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Benzene-induced hydro(solvo)thermal synthesis of Cu^{2+} and Zn^{2+} coordination polymers based on 1,3-benzenedicarboxylate

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HIGHLIGHTS

- Two different Cu and Zn-coordination polymers were prepared by hydro(solvothermal) method.
- Presented method does not require the use of an organic base.
- Benzene provokes crystallization of the frameworks.
- Cu-coordination polymer suffers framework decomposition in air.
- Zn-coordination polymer is an air-atmosphere and thermally stable framework.

ARTICLE INFO

ABSTRACT

Hydro(solvo)thermal reactions of $Cu(NO_3)_2 \cdot 2.5H_2O$ or $Zn(NO_3)_2 \cdot 6H_2O$ with $1,3-H_2bdc$ provided two structurally different coordination polymers; a 2D network "metallacalixarene" based on the paddlewheel-type cluster with the formula $[Cu(1,3-bdc) \cdot H_2O] \cdot H_2O$ (**1**) and a 3D framework $[Zn(1,3-bdc)]$ (**2**), respectively. The use of a base was unnecessary in this synthesis; however, the presence of benzene played a crucial role in the crystallization of the desired products and could act as a template molecule in the synthesis of polymer (**1**). The unsaturated apical sites in the Cu_2 cluster of polymer (**1**) were occupied by water molecules, what consequently led to the structural decomposition of the framework after exposing the sample in air, which was not the case of the Zn-coordination polymer (**2**).

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1. Introduction

Currently, coordination polymers attract great attention due to their use in a number of applications, specifying only a few of them: in adsorption and separation of gases [1], catalysis [2], magnetism [3], luminescence [4], and drug delivery [5]. Many research efforts have focused on controlling the route of synthesis to obtain materials with desired properties.

In many cases, the topological features of coordination polymers can be predicted based on both, the selected organic linker and the type of metal cluster formed in solution [6]. Whereas the organic linkers are usually rigid-body molecules, metal clusters can show different numbers of atoms, sizes and conformations [7]. There are several factors which drive the formation of a metal cluster in solution and, consequently, the final structure of the coordination polymer. Such factors include the molar ligand to metal ratio, pH, the temperature and time of reaction, the type of solvent, the type of counterions etc. [8—11].

Moreover, the appropriate selection of the synthetic route affects the structure of the resulting coordination polymer. Among the most common methods of synthesis, the slow diffusion of solutions at room temperature, hydro- or solvothermal synthesis assisted by microwaves irradiation or conventional heating in an oven, can be distinguished [12]. In all these cases, more than one type of structure might be formed from the same metal cluster and organic linker depending on the crystallization conditions,

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including the type of solvent, presence and type of template molecules and identity of the axial ligands [13,14]. The number of these factors demonstrates the difficulty in the perfect control of the synthesis of supramolecular structures and a systematic study of the solvent influence on the synthesis of coordination polymers is still required [15].

One of the biggest advantages of using water in a synthesis of coordination polymers is the environmental benefit, the low cost and its volatility so it can be removed from the product very easily. Although hydrothermal synthesis requires relatively long time of reaction in comparison with, for example, microwave-assisted method, it offers many advantages over other conventional and non-conventional synthetic methods. The hydrothermal method frequently allows growing single crystals suitable for X-ray diffraction what is in general more difficult to achieve by another synthetic route. Therefore, the hydrothermal synthesis is still used as a common method of preparation of coordination polymers [16,17].

Although some copper and zinc coordination polymers based on 1,3-benzenedicarboxylate have been reported, the synthetic methods applied required the presence of an organic and/or inorganic base [18–21]. The organic bases such as triethylamine, pyridine, imidazole or benzimidazole have been widely used in hydrothermal and/or solvothermal synthesis. However, it has been observed that triethylamine or pyridine can be easily coordinated to the metal cluster due to the presence of the nitrogen donor atoms in theirs structure, and the removal of these molecules at high temperature may lead to the structure collapse [18].

On the other hand, imidazole and benzimidazole are large enough to prevent the possibility of being coordinated to the metal center. Nevertheless, they can act as a co-ligand in the synthesis and they can be included in the final structure [22,23]. This fact can be sometimes advantageous but it does not allow the perfect control of the synthesis, frequently leading to unpredictable structures.

