DUT-5 modified Pd metal-nanoparticles: synthesis, chemical stability, and hydrogen sorption studies

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Abstract
The safety and effectiveness of hydrogen storage are important factors in supporting the utilization of hydrogen as fuel. One of the MOFs materials might be potentially used for hydrogen storage is MOF based on Al\(^{3+}\) including ([Al(OH)BPDC]) (BPDC: 4,4′-biphenyl dicarboxylic acid) which well known as Dresden University of Technology (DUT-5). This study aims to investigate the influence of Pd metal nanoparticles embedded into DUT-5 with a variation: 1 and 5 wt% toward the structural feature and hydrogen sorption properties. According to XRD and FTIR spectroscopy, nitrogen sorption isotherm and SEM-EDX analysis, the basic structure of DUT-5 were partially decomposed after the impregnation process. TEM analysis showed Pd nanoparticles were well dispersed into DUT-5. Nitrogen sorption isotherm revealed that Pd loading into DUT-5 lead to decrease surface area and pore volume of DUT-5 up to 85.82%. Based on thermogravimetry analysis, thermal stability of the material did not change after impregnation. Hydrogen sorption measurement revealed the presence of Pd is not able to increase hydrogen storage capacity of DUT-5 due to the framework destruction, however, in Pd/DUT-5 materials, the increasing of Pd concentration can increase the hydrogen storage capacity which might be due to a spillover effect.

1. Introduction
Research on renewable energy has become a major focus in this era. The increasing demand for fossil fuels for human activities such as industries, transportation, and household lead to depletion and scarcity crisis. In addition, the use of fossil based fuels as an energy source is not environmentally friendly due to the emission increase of atmospheric pollutants such as CO\(_2\), CH\(_4\), NO\(_x\) and SO\(_x\) gases [1]. According to the National Oceanic and Atmospheric Administration, the global temperature for March 2018 was 0.83 °C above the average global temperature of the 20th century [2]. In 2017, global CO\(_2\) rose by 1.4% from 32.04 Gt to 32.5 Gt, an increase of 460 million ton (Mt) CO\(_2\) emission is equivalent to the emission from 170 million additional cars [3], consequently, environmentally friendly alternative energies are urgently needed. Hydrogen is one of the options of a clean energy carrier because the combustion product only produces water as its by-product [4]. In addition, hydrogen energy has the highest energy density compared to fossil fuels and other alternative fuels, therefore hydrogen energy is a promising source of renewable energy to replace fossil-based fuels especially as a source of energy for transportation [5].

The safety and effectiveness of hydrogen storage are important factors when used in transportation. There are many ways to store hydrogen such as storing it as compressed gas (in pressurized tanks), liquefied hydrogen
Aluminum nitrate nonahydrate (in cryogenic tanks), and adsorption to solid materials [6]. The storage of hydrogen as pressurized gas and cryogenic liquid requires a bulky tank and has a high risk because both ways require high operational pressure. As an alternative, the utilization of solid material is one of the safest and effective ways to stored hydrogen [7–10]. US Department of Energy (DOE) has set the target for onboard hydrogen storage system: 4.5 wt% (30 g L⁻¹) in 2020 and 5.5 wt% (40 g L⁻¹) in 2025 [11].

Metal–Organic Frameworks (MOFs) are multifunctional materials with unique characteristics such as highly flexible in structural design with various pores and coordination sites, high crystallinity, and rich in chemical and physical properties [12]. This kind of material formed by the coordinative bond of the organic linker to metal ions or metal oxide clusters. MOFs constructed from highly electronegative and low-density metal ions such as Na⁺, Li⁺, K⁺, Mg²⁺, and Al³⁺, have potential as hydrogen storage material [13] such as [Al(OH)BDHC], [Mg₂(NDC)] [14], and [Na₂(BDA)₂CH₂OH(H₂O)] [15], [Al(OH)(BPD)] (BPD = biphenyl-4,4′-dicarboxylic acid) or known as Dresden University of Technology-5 (DUT-5) is Al³⁺ based MOF with high surface area (2335 m² g⁻¹) and pore volume (0.81 cm³ g⁻¹) that also has potential as the hydrogen storage material. According to the previous study, DUT-5 has a hydrogen storage capacity of 3.3 wt% at 40 bar and −196 °C [16].

