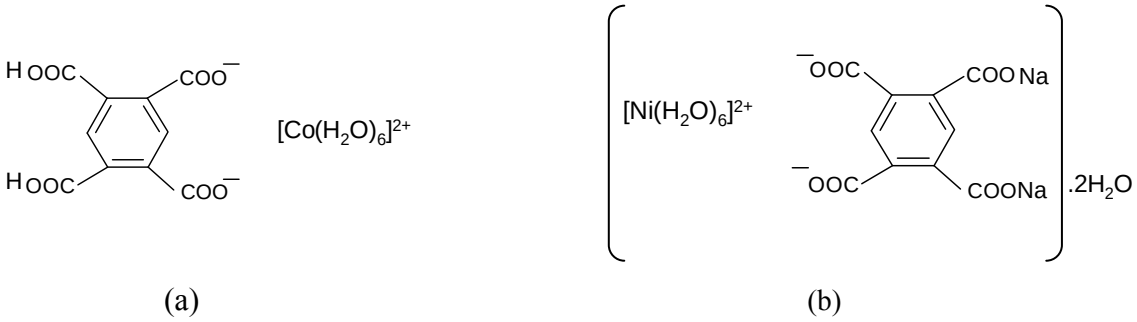
The stretching frequencies for **I** at 1662, 1585, and 1398 cm^{-1} attributed to ν_{as}(COO) and ν_s(COO) vibrations of the carboxylate groups. Stretching frequencies of the infrared spectra at 3419, 2700, 2500, 1709 and 1602 cm^{-1} for I confirmed the presence of the non-ionized COOH[6].

The stretching frequencies for **II** at 1550 and 1369 cm^{-1} assigned to ν_{as}(COO) and ν_s(COO) vibrations of the carboxylate groups.

The infrared spectra of I and II are quite similar with a broad band centred at 3452 and 3459 cm^{-1}, which is due to the O–H stretching vibration of water molecules involved in extensive hydrogen bonding interactions [6].

The composition analyzing for I and II is in good agreement with the calculated values based on the empirical formulas for $C_{10}H_{16}CoO_{14}$ and $C_{10}H_{18}Na_2NiO_{16}$.

The proposed structures for I and II have been given in Scheme. 1.



Scheme 1. The suggested structures of a) $C_{10}H_{16}CoO_{14}$ and b) $C_{10}H_{18}Na_2NiO_{16}$

Experimental Procedure

Synthesis of $C_{10}H_{16}CoO_{14}$ (I). A solution of $Co(NO_3)_2.6H_2O$ (0.582 g, 2 mmol) in water (10 mL) was added to a solution of H_4btec (0.254g, 1 mmol) and KOH (0.224 g, 4 mmol) in water (10 mL) and then irradiated at 180 W. The purple powder was obtained after 6 min. The progress of the reaction was monitored by TLC. After completion of the reaction, product was filtered and washed with water.

Anal. Calcd. for $C_{10}H_{16}CoO_{14}$: C, 28.65; H, 3.85; Co, 14.06. Found: C, 28.79; H, 3.71; Co, 14. 13%.

IR spectrum(KBr, ν, cm^{-1}): 247 w, 422 m, 763 s, 885 w, 1095 m, 1139 vs, 1288 s, 1398 vs, 1585 s, 1662 s, 3452 vs.

Synthesis of $C_{10}H_{18}Na_2NiO_{16}$ (II). A solution of $Ni(NO_3)_2.6H_2O$ (0.580, 2 mmol) in water (10 mL) was added to a solution of H_4btec (0.254g, 1 mmol) and NaOH (0.16 g, 4 mmol) in water (10 mL) and then irradiated at 180 W. The green powder was obtained after 9 min. The progress of the reaction was monitored by TLC. After completion of the reaction, product was filtered and washed with water.

Anal. Calcd. for $C_{10}H_{18}Na_2NiO_{16}$: C, 24.07; H, 3.64;; Ni, 11.77, Na, 9.22. Found: C, 23.89; H, 3.95; Ni, 11.63; Na, 9.14 %.

IR spectrum (KBr, ν, cm^{-1}): 478 s, 507 m, 561 vs, 767 s, 811 s, 914 s, 1139 vs, 1299 s, 1369 vs, 1550s, 3424 s, 3593 s.

Acknowledgment

Financial support by Iran University of Science and Technology is gratefully acknowledged.

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