Inorganic bases present in the reaction medium can easily generate the inorganic compounds such as oxides and hydroxides with the metal of interest. As the oxides of transition metals are poorly soluble in water and insoluble in organic solvents, their removal is difficult and they remain in the phase of the main product. Consequently, the presence of unwanted phases influences the properties of materials.

Thus, the development of a synthesis without the need of use of an organic and/or inorganic base is important and advantageous. Not only we can better control the synthesis and predict the structure of coordination polymers, but also we can eliminate the formation of hardly removal inorganic impurities caused by the presence of a base in the reaction medium.

Therefore, in this paper we present a hydrothermal synthesis of Cu^{2+} and Zn^{2+} coordination polymers based on 1,3-benzenedicarboxylate without the use of a base, and the role of benzene as an auxiliary solvent in this synthesis.

2. Experimental

2.1. Materials and methods

Raw materials were used as received without further purification. Isophthalic acid (1,3-H_2bdc) was purchased from Sigma Aldrich. $Cu(NO_3)_2 \cdot 3H_2O$, $Zn(NO_3)_2 \cdot 6H_2O$ and solvents: benzene and ethanol were purchased from Vetec.

Single crystal X-ray diffraction data were collected on a KUMA KM4CCD *j*-axis diffractometer with graphite monochromated Mo Kα radiation ($\lambda = 0.71073$ Å). Powder X-ray diffraction (PXRD) data were recorded on a Bruker D8 Advance diffractometer with Cu Kα

($\lambda = 1.5406$ Å) radiation. The morphology of the samples was verified using a scanning electron microscope Model: XL-30 – ESEM Phillips and 20 kV electron beam. Thermogravimetric analyses (TGA) were performed on a Shimadzu DTG-60H thermal analysis system in a continuous nitrogen flow atmosphere. Elemental analysis was performed on CE Instruments CHNS-O EA 1110. FT-IR spectra were obtained by using a Bruker spectrometer (model IFS66) and KBr pellets. Absorption peaks were described as follows: strong (s), medium (m) and weak (w).

2.2. Synthesis of [Cu(1,3-bdc)·H_2O]·H_2O (**1**)

A suspension of isophthalic acid 1,3-H_2bdc (0.166 g, 1 mmol) and $Cu(NO_3)_2 \cdot 3H_2O$ (0.242 g, 1 mmol) in a water/benzene (5/3 mL) solvent mixture was placed in a 23 mL Teflon-lined steel reactor and heated at 180 °C for 3 days. Subsequently, the reactant mixture was cooled to room temperature and the resulting solution was filtered off. The blue microcrystals of (**1**) obtained with a yield of 55% (based on isophthalic acid) were mechanically separated from the green impurity, washed with water and ethanol, and dried in oven at 60 °C for 2 h.

IR (KBr pellet, ν/cm^{-1}): 3492 (w), 3353 (w), 1615 (s), 1579 (m), 1455 (w), 1389 (s), 754 (m), 725 (m), 492 (w).

Anal. Calcd for [Cu(1,3-bdc)·H_2O]·H_2O: C, 36.45%; H, 3.03%. Found: C, 36.91%; H, 2.73%.

2.3. Synthesis of [Zn(1,3-bdc)] (**2**)

Equimolar amounts of $Zn(NO_3)_2 \cdot 6H_2O$ and 1,3-H_2bdc were dissolved in a water/benzene (5/3 mL) mixture and heated in a 23 mL Teflon-lined steel reactor at 180 °C for 3 days. The resulting mixture was cooled to room temperature to produce the X-ray-quality sheet-like crystals of (**2**) with a yield of 24.6% collected by filtration, washed with water and ethanol, and then dried in oven at 60 °C for 2 h.

IR (KBr pellet, ν/cm^{-1}): 1603 (s), 1556 (s), 1539 (s), 1466 (s), 1408 (s), 1085 (w), 930 (w), 848 (w), 828 (w), 748 (m), 735 (m).

2.4. X-ray crystallography

This analysis was performed only in the case of polymer (**2**) since the crystal dimensions of the polymer (**1**) were inadequate. Experimental data were collected on a KUMA KM4CCD *j*-axis diffractometer with graphite monochromated Mo Kα radiation ($\lambda = 0.71073$ Å) at 273.2 K. The data were processed using the CrysalisPro (Oxford Diffraction) program package [24]. Measurement of diffraction data was performed at 298 K. Empirical absorption correction was applied using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm of the CrysalisPro package. An initial structure model was obtained by charge flipping (SUPERFLIP, Palatinus [25]). Calculations were carried out using the SHELX system [26] run under WINGX environment [27].