Mostly MOFs exhibited higher storage capacity at −196 °C but then decrease at ambient temperature. This condition can be enhanced by noble metal nanoparticle impregnation. The impregnation of noble metals such as Pd and Pt can reach good interaction with hydrogen gas and cause spillover effects [17]. The increasing of hydrogen storage in Pd@MOF has been reported by Zlotea et al [18] who modified Pd against MIL-100 (Al). The result showed an increase in hydrogen adsorption at 25 °C and 4 MPa two times higher. Sabo et al [19] had modified Pd against MOF-5 and showed an increase in hydrogen adsorption from 1.32 wt% into 1.86 wt% at 1 bar and −196 °C. Palladium modification over DUT-5 so far has not been investigated. Herein, we study the effect of Pd metal nanoparticles loading toward its structural feature and hydrogen sorption properties.

2. Materials and methods

2.1. Materials
Aluminum nitrate nonahydrate (Al(NO₃)₃·9H₂O) 98%, 4,4′-biphenyl dicarboxylic acid 97%, Palladium(II) acetylacetonate (Pd(acac)₂) 99%, and Palladium chloride 99.9% were purchased from Sigma Aldrich, Germany. The hydrochloric acid (HCl) 37%, Chloroform, and N-N dimethylformamide (DMF) 98% were commercially obtained from Merck, Germany. In all the cases, hydrogen gas of 5.0 purity (99.999 %) was used for the adsorption measurement and the distilled water was supplied from the Chemistry Laboratory of Sebelas Maret University.

2.2. Method

2.2.1. Synthesis of DUT-5
DUT-5 material was synthesized using the solvothermal method based on the reported procedure [16]. In a typical synthesis, 4,4′-biphenyl dicarboxylic acid (0.26 g, 1.2 mmol) and Al(NO₃)₃·9H₂O (0.52 g, 1.4 mmol) were dissolved in 30 ml DMF and placed the solution into a 250 ml Teflon-lined stainless steel autoclave. The autoclave was heated at 120 °C for 24 h in oven. Then the autoclave was cooled down to room temperature naturally. The suspension was separated by centrifugation, washed with DMF three times and dried at ambient temperature with a yield of 94.8%.

2.2.2. Synthesis of Pd/DUT-5
In this study, the impregnation of Pd nanoparticles is carried out using the method reported by Zlotea et al which embedded Pd nanoparticles into MIL-100(Al) using Palladium chloride as precursor [18]. The addition of Pd metal is 5% of 2 g of DUT-5. PdCl₂ salt was dissolved in 20 ml of 10% HCl solution to form a homogeneous brown solution, then DUT-5 was added into the solution and stirred for 3 h. The mixture was dried at 57 °C and then reduced in the flow of H₂ gas at 150 °C for 2 h.

The synthesis of Pd/DUT-5 was also carried out using Palladium acetylacetonate (Pd(acac)₂) as a precursor salt [19]. Pd(acac)₂ was used as Pd source doped into DUT-5 with concentration 1 and 5 wt% of 2 g DUT-5.

Pd(acac)₂ was dissolved in 5 ml chloroform, then added to DUT-5 drop by drop under continuous stirring forming an orange paste. The paste was dried overnight at ambient temperature to yield slightly yellow powder then placed in stainless steel tube and heated in H₂ flow at 200 °C for 1 h. After reduction procedures, the powder was washed with chloroform and dried at ambient temperature.

2.3. Materials characterization
The structure and crystallinity of the synthesized material were analyzed by XRD instrument (Rigaku Miniflex 600 Benchtop) using Cu-Kα radiation (λ = 1.5406 Å) at 30 kV and 20 mA with a scan rate 1° min⁻¹. The
recorded specific peak intensities and 2θ values were further identified by a computer database system (JPCDS). Infrared (IR) spectra were recorded by Shimadzu IR Prestige-21 in KBr pellets between 400–4000 cm\(^{-1}\). The morphology and particle size of material were carried out by SEM (FEI type Inspect S50) and TEM.

Thermogravimetric analysis was carried out using a STA Linseis PT-1600 under nitrogen atmosphere with a heating rate of 10 °C min\(^{-1}\) in the range from 20 °C–600 °C. The surface area of MOFs materials were characterized by N\(_2\) physisorption at \(-196 °C\) on a volumetric sorption analyzer (BEL Japan, BELSORP-mini). The total pore volume was elucidated from the data point at \(p/p_0 = 0.99\).

2.4. Hydrogen sorption measurement
Hydrogen adsorption isotherms were measured with an automatic volumetric instrument Quantachrome (iSorbHP1) to perform high-pressure measurements up to 50 bar. About 300 mg of the sample was located in the sample holder, where it was degassed at 150 °C for 6 h under vacuum, in order to prepare the sample for the measurement. The hydrogen adsorption measurement was performed at 25 °C and 50 °C up to 50 bar. The instrument was regularly tested for leaks, and the reference volume (<19 cm\(^3\)) was determined by the calibrated volume. The volume on the sample side was determined by helium gas before measurement with a range between 19.1 and 25.2 cm\(^3\). The temperature of reference was set at 45 °C, while the sample was set at 25 °C and 50 °C which is controlled by a thermostat.

