

Flow Synthesis of Ni–BDC: Effects of Flow Rate and Temperature on Yield and Apparent Crystallinity

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Abstract

Nickel terephthalate (Ni–BDC) was synthesized in a sonicated coiled-tubing flow reactor to examine the effects of flow rate and temperature on isolated yield and apparent crystallinity. A solvothermal product prepared at 140 °C for 12 h was used as the reference. Flow synthesis was evaluated at 0.10, 0.20, and 0.40 mL min^{−1} at 75 °C, followed by temperature variation from 70 to 80 °C at 0.20 mL min^{−1}. Product formation was assessed by powder X-ray diffraction and Fourier-transform infrared spectroscopy, while isolated yield and apparent crystallinity were compared. Increasing flow rate from 0.10 to 0.40 mL min^{−1} reduced yield from 60.76% to 42.78% and apparent crystallinity from 18.54% to 11.39%. At 0.20 mL min^{−1}, increasing temperature raised yield from 29.22% at 70 °C to 72.66% at 80 °C; apparent crystallinity at 80 °C reached 25.26%. Two independently synthesized batches prepared at 80 °C and 0.20 mL min^{−1} showed close qualitative agreement in their PXRD profiles. Compared with the solvothermal route, the flow condition operated at a temperature 60 °C lower and required approximately one-quarter of the processing time while providing higher yield and comparable apparent crystallinity. These results support the feasibility and qualitative reproducibility of the investigated flow synthesis procedure.

Keywords: nickel terephthalate; Ni–BDC; flow synthesis; powder X-ray diffraction

INTRODUCTION

Metal–organic frameworks (MOFs) are crystalline coordination materials formed from metal ions or clusters and multitopic organic linkers. Their structural and compositional diversity enables the adjustment of pore environments, framework dimensionality, and physicochemical properties through the choice of metal node, linker, solvent, and synthesis conditions. As a result, MOFs have been investigated for adsorption and separation, catalysis, sensing, and energy-related applications. However, their properties depend strongly on reproducible synthesis and characterization because changes in precursor concentration, solvent composition, temperature, reaction time, and post-synthetic treatment can affect phase identity, defect concentration, crystallinity, and accessible porosity (Howarth et al., 2017; Palapessy et al., 2025; Raptopoulou, 2021).

Nickel terephthalate, commonly denoted Ni–BDC, is formed through coordination between Ni²⁺ centers and deprotonated benzene-1,4-dicarboxylate

(BDC^{2−}) linkers. However, the term Ni–BDC may refer to materials with different coordination environments, dimensionalities, guest contents, and morphologies. Recent studies have reported Ni–BDC and related M–BDC materials for adsorption, extraction, photocatalysis, and electrochemical applications (Azhar et al., 2024; Chang et al., 2021; de Jesus et al., 2024; Sadeghi et al., 2023). These studies demonstrate the broad interest in nickel–terephthalate frameworks while also emphasizing that structural and performance claims must be linked to the specific synthesis and characterization of each material.

Solvothermal and hydrothermal methods remain widely used for MOF synthesis because elevated temperature and autogenous pressure can promote linker dissolution, deprotonation, metal–ligand coordination, nucleation, and crystal growth. Although conventional batch synthesis can produce highly crystalline materials, it often requires extended heating and cooling periods and combines mixing, thermal history, and reaction time within a single vessel. Changes in heat transfer, mixing, supersaturation, and solids handling may also influence batch-to-batch

consistency and complicate scale-up (Rubio-Martinez et al., 2017). These limitations do not make solvothermal synthesis unsuitable; rather, they provide a rationale for evaluating complementary synthesis routes.