3. Results and discussion

3.1. Synthesis

Hydro(solvo)thermal reaction of $Cu(NO_3)_2 \cdot 3H_2O$ or $Zn(NO_3)_2 \cdot 6H_2O$ with 1,3-H_2bdc in the presence of benzene, provided two coordination polymers of the formulas: [Cu(1,3-bdc)·H_2O]·H_2O (**1**) and [Zn(1,3-bdc)] (**2**). The synthesis was performed according to the literature procedure [28].

The synthesis was also attempted using pure water in order to understand the role of benzene in this synthesis. However, in both cases, the reaction failed in producing coordination polymers and

the unconverted starting material 1,3-H$_2$bdc (designated as (**3**) and (**4**), see Fig. 1) was recuperated in 43 and 45%, respectively.

When the resulting solution was left standing at room temperature for several months, the formation of coordination polymers did not occur. Therefore, it can be assumed that the presence of benzene in the synthesis somehow promotes the formation of coordination polymers.

The isophthalic acid is poorly soluble in water, but under the hydrothermal conditions, water has a reduced viscosity and the diffusivity of reactants is facilitated which means that solids can dissolve and migrate more rapidly.

Adding a small amount of auxiliary solvent is often used to adjust the solution polarity and to enhance the crystal growth [29]. Moreover, we considered as well, that benzene might play a template role in this synthesis, what has been often reported [30].

3.2. IR spectra

Fig. 2 presents the IR spectra of obtained samples (**1**–**4**) and for comparison of starting material 1,3-H$_2$bdc.

The identity of (**3**) and (**4**) with 1,3-H$_2$bdc was first confirmed by the presence of a characteristic peak in their IR spectra at approximately 1695 cm^{-1}, corresponding to the stretching vibrations of COOH groups.

The IR spectra of the two coordination polymers (**1**) and (**2**) are significantly different. However, in both spectra, no bands in the region 1690–1730 cm^{-1} are present, indicating the complete deprotonation of the carboxyl groups. For (**1**), the significant $\nu_{as}(COO^-)$ bands at 1615 cm^{-1} (very strong) and 1579 cm^{-1} (weaker), as well as $\nu_s(COO^-)$ band at 1389 cm^{-1} appeared. The difference between $\nu_{as}(COO^-)$ and $\nu_s(COO^-)$ provides useful information about the nature of carboxylate coordination [31]. $\Delta\nu = 208$ cm^{-1} signifies that each carboxylate in (**1**) is bonded to two copper atoms in a bi-monodentate fashion.

In the IR spectrum of (**2**), the bands at 1603–1539 cm^{-1} and at 1466–1408 cm^{-1} corresponding to the stretching asymmetric and symmetric vibrations of carboxylate groups, respectively, appeared. $\Delta\nu = 134$ cm^{-1} indicates that each carboxylate in (**2**) adopts bidentate bridging modes.

In addition, the appearance of a large band at around 3400 cm^{-1} in the IR spectrum of (**1**) (see peaks description) may suggest the presence of water molecules in the structure, what was not the case of polymer (**2**) for which no such a band was observed.

3.3. X-ray crystal structure determination

X-ray structure analysis revealed the formation of a 3D coordination polymer (**2**) with three crystallographically independent Zn^{2+} ions (Zn1, Zn2 and Zn3), all in distorted tetrahedral coordination (Fig. 3). Atoms Zn1 and Zn3 sit on 2-fold rotation axes so the molar ratio of Zn1:Zn2:Zn3 is 1:2:1. It can be seen that each Zn atom is coordinated by four oxygen atoms coming from four different 1,3-bdc ligands.