3. Results and discussion

3.1. Materials characterization
XRD pattern of synthesized DUT-5 (figure 1) shows four main characteristic peaks at 2θ: 6.02° (101), 12.06° (202), 14.16° (011), and 18.12° (303) which confirmed the similarity with simulated pattern CCDC No. 69179 and did not show peaks characteristic of precursor compound which indicate DUT-5 material was perfectly performed. The compatibility of DUT-5 diffractogram with standard CCDC No.69179 can be analyzed quantitatively by refinement using Le Bail method (figure S1 is available online at stacks.iop.org/MRX/6/1250d4/mmedia). Based on the refinement result, the value of % Rwp and %Rp are less than 10% and GOF value is less than 4% which indicated the synthesized material has high suitability with the standard (table S1).

Based on the result of XRD analysis (shown in figure 2), the impregnation of Pd nanoparticles into DUT-5 using this method was not successful because the DUT-5 framework has completely decomposed into ligand which indicated by the appearance of ligand characteristic peaks instead of characteristic peaks of DUT-5. This is might occur because of the use of strong acid during the impregnation process. It is different from the impregnation of Pd nanoparticles into MIL-100 (Al) which only decreases crystallinity slightly, so it might be assumed that DUT-5 material is not chemically stable against strong acid compared to MIL-100(Al). In addition, MIL-100(Al) which is constructed of Al\(^{3+}\) metal ions and H\(_2\)BTC ligand has a more rigid structure, so it has a more stable framework.

Figure 3 shows the XRD diffractogram of DUT-5 after embedding of Pd nanoparticles using Pd(acac)\(_2\) precursor after reduction. The presence of Pd nanoparticles on DUT-5 can be observed from the diffraction peak of Pd/DUT-5 which shows two additional peaks at 2θ: 40.2° (111) and 46.5° (200). This data are in line with the results published by Zlotea et al who studied Pd nanoparticles embedded into MIL-100 (Al) [18]. The success of Pd impregnation can be analyzed quantitatively by refinement using Le Bail method (figure S2). In this
analysis was used 3 prediction phase that is DUT-5, H$_2$BPDC ligand, and Pd metal. Based on the value of \%Rp, Rwp, and GOF it can be concluded that the synthesized material has high suitability with the prediction phase (table S2). However, based on the diffractogram of Pd/DUT-5, impregnation of Pd metal reduce the crystallinity significantly of DUT-5 indicates by the decrease peak intensity to 1/50 smaller due to framework decomposition. This phenomenon also occurs in MOF-5 exposed to ammonia gas. After the exposure process, there was a significant decrease in intensity from the highest characteristic peak of MOF-5 which indicates that the material structure of MOF-5 has been decomposed [20]. Moreover, the peak intensity of Pd is equivalent to concentration and the increase of Pd metals concentration will decrease the crystallinity of DUT-5. Framework decomposition of DUT-5 can be caused by a flexible framework due to linker rotation, so the presence of guest molecules can easily interfere with framework stability.

Figure 2. Diffractogram of synthesized 5\% Pd/DUT-5 using PdCl$_2$ as a precursor compared to unmodified DUT-5, standard pattern of Pd and H$_2$BPDC ligand.

Figure 3. Diffractogram of Pd/DUT-5 compared to unmodified DUT-5.
The nitrogen sorption isotherms measurement revealed that the DUT-5 material has isotherm curve type I and IV (figure 4), characteristic for microporous material and small mesopores. Based on nitrogen sorption isotherm and single x-ray data, DUT-5 has a pore diameter of about 11.9 Å and 19.2 Å. The calculation using the Brunauer-Emmett-Teller (BET) method shows the specific surface area of DUT-5 is 1467 m² g⁻¹. Using the Barret-Joyner-Halenda (BJH) method the micropore volume of DUT-5 is 0.71 cm³ g⁻¹. The specific surface area and pore volume of synthesized DUT-5 have a similar value with DUT-5 synthesized by Senkovska et al [16].