Several process-intensified approaches have been investigated for MOF preparation methods, including microwave-assisted synthesis, sonochemical, mechanochemical, and flow synthesis (Główniak et al., 2021; Phan et al., 2023). Flow synthesis is particularly attractive because precursor streams can be delivered through a reactor under controlled flow and thermal conditions. Small reactor dimensions may improve heat transfer and mixing while allowing systematic adjustment of flow rate, temperature, and processing time. Flow-based production has been demonstrated for several MOF families using tubular, microfluidic, microwave-flow, and oscillatory baffled reactors (Bayliss et al., 2014; Dunne et al., 2016; Laybourn et al., 2019; Taddei et al., 2016). Nevertheless, flow synthesis is not universally superior, and issues such as reactor fouling, solids transport, solvent consumption, downstream washing, and operational stability remain important considerations (Bagi et al., 2021; Senthil Raja & Tsai, 2024). Flow rate and temperature can influence precursor conversion, linker deprotonation, coordination, nucleation, and crystal growth. Sonication may also assist dispersion and limit deposition in solids-forming systems, although its contribution to the present reactor configuration was not isolated (Ogura et al., 2023). Accordingly, this study evaluates the Ni-BDC formations by flow synthesis and examines the effects of flow rate and temperature on isolated yield and structural order.

Methods

Materials and Instrumentation

Nickel acetate tetrahydrate ($\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$) served the nickel source, and benzene-1,4-dicarboxylic acid (H_2BDC ; terephthalic acid) as the organic linker. *N,N*-Dimethylformamide (DMF), ethanol, and potassium bromide (KBr) were purchased from Sigma-Aldrich and used without further purification, while distilled water was used to prepare the precursor solutions. The apparatus comprised standard glassware, a magnetic stirrer and stir bar, an analytical balance, an oven, a Teflon-lined autoclave, two syringe pumps, and a coiled-tubing flow reactor immersed in an ultrasonic water bath. The reactor consisted of 1 m of PTFE tubing with an internal diameter of 0.8 mm and an outside diameter of 1.6 mm. Functional groups were identified by Fourier-transform infrared spectroscopy (FT-IR; Shimadzu

470) over $4000\text{--}400\text{ cm}^{-1}$ using the KBr pellet technique. Structural characterization was performed by powder X-ray diffraction (P-XRD) using a Rigaku MiniFlex diffractometer with Cu $K\alpha$ radiation ($\lambda_1 = 1.540593\text{ \AA}$; $\lambda_2 = 1.544414\text{ \AA}$).

Methodology

1. Solvothermal reference synthesis

Two precursor solutions were prepared. Solution A contained 5 mmol of $\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ in 18 mL of distilled water. Solution B contained 5 mmol of H_2BDC in 18 mL of DMF. Solution B was added slowly to solution A under magnetic stirring to obtain a homogeneous mixture and limit premature precipitation during mixing. The combined solution was transferred to a Teflon-lined autoclave and heated at $140\text{ }^\circ\text{C}$ for 12 h. After the reactor had cooled to room temperature, the solid product was washed three times: once with DMF and twice with ethanol. The solid was then filtered and dried at room temperature for 24 h. This batch product served as the solvothermal reference for comparison with the flow products.

2. Flow synthesis of Ni-BDC

The precursor compositions used for the solvothermal reference synthesis were retained for all flow experiments. Solutions A and B were loaded into separate syringes connected to independent syringe pumps. The two streams were pumped simultaneously and merged upstream of the reactor coil. In the baseline configuration, each stream was delivered at 0.05 mL min^{-1} , resulting in a combined flow rate of 0.10 mL min^{-1} . The reactor coil was immersed in the water bath of an ultrasonic cleaner. The bath was set to the desired temperature, after which pumping and sonication were initiated simultaneously. The effluent was collected as a suspension containing the Ni-BDC product. In this study, the processing time was defined as the time required to pump the entire precursor volume through the synthesis loop. At constant temperature, the processing time was varied to determine the optimum flow condition. Subsequently, the temperature was varied under the selected flow condition to evaluate its effect on Ni-BDC formation.