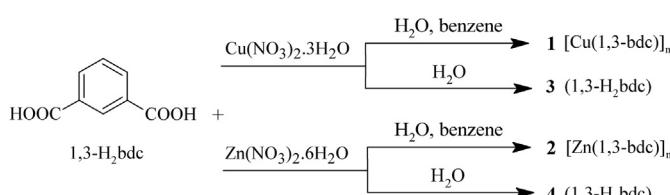Crystal data for polymer (**2**): Empirical formula: C$_{16}$H$_8$Zn$_2$O$_8$; M = 459 g mol^{-1}; crystal system: tetragonal; space group: P4$_3$2$_1$2;
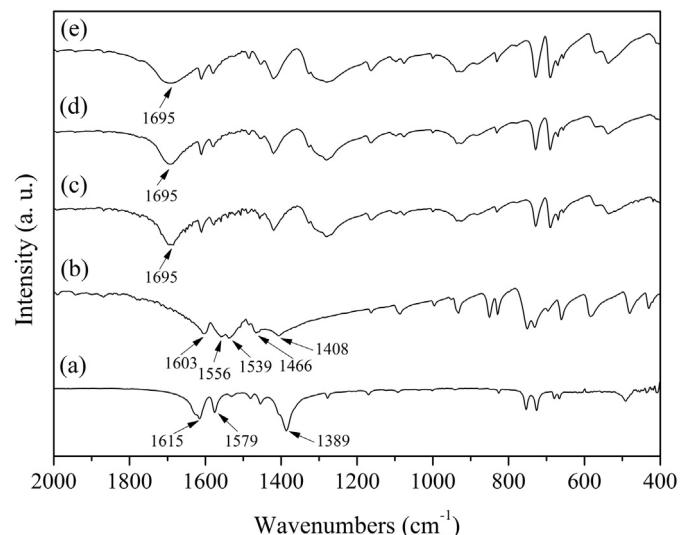
Fig. 2. FT-IR spectra of: (a) compound (**1**), (b) compound (**2**), (c) and (d) samples prepared in the absence of benzene (**3**) and (**4**), (e) starting material 1,3-H$_2$bdc.

a (Å) = 9.6432(4), b (Å) = 9.6432(4), c (Å) = 32.2446(18). The same unit cell parameters have been already reported for C$_{16}$H$_8$Zn$_2$O$_8$ [21], which confirm the identity of the structures, despite the use of different synthetic conditions. In Ref. [21], the authors used a 1:2 (ligand to metal) molar ratio, as well as melamine and NaOH as bases. We show in our study that the same structure can be obtained without the use of a base.

We solved the phase problem again, and the result is identical as reported before. Since the quality of the new solution is similar, we have not deposited it at CCDC. It was verified that there are no voids available for hosting guest benzene molecules in the structure of (**2**). Therefore, it can be confirmed, that the role of benzene in the case of preparation of polymer (**2**) is limited only to aid in the crystallization process. Furthermore, no water molecules in the structure of (**2**) were found, which confirms the IR results.

3.4. Powder X-ray diffraction (PXRD)

The PXRD patterns of obtained samples (**1**–**4**) and for comparison of 1,3-H$_2$bdc are depicted in Fig. 4. The simulated PXRD patterns of the polymers (**1**) and (**2**) are also shown. These patterns were calculated from the single crystal data available in the literature [21,28].

The PXRD patterns of the samples prepared in the absence of benzene show a good agreement with the pattern obtained for the starting material 1,3-H$_2$bdc, what is in line with IR results. However, a significant preferential orientation for the diffraction peak at 28° can be observed in these patterns, what is not the case observed in the pattern of the starting material (see Fig. 4g). This fact can indicate that under these synthetic conditions, 1,3-H$_2$bdc becomes soluble in water and then it is recrystallized, presenting a new morphology.

The PXRD patterns of the samples (**1**) and (**2**) are consistent with their corresponding simulated patterns obtained from the literature data [21,28]. The relative intensity of the diffraction peaks in each pattern depends on the morphology of the powder samples. For instance, the prepared polymer (**1**) had the same framework as the one described in Ref. [28], based on position of the peaks in their PXRD patterns. However, the relative intensities of these peaks differed significantly from those observed in a simulated pattern based on a single-crystal data.
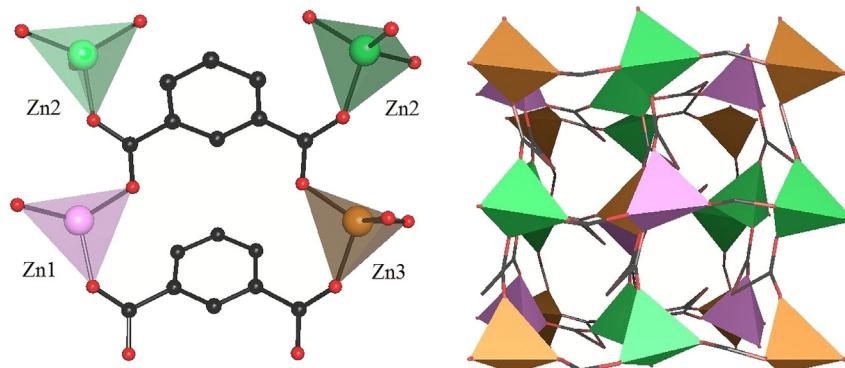
Fig. 1. Synthesis scheme.