The N₂ adsorption/desorption isotherms of DUT-5 and Pd/DUT-5 at −196 °C show a mixture of type I and IV curves (figure 4, table 1) [21]. For DUT-5, the steep increase in the adsorbed volume at low relative pressure relates to microporosity, and the slight secondary uptake points to the existence of mesopores. The adsorption/desorption hysteresis loop for Pd/DUT-5 denotes the existence of developed mesoporosity, while the final almost vertical tail at relative pressure near \( p/p₀ = 1.0 \) indicates the presence of macroporosity. The textural parameters (table 1) show that the specific surface areas calculated by the BET method are 1480 to 209 m² g⁻¹ for DUT-5 and Pd/DUT-5, respectively. The total pore volumes, as calculated from the adsorption value at relative pressure \( p/p₀ = 0.99 \), are 0.71 and 0.39 cm³ g⁻¹ for DUT-5 and Pd/DUT-5, respectively. The decrease of specific surface area, micropore, and total pore volumes indicate that the insertion of Pd nanoparticles modifies the pore structure of the pristine DUT-5 [18].

Moreover, the significant decrease of the specific surface area can also be assumed as a result of partial framework decomposition which is supported by the XRD analysis result. Based on table 1, the greater concentration of embedded Pd nanoparticles the surface area will tend to decrease.

The SEM images (figure 5) show the synthesized material DUT-5 has round-shaped with average crystallite size around 0.72 ± 0.19 μm which similar to morphology reported in previous literature [16]. In figure 5 also can be seen that the morphology of material DUT-5 became irregular after doped with Pd metals nanoparticles. The embedded of Pd nanoparticles also affect the particle size of DUT-5 to be smaller than the initial material, this is related to the result of XRD and SAA analysis previously discussed that there is partial framework decomposition of DUT-5. 1 and 5% Pd/DUT-5 have average size particle respectively are 0.42 ± 0.09 μm, and 0.48 ± 0.10 μm. The concentration of Pd loaded into DUT-5 can be measured by SEM-EDX analysis and show EDX spectra which contained peaks of Al, C, O, and Pd atoms. Table 2 shows the concentration of Pd in Pd/DUT-5 was not similar to the theoretical calculation. It may because Pd lost during filtering or washing or replacing the Al³⁺ partially in the frameworks.

The bright-field transmission electron microscopy images (figure 6) show black parts that are attributed to the Pd nanoparticles. There are two different parts in figure 6, the first is Pd nanoparticles form black dots, which means Pd nanoparticles are well spread onto the MOF crystals and the other one is a big black part where Pd

![Figure 4. N₂ adsorption/desorption isotherms at −196 °C of the DUT-5 and Pd/DUT-5 samples.](image)

### Table 1. Surface area and pore volume of DUT-5 and Pd/DUT-5.

| Material      | BET Surface area (m² g⁻¹) | Pore volume (cm³ g⁻¹) | Average pore size (nm) |
|---------------|---------------------------|-----------------------|------------------------|
| DUT-5         | 1467                      | 0.71                  | 1.19                   |
| 1% Pd/DUT-5   | 208                       | 0.36                  | 6.96                   |
| 5% Pd/DUT-5   | 204                       | 0.39                  | 7.57                   |
agglomerates. According to Talik et al Pd has particle diameter about 0.14 nm [22], so particles Pd is small enough to enter the pore of material DUT-5 with a diameter 11.9 Å but based on TEM images Pd particles can agglomerate which indicate few Pd particles will laying on the DUT-5 surface. Pd particles located within the porous will contribute to the change of surface area and pore volume of the host material. This statement is in line with SAA analysis results as explained previously, where material Pd/DUT-5 has a lower specific area and pore volume than DUT-5.

IR spectra of synthesized material DUT-5 (figure 7) show a vibrational band at 3431 cm\(^{-1}\) corresponds to O–H vibration that attached to ion metal Al\(^{3+}\). The significant shift of C=O vibration of H\(_2\)BPDC from 1683 cm\(^{-1}\) to 1601 cm\(^{-1}\) indicates the deprotonation of ligand and coordination between the ligand and Al\(^{3+}\) metal ion to form DUT-5 has occurred. In addition, the sharp peak at 1433 cm\(^{-1}\) is characteristic of the C–O vibration of the carboxylate group. Other bands that appear on IR spectra are C=C aromatic vibrational bands at 1502–1546 cm\(^{-1}\) and of Al–O vibrational bands located in 774 cm\(^{-1}\). The IR spectra of synthesized material DUT-5 has been confirmed with a previous report [13]. In contrast to the result of XRD analysis, FTIR analysis shows the structure of DUT-5 was remaining stable after doped Pd nanoparticles which indicated the absence of significant shift of C=O, O–H and C–O vibrations (figure 7) due to inability FTIR analysis to detect framework defect at MOF, this is because framework defect did not cause damage to the overall bond so the functional groups were still identified.
Based on thermogravimetric analysis (figure 8), the decomposition of DUT-5 occurred through two steps as reported by Senkovska et al.\textsuperscript{16}. The first mass loss of 30.43\% occurred at 25 °C–160 °C attributed to the removal of two water molecules H₂O and 1.2 molecule DMF which lied on the surface or remaining on the pore, the last step of decomposition occurred at 483 °C with mass loss significantly of 43.58\% correspond to decomposition of the linker which indicates the framework starts to collapse. At 615 °C, the mass was stable until 700 °C which indicates DUT-5 has decomposed completely to form metal oxide Al₂O₃ with a mass residue of 17.91\%.