Table 1. Experimental matrix for the flow synthesis experiments

Series	Bath temperature (°C)	Total flow rate (mL min ⁻¹)	Processing time (min)
Flow-rate series	75	0.10	360
Flow-rate series	75	0.20	180
Flow-rate series	75	0.40	90
Temperature series	70	0.20	180
Temperature series	75	0.20	180
Temperature series	80	0.20	180

3. Product isolation and washing

The collected flow suspension was washed once with DMF and twice with ethanol, then filtered and dried at room temperature for 24 h. The same washing and drying procedures were applied to the flow products.

RESULTS AND DISCUSSION

Synthesis and Structural Confirmation of Ni-BDC

To establish a structural reference for evaluating the flow products, the solvothermal Ni-BDC sample was first examined by powder X-ray diffraction (PXRD). The resulting diffractogram was compared with the reported Ni-BDC reference pattern to assess phase formation and the structural characteristics of the material obtained under the batch-synthesis conditions.

As shown in Figure 1, the solvothermal product exhibited reflections at 2θ values of 9.22, 11.77, 12.22, 15.56, 18.30, 18.53, and 23.72° that match the CCDC 638866 reference. This agreement supports the formation of crystalline Ni-BDC and provides a baseline for assessing the phase formation and structural development of the flow products. The product showed an apparent crystallinity of 29.34%, calculated from the integrated area of the crystalline reflections relative to the total diffractogram area. Because this area-based value is sensitive to background subtraction, peak selection, and integration limits, it is used as a comparative parameter

rather than an absolute measure of crystallinity or phase purity.

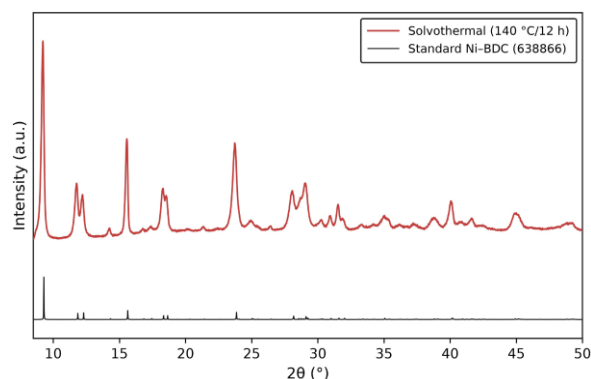


Figure 1. X-ray diffraction patterns of solvothermally synthesized Ni-BDC and the reported Ni-BDC reference

The FTIR spectrum of the solvothermally synthesized Ni-BDC was compared with that of the H₂BDC precursor to evaluate changes in the linker functional groups after framework formation. The H₂BDC precursor exhibited a prominent absorption minimum at approximately 1681 cm⁻¹, attributed to the C=O stretching vibration of the protonated carboxylic acid groups, together with a feature near 1424 cm⁻¹ associated with carboxyl-group C–O vibrations. In the solvothermal Ni-BDC product, the 1681 cm⁻¹ precursor feature was no longer dominant, while new bands appeared at approximately 1577 and 1382 cm⁻¹.

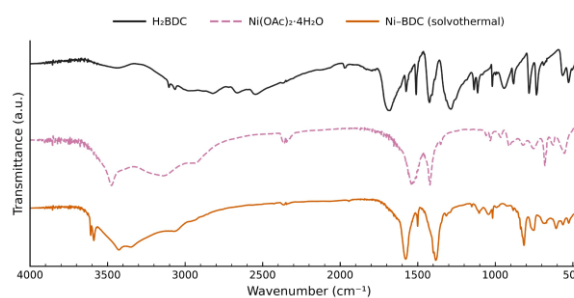


Figure 2. FTIR spectra of H₂BDC, nickel(II) acetate tetrahydrate, and solvothermally synthesized Ni-BDC

These bands can be assigned to the asymmetric and symmetric stretching vibrations of coordinated carboxylate groups, respectively. Their appearance is consistent with deprotonation of H₂BDC followed by coordination of the resulting BDC²⁻ linker to Ni²⁺ during formation of the Ni-BDC framework. A weak broad feature in the 3600–3200 cm⁻¹ region may be associated with O–H-containing guest species or

adsorbed moisture. Overall, the changes in the carboxyl-related bands support the formation of Ni-BDC through the solvothermal route. Comparable complementary use of PXRD and FTIR to evaluate framework preservation and chemical modification has also been reported for modified MOF-303 (Ariesta et al., 2026; Azhar et al., 2024; Hadjiivanov et al., 2021).