Fig. 3. Coordination environments of Zn^{2+} atoms (on the left) and a partially expanded 3D framework of the coordination polymer [Zn(1,3-bdc)] (**2**) (on the right). For clarity, benzene rings in the net are represented as simple linkers and hydrogen atoms are removed. Color code: black − carbon, red − oxygen, magenta − zinc 1, green − zinc 2, orange − zinc 3. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

In spite of applying the same synthetic conditions, the different geometry preference; square planar for copper and tetrahedral in the case of zinc, led to the formation of two structurally different coordination polymers.

Polymer (**1**) is a 2D-framework composed of nanometer bowls, each consisted of four square paddle-wheel SBU (Secondary Building Unit) [Cu₂(1,3-bdc)₄] (Fig. 5). Because of their similarity to the classical organic calixarenes, due to the shape of the nano-bowls, such coordination polymers have received their name-metallacalixarenes [32]. In this case, we can assume that benzene not only helps in the crystallization, but also may act as a template molecule, which facilitates the self-assembly process and forces the shape of the final product. Our assumption is based on the fact that the size of these nano-bowls is adequate to accommodate a molecule of benzene.

The structure of coordination polymer (**1**) requires some further considerations. The hydro(solvo)thermal reaction led to the formation of a mixture of two phases, from which the desired phase of coordination polymer (**1**), was isolated from a green impurity. Fig. 6 presents the PXRD patterns of both phases (a and b). Although we were not able to identify the phase of impurity, we observed that a similar pattern was obtained for the coordination polymer (**1**) which was dried in air and not in an oven (c).

We presumed that the structure of (**1**) changes when exposed to air-atmosphere for a long time. The unsaturated apical coordination sites in the paddle-wheel cluster of copper are available for the water molecules which can bind strongly to the Cu cluster in the reaction medium, leading to hydrolysis and structural decomposition of the
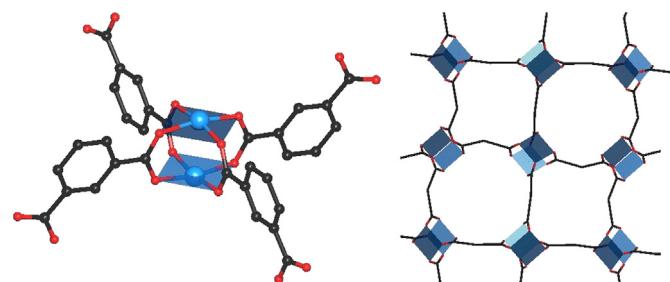

Fig. 5. Structure of [Cu(1,3-bdc)·H₂O]·H₂O (**1**): coordination environments of paddle-wheel Cu^{2+} cluster (on the left) and a partially expanded 2D layer (on the right). For clarity, the coordinated and lattice water molecules and hydrogen atoms are removed; benzene rings in the net are represented as simple linkers. Color code: black − carbon, red − oxygen, blue − copper.
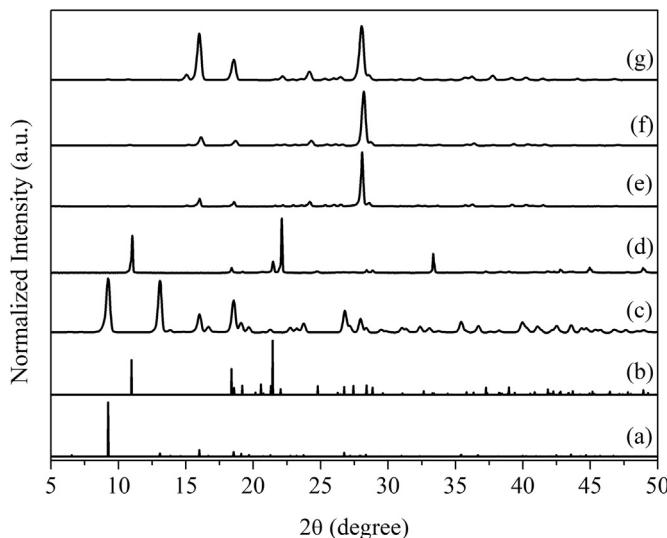

