



Fabrication of hybrid coating material of polypropylene itaconate containing MOF-5 for CO₂ capture



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ABSTRACT

Novel hybrid coating material based on polypropylene itaconate (PPIA) and MOF-5 [Zn₄O(BDC)₃] (BDC: benzene-1,4-dicarboxylic acid) has been successfully prepared and characterized. Herein, the influence of certain percentage additions of MOF-5 (from 1, 3, 5, 10, 20, and 40 wt.%) into PPIA on its structure, morphology, density, and thermal stability were investigated. However, extensive damage was observed in MOF-5 structures according to PXRD, FTIR and nitrogen sorption isotherm analyses. The obtained materials were applied for CO₂ capture and monitored by the volumetric method. The higher percentage of MOF-5 incorporated into PPIA induced higher CO₂ adsorption. A significant increase of CO₂ capture was seen after the addition of 1–20 wt.% of MOF-5 into the PPIA, while an addition of 40 wt.% of MOF-5 indicated there is no more benefit in increasing the MOF-5 concentration in the composite and reached a CO₂ adsorption capacity of about 65 wt.% of the adsorption value of MOF-5 only. However, the incorporation of MOF-5 into PPIA caused the thermal stability of the hybrid coating material to be lower than the original PPIA coating and the MOF-5 itself. The texture of the film after addition of about 20 wt.% of MOF-5 changed from a smooth to a coarser and denser form. This study may open the way to investigating how PPIA coating containing MOF-5 could serve as a coating material with the function of CO₂ capture to contribute to reducing the greenhouse effect.

1. Introduction

The lack of non-renewable resources stimulates the discovery and use of renewable resources in the innovation of coating materials. Thus far, innovative research is being done in the manufacture of renewable raw materials in order to obtain biomass-based products [1,2]. As an example, one polymer that has been developed from renewable, biodegradable, inexpensive and non-toxic raw materials is polyester which can be synthesized through a condensation reaction of a difunctional monomer, such as diesters, diacids, and diol [3] or glycerol, for succinic acid-based polyester diols [4,5], its resin [6], polypropylene itaconate (PPIA) and its modification [7,8], polyester adipic acid-glycerol [4,9], etc.

The utilization of organic, inorganic and hybrid functionalized coating material, including based on polyester has been intensively investigated [10]. This kind of material has wide applications, such as anti-corrosion [11], energy harvesting [12], hydrolysis protection [13],

anti-fouling, and fire-retardation, among others [10]. Currently, fabrication of organic coatings embedded with gas-adsorbent materials has been an interesting research topic to be deeply explored [7,14,15]. Some porous materials have been applied for CO₂ gas adsorption and separation, such as zeolite [16,17], borane modified graphene [18,19], porous carbon [20], mesoporous silica [21], and metal-organic frameworks (MOFs) [22]. Thus, to study the incorporation of these materials into organic coating material will be challenging.

MOFs belong to a class of crystalline compounds consisting of metal ions or metal-oxide clusters coordinated to organic ligands as linkers to form three-dimensional porous structures [23]. A MOF itself has gas adsorption properties and can be used for the storage of hydrogen [24] or methane [25], and the capture of carbon dioxide [22], and it can function as a catalyst [26]. The pore size of a MOF typically can accommodate different types of gases, such as CO₂, O₂, N₂, and CH₄, and volatile organic compounds [27]. A MOF offers a promising alternative compared to zeolites and other materials, since a MOF has a tunable,

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high surface area, large pore size, selective sorption, separation ability, and outstanding gas storage capacity [28]. MOF-5 [$\text{Zn}_4\text{O}(\text{BDC})_3$] was first synthesized by Yaghi et al. [29], and it has been intensively studied for gas storage [30,31] and sorption application [32]. However, in application, MOFs require supporting material in order to form flexible features so they can be used in ways such as in a coating or in a mixed matrix membrane. Good flexibility could be obtained by using polymers such as PMMA [33] as well as polyesters [7] as renewable polymers as a host matrix. In addition, some MOFs based on zinc(II) like MOF-5 is also moisture sensitive [34], while polyesters have good flexibility and stability toward water and moisture [35] so they can be useful as reinforcing material in a MOF.

In this study, preparation of hybrid coating material using PPIA containing the simple MOF-5 [$\text{Zn}_4\text{O}(\text{BDC})_3$] will be discussed. The influence of certain percentage additions of MOF-5 into PPIA, and its chemical and physical properties, is investigated. The addition of MOF-5 into PPIA is expected to form a functional hybrid coating that also can be used as a CO_2 capture material. This study may also open the way to other uses of various gas adsorbent materials that can be incorporated into coating materials such as in a chamber, building or pipeline.

2. Materials and methods

2.1. Experimental materials

For our experiments, propane-1,3-diol (98%), hydroquinone (99%), $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ (98%), $\text{Zn}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ (98%), and the solvents (DMF, chloroform) were purchased from Merck (Germany). Itaconic acid (99%; Sigma-Aldrich, Germany), terephthalic acid (95%; ABCR, Germany), and all other chemicals were used without any further purification.