Effect of Flow Rate on Ni-BDC Formation

Flow synthesis was evaluated at 75 °C using combined flow rates of 0.1, 0.2, and 0.4 mL min⁻¹. Thermal energy supplied by the water bath was used to promote framework formation, while sonication was applied to mitigate the deposition and blockage of suspended solids within the tubing. Because the same total precursor volume was processed in each experiment, the processing time was not constant but was determined by the combined flow rate according to $t_{\text{processing}} = V_{\text{total}}/Q_{\text{total}}$. Thus, increasing the flow rate shortened the time required to process the precursor volume and reduced the nominal residence time within the reactor coil, whereas lower flow rates prolonged both. Consequently, the flowrate experiments simultaneously varied the flow rate, residence time, and thermal exposure of the reaction mixture.

The resulting XRD patterns exhibited reflections consistent with Ni-BDC, indicating at least partial formation of the target framework under flow conditions (Figure 3). Additional reflections observed in several samples may correspond to unreacted precursors, reaction intermediates, or secondary phases; definitive assignment would require independent phase identification. The observed dependence of phase development on flow conditions is consistent with previous continuous-flow studies showing that residence time and the local crystallization environment can strongly influence MOF formation (Bagi et al., 2021; He et al., 2021).

Increasing the flow rate from 0.1 to 0.4 mL min⁻¹ progressively reduced both isolated yield and apparent crystallinity (Table 2). At 0.1 mL min⁻¹, the product exhibited a yield of 60.76% and an apparent crystallinity of 18.54%; these values decreased to 42.78% and 11.39%, respectively, at 0.4 mL min⁻¹. The trend indicates that faster throughput provided less time for precursor conversion, framework assembly, and crystal growth under the selected reactor conditions. The broader reflections of the flow products may reflect smaller coherent diffraction domains, greater structural disorder, or both. Similar residence-time and crystallization-kinetic effects have

been reported for MOF formation in continuous-flow reactors (Bagi et al., 2021).

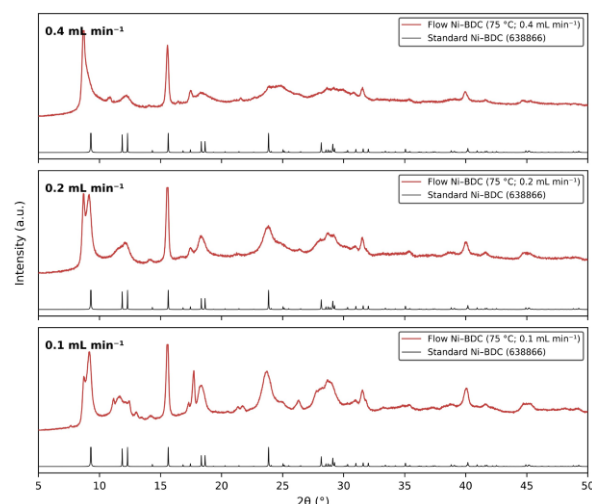


Figure 3. X-ray diffraction patterns of Ni-BDC synthesized at 75 °C using different flow rates.

Table 2. Effect of flow rate on Ni-BDC synthesis at 75 °C.