Fig. 4. Simulated PXRD patterns of polymers: (a) (**1**), (b) (**2**) and PXRD patterns of samples obtained: (c) polymer (**1**), (d) polymer (**2**), (e), (f) samples prepared in the absence of benzene (**3**) and (**4**), (g) starting material 1,3-H₂bdc.
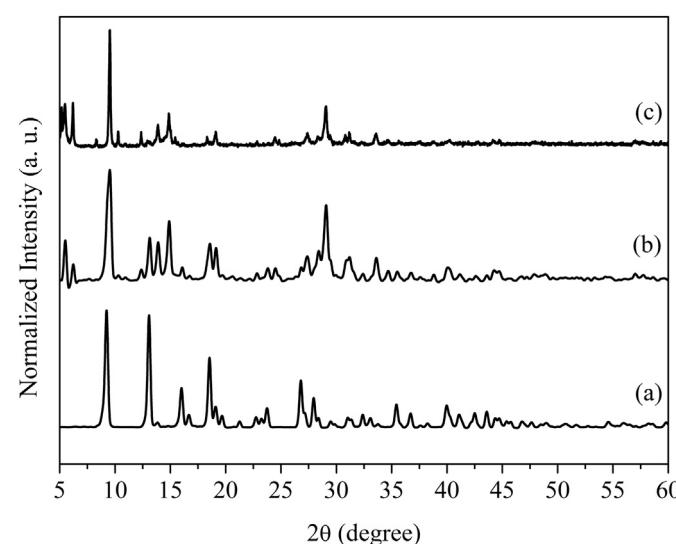

Fig. 6. PXRD patterns of: (a) phase-pure polymer (**1**), (b) isolated green impurity, (c) sample of (**1**) dried in air.
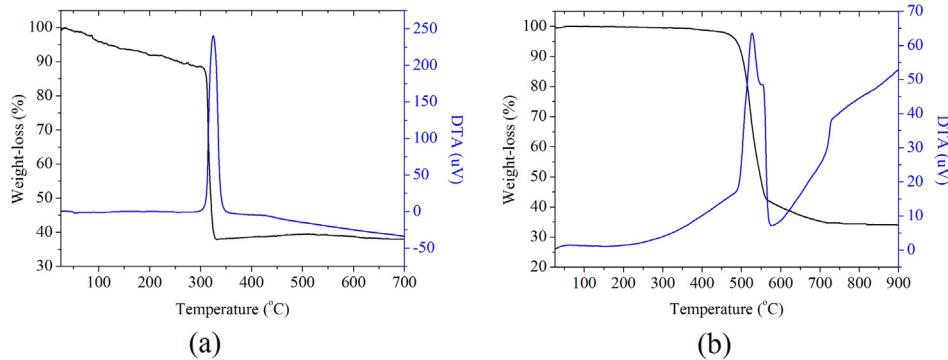
Fig. 7. TGA/DTA profiles of: (a) polymer (**1**), (b) polymer (**2**).

framework after exposing to air. That was not the case of coordination polymer (**2**), in which the completely saturated tetrahedral coordination sites of zinc, do not allow the binding of water molecules.

3.5. Thermogravimetric analysis (TGA)

Thermogravimetric analyses (TGA) were carried out to determine the thermal stability of obtained coordination polymers (**1**) and (**2**). The corresponding TGA profiles are presented in Fig. 7.

The TGA curve of (**1**) shows an initial loss of 11% up to 302 °C probably attributed to the guest water molecules (coordinated and lattice) and/or benzene present in the structure. The further sharp weight loss of 51% in the range of 302–328 °C corresponds to the structure decomposition, following the transformation to the copper oxide at 328 °C.

A comparison of the weight loss profiles shows that the Zn(1,3-bdc) polymer (**2**) is much more stable than [Cu(1,3-bdc)·H$_2$O]·H$_2$O (**1**) and starts to decompose at 450 °C. Moreover, no weight loss attributed to the water and/or benzene molecules is observed,

which confirms that no such molecules are coordinated to the zinc nodes or present in the polymer lattice.

3.6. Scanning electron microscopy (SEM)

The morphology of samples (**1**–**4**) was studied by scanning electron microscopy (SEM) and the images are presented in Fig. 8.