2.2. Synthesis of polypropylene itaconate (PPIA)

The PPIA was prepared according to a modified procedure described in previous literature [8]. In this study, zinc(II) acetate was used as a catalyst instead of titanium(IV) butoxide. Itaconic acid and propane-1,3-diol (in mol ratio 1:1.05) were placed in a 250 mL three neck halls reactor equipped with a water condenser under a nitrogen atmosphere. Hydroquinone (1% of mol total) and zinc(II) acetate (0.25% of acid mole) were added into the reactor and continuously stirred and heated ca 15 min to reach a temperature of 150 °C until all of the mixture was melted. Condensation reaction was continuously run at 170 °C for 100 min. Nitrogen was flown to push the water out of condenser. The vacuum was applied to the reactor gradually up to 5 mBar for about 5 h to remove the water content and obtain the bright yellow gel as a product.

2.3. Synthesis of MOF-5 [$\text{Zn}_4\text{O}(\text{BDC})_3$]

$[\text{Zn}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}]$ and terephthalic acid (mol ratio of 2:1) were dissolved in DMF separately. A solution containing 2.834 g of $[\text{Zn}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}]$ in 50 mL of DMF was added into the solution of terephthalic acid (0.900 g, in 50 mL DMF) dropwise under continuous stirring. The solution was refluxed at a temperature of 130 °C for 4 h until it formed a yellowish to white precipitate and then cooled to room temperature. The precipitate was filtered and then purified by washing it with chloroform three times. The product was dried at room temperature for 24 h and activated at 200 °C for 2 h in atmospheric air condition. Activated MOF-5 then was placed into a glass vial and kept in a desiccator. It was also exposed to air during preparing the hybrid films with PPIA and during measurements and characterizations.

2.4. Preparation of the PPIA-MOF-5 coating film

A hybrid film of PPIA-MOF-5 was made with a total weight of 2 g.

MOF-5 was suspended in ethanol and then sonicated for 1 h. Meanwhile, PPIA was dissolved in chloroform and gradually added into MOF-5 and further sonicated for 10 min for each addition of 10 wt.% of PPIA. The composition of MOF in the PPIA matrix was varied from 1, 3, 5, 10, 20 and 40 wt.% of 2 g total weight. The thin coating film was made by casting the film on a glass Plate 2×5 cm with the thickness of about 0.5 mm. The curing of the polymer matrix was done by mean heating of the film at 180 °C for 15 h and then cooled at room temperature. A thin PPIA film without MOF was also prepared in the same way.

2.5. Measurements and characterization

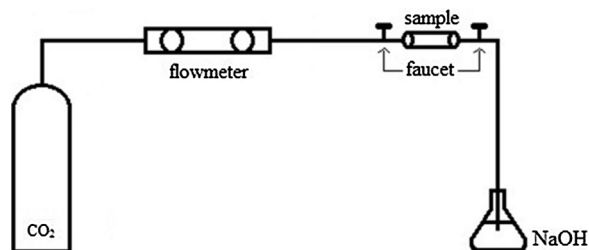
The dispersion of MOF-5 into PPIA was conducted in an ultrasonic bath (SKYMEN JP-040s, frequency of 40 kHz, power of 200 W). The surface morphologies of the PPIA and MOF-5@ PPIA were characterized by scanning electron microscopy (SEM, EVO MA 10). The structure of MOF-5 and PPIA-MOF-5 coatings was characterized by X-ray diffraction (XRD) using a Rigaku Miniflex 600 Benchtop XRD with Cu radiation (40 kV, 40 mA). The scanning speed and step size were 2 min^{-1} and 0.02° , respectively. The Fourier-transform infrared (FTIR) spectra were recorded using the Shimadzu type 8201 PC in KBR and in air condition. The nitrogen sorption isotherm of MOF-5 and the film were recorded on the micromimetic Nova 1200e at 77 K. Thermal stability of the materials was observed using the Shimadzu type DTG 60H Linseis, with a heating rate of 10 °C/min under a nitrogen atmosphere.

2.6. Adsorption of CO_2 gas

The adsorption of CO_2 gas was conducted according to modified literature procedure [36]. The system for CO_2 adsorption is depicted in Scheme 1.

CO_2 gas was flown into the chamber containing a 0.1 g sample with the flow rate of 50 cc/min, at a pressure of 5 bar for ca 10 min. CO_2 gas which passed through the chamber was collected into NaOH solution (100 mL, 0.1 M, temperature of $\pm 2^\circ\text{C}$). To ensure that the system was leakage free for desired period of time, complete conditioning process of the reactor prior being used was carried out, in which gas was constantly flown through the system for 1 h and the leakage was checked using bubble soap. The 60% efficiency was reached, and most importantly the trend of adsorption capacity of tested materials could be obtained.

Quantitative analysis of the CO_2 content in the solution was done using a titration method. Each 10 mL of collected solution was titrated with an HCl solution (0.1 M). Titration was done with phenolphthalein (PP) and methyl orange (MO) as indicators for the first and second equivalent points, respectively. The dissolved CO_2 in the NaOH solution was determined by the sum of the mole of HCl needed for total titration. The CO_2 adsorbed by MOF-5 and the hybrid films were calculated by the mol difference of the CO_2 without film (adsorbent) and the mole of the remaining CO_2 in the NaOH solution. To ensure the quantification of titration of CO_2 in the solution, the HCl (0.1 M) solution was standardized using sodium tetraborate solution (0.1 M). The



Scheme 1. The apparatus to test CO_2 adsorption.

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