Method	Condition	Yield (%)	Apparent crystallinity (%)	Time (min)
Solvo thermal	140 °C, 12 h	62.30	29.34	720
Flow	75 °C, 0.1 mL min ⁻¹	60.76	18.54	360
Flow	75 °C, 0.2 mL min ⁻¹	56.12	14.26	180
Flow	75 °C, 0.4 mL min ⁻¹	42.78	11.39	90

Although 0.1 mL min⁻¹ produced the highest yield and apparent crystallinity in the flow-rate series, 0.2 mL min⁻¹ was selected as a practical operating condition because it reduced the processing time by half while maintaining a comparable yield. This flow rate was therefore used for the subsequent temperature-optimization experiments and comparison with the solvothermal reference. The processing times reported in this study correspond to the treatment of a fixed precursor volume and are distinct from the hydraulic residence time, which depends on the reactor volume and total volumetric flow rate. This distinction is

important when interpreting the effects of flow conditions on framework formation (Bagi et al., 2021)

Effect of Reaction Temperature

Temperature was subsequently varied at the selected flow rate of 0.2 mL min^{-1} . Raising the temperature from 70 to 80 °C markedly improved Ni-BDC formation (Figure 4). At 70 °C, the XRD pattern contained several reflections that did not match the target pattern, indicating incomplete reaction or additional phases; apparent crystallinity was therefore not calculated. The unmatched reflections decreased at 75 °C and were least prominent at 80 °C, showing that higher temperature favored target-phase formation within the investigated range. The temperature dependence is consistent with the role of thermal conditions in controlling precursor conversion and crystallization during continuous processing (He et al., 2021). Comparable effects of synthesis conditions on phase development, product purity, and isolated yield have been reported for hydrothermal sodalite synthesis (Amin et al., 2025).

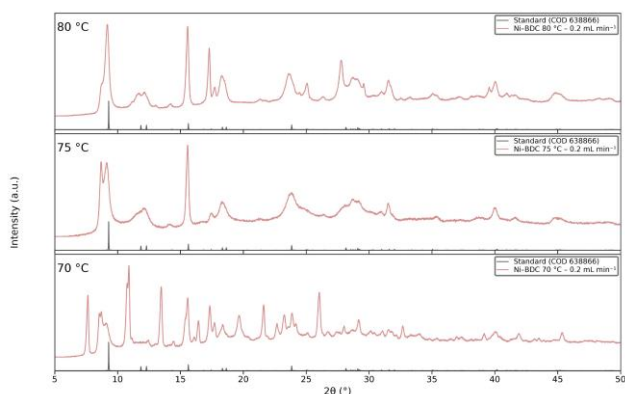


Figure 4. X-ray diffraction patterns of Ni-BDC synthesized at 0.2 mL min^{-1} at different temperatures.

The isolated solid yield increased from 29.22% at 70 °C to 56.12% at 75 °C and 72.66% at 80 °C. Apparent crystallinity was not determined for the sample synthesized at 70 °C because its PXRD pattern contained substantial contributions from unreacted precursors, reaction intermediates, and/or secondary phases. In addition, the Ni-BDC reflections were not sufficiently dominant to permit reliable assignment of the crystalline contribution. Therefore, the 29.22% value at 70 °C should be interpreted as the yield of recovered solid rather than as a confirmed yield of phase-pure Ni-BDC. For the samples evaluated at higher temperatures, the apparent crystallinity increased from 14.26% at 75 °C to 25.26% at 80 °C

(Table 3). This behaviour is consistent with increased precursor conversion and accelerated linker deprotonation, Ni-carboxylate coordination, nucleation, and crystal growth at elevated temperatures. Accordingly, among the conditions evaluated, 80 °C and 0.2 mL min^{-1} were identified as the best-performing conditions, providing the highest isolated solid yield and apparent crystallinity.

Table 3. Effect of temperature at 0.2 mL min^{-1} .