The SEM images confirm the phase purity of samples (**1**–**4**). The morphology of polymer (**1**) consists of aggregated whiskers (Fig. 8a), whereas the phase of (**2**) exhibits a multi-layered sheet structure. Images of samples (**3**) and (**4**) are similar and are made up of big-plate aggregates (>100 μm), significantly larger than microstructures observed for polymers (**1**) and (**2**). Noteworthy is the fact that the starting material, 1,3-H$_2$bdc, possesses no specific morphology (not shown here), whereas 1,3-H$_2$bdc isolated after hydrothermal reaction, designated as samples (**3**) and (**4**), have well-defined plate-like morphology. This fact confirms the preferential orientation observed for the diffraction peak at 28° in the PXRD patterns of (**3**) and (**4**).
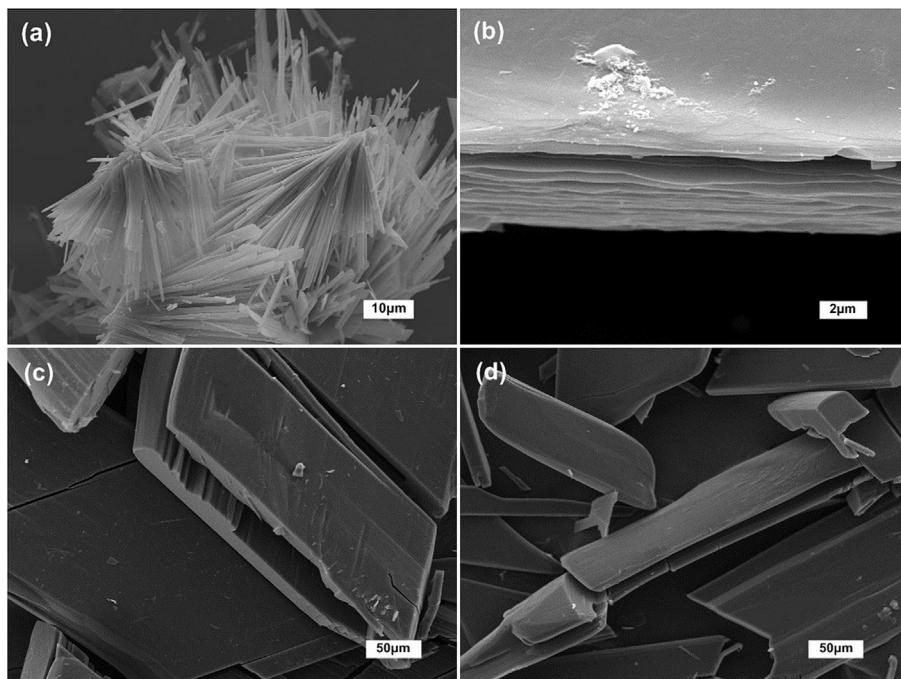

Fig. 8. SEM images of: (a) compound (**1**), (b) compound (**2**), (c) and (d) samples prepared in the absence of benzene (**3**) and (**4**).

4. Conclusions

Two coordination polymers based on 1,3-benzenedicarboxylate and Cu^{2+} or Zn^{2+} cations were obtained in hydro(solvo)thermal reaction without the use of a base. Nevertheless, we observed that the addition of a small amount of benzene was necessary to provoke the formation of frameworks in such conditions.

The obtained polymers represented different topology, mostly because of the zinc cation prefers tetrahedral coordination instead of square planar coordination in the case of copper. Copper coordination polymer (**1**) was a 2D-framework, metallacalixarene, based on paddlewheel-type of cluster, whereas Zn-based polymer (**2**) was a 3D coordination polymer with three crystallographically independent Zn^{2+} ions, all in tetrahedral coordination. We presumed that in the case of polymer (**1**), benzene might additionally play a template role in the synthesis, due to the size of the cavities in the structure, which were appropriate to host a molecule of benzene.

The open apical coordination sites in the copper cluster allowed water molecules to be strongly bonded, in the aftermath leading to the hydrolysis and structural changes of the framework. Due to the completely saturated, tetrahedral coordination environment of the zinc cation, the hydrolysis was not observed in the case of coordination polymer (**2**), providing air-atmosphere and thermally stable framework. The isolated post-reaction isophthalic acid exhibited well-defined morphology, in contrast to the starting material, which did not show any particular morphology.

Acknowledgments

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