Temperature (°C)	Yield (%)	Apparent crystallinity (%)
70	29.22	Not determined
75	56.12	14.26
80	72.66	25.26

The qualitative reproducibility of the PXRD profile under the best-performing flow condition was evaluated using two independently synthesized Ni-BDC batches prepared at 80 °C and 0.2 mL min^{-1} (Figure 5). The two diffraction profiles showed close agreement in their overall patterns and principal reflection positions, with no evident changes in the dominant phase-related features. These results support the reproducibility of the selected flow synthesis procedure, which consistently produced Ni-BDC with

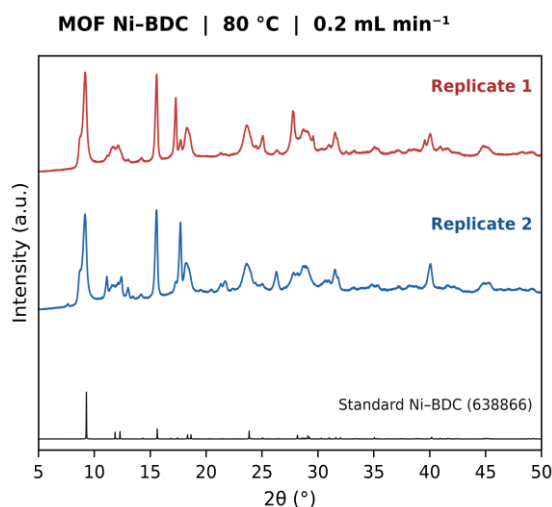


Figure 5. Stacked PXRD patterns from two independently synthesized Ni-BDC batches prepared at 80 °C and 0.2 mL min^{-1} , compared with the reported CCDC 638866 Ni-BDC reference pattern.

a comparable qualitative PXRD profile under the investigated conditions.

Comparison of Flow and Solvothermal Synthesis

The selected flow condition was compared with the conventional solvothermal method to evaluate differences in processing severity, yield, and structural order (Table 4). Solvothermal synthesis at 140 °C for 12 h afforded a yield of 62.30% and an apparent crystallinity of 29.34%. In comparison, flow synthesis at 80 °C and 0.2 mL min⁻¹ produced a higher yield of 72.66% after approximately 3 h, although its apparent crystallinity was slightly lower at 25.26%. Under the conditions investigated, flow synthesis reduced the operating temperature by 60 °C and the nominal processing time by approximately fourfold while increasing the isolated yield and maintaining comparable apparent structural order. This result is consistent with the broader rationale for continuous-flow MOF synthesis, in which controlled heat and mass transfer can reduce processing severity while retaining framework formation (Rubio-Martinez et al., 2017; Taddei et al., 2016).

Table 4. Comparison of solvothermal and selected flow synthesis conditions.

Method	Condition	Yield (%)	Apparent crystallinity (%)	Time (h)
Solvo thermal	140 °C, 12 h	62.30	29.34	12
Flow	80 °C, 0.2 mL min ⁻¹	72.66	25.26	3

CONCLUSIONS

This study demonstrated that Ni-BDC can be synthesized in the investigated sonicated coiled-tubing flow reactor at lower temperature and shorter nominal processing time than the solvothermal reference. At 75 °C, increasing the total flow rate from 0.10 to 0.40 mL min⁻¹ reduced both isolated yield and apparent crystallinity, indicating less favourable conditions for precursor conversion and framework production. The 0.20 mL min⁻¹ condition was therefore selected as a practical processing compromise. At this flow rate, increasing the temperature from 70 to 80 °C increased the yield from 29.22% to 72.66% and raised the apparent crystallinity at 80 °C to 25.26%, making 80 °C and 0.20 mL min⁻¹ the best-performing condition investigated. PXRD and FTIR results supported Ni-BDC formation, while two independently synthesized batches prepared under the best-performing condition

showed close qualitative agreement in their PXRD profiles. Relative to the solvothermal reference, the selected flow condition reduced the temperature by 60 °C and the nominal processing time from 12 h to approximately 3 h, while producing a higher yield and comparable apparent crystallinity. These results support the feasibility and qualitative reproducibility of the investigated flow synthesis procedure. Further work should quantify residence time, uncertainty, continuous-run stability, energy demand, and product morphology.

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