Figure 9 shows a comparison of the thermogram profile between DUT-5 and Pd/DUT-5. The decomposition of Pd/DUT-5 occurred through three steps. The first step was might be the removal of DMF as coordinating solvent, the second step was free ligand decomposition resulting from the framework decomposition during the impregnation process, and the last step was the decomposition framework of DUT-5. In addition, the amount of mass loss at the step of framework decomposition decrease along with the increasing Pd concentration. According to Ren et al.\textsuperscript{23} if the amount of mass loss is lower than the calculation mass lost is theoretically, it is an indication of defect due to loss of ligand from the framework. However, the embedded Pd nanoparticles into DUT-5 did not change the thermal stability of DUT-5. In the thermogravimetric analysis of 1,
and 5% Pd/DUT-5 were obtained residue from analysis sequentially by 18.59, 23.89, and 28.02% which is estimated as a mixture of Al₂O₃ and PdO, so the amount of residue is increased along with the increasing Pd concentration.

3.2. Hydrogen sorption capacity
The hydrogen adsorption isotherms of all samples were measured at 25 and 50 °C up to 50 bar are shown in table 3 and figure 10, respectively. Table 3 shows that the quantity of hydrogen adsorbed in DUT-5 synthesized is significantly different from DUT-5 synthesized by Senkovska et al [13] due to the differences in adsorption temperature. This proves the statement of Langmi et al [7] which states that MOF will adsorb hydrogen optimally at −196 °C because MOF will reach saturation capacity [24]. At room temperature, H₂ and MOF will form a weak bond with the interaction energy of 4–7 kJ mol⁻¹.

Hydrogen uptake on different samples under conditions of 50 bar pressure and two different temperature 25 and 50 °C are shown in figure 10. As indicated in figure 10, a local adsorption maximum of approximately 0.20 wt% is obtained with the unmodified sample (DUT-5) at 25 °C. Further, it is noticed that the hydrogen uptake capacity inversely decreases as the temperature increase for the unmodified sample, typically for hydrogen adsorption by physisorption. The lower hydrogen sorption properties at ambient temperature can be attributed to the lower specific surface area of the Pd-doped samples and that has the key role in the hydrogen sorption.

The hydrogen uptake capacities are normalized by their corresponding specific surface areas (listed in table 3). It is noted that each square meter of the surface area contributes to approximately 1.36 10⁻³ g and 0.97 10⁻³ mg of hydrogen uptake at 25 and 50 °C, respectively, these uptake capacities appears affected by the amount of Pd present in the sample at 50 °C. Since chemisorbed hydrogen molecules are most likely to occur at
the metal surface (metal hydride), the consistent normalized sorption capacity of the DUT-5 and 1% Pd/DUT-5 might imply that the hydrogen molecules are mainly adsorbed by the process of physisorption. The great hydrogen sorption at 50 °C for 5% Pd/DUT-5 can be attributed to a corresponding spillover mechanism, in which Pd metals act as catalysts for molecular hydrogen dissociation. The hydrogen adsorption characteristics with an initial rapid adsorption phase at low pressure followed by slower adsorption after the pressure had been raised by a few bar (<10 bar). At low pressures, the H/Pd atomic ratios in chemically doped palladium samples were higher than in bulk palladium, as a consequence of the strong interaction between the metallic Pd and H₂, indicated the formation of PdHₓ [15, 22].

This study clearly revealed that the lower size of nanoparticles (Pd) improved hydrogen storage efficiency. In the sample with the largest cluster, noted as Pd-DUT-5, the uptake process was the lowest even lower than unmodified DUT-5. The reason for this is probably lower diffusion hydrogen from the metal nanoparticles to the microporous of DUT-5. This uptake is less effective in comparison with unmodified DUT-5 due to hindered access to the surface and less content of MOF.

Anson et al suggested that this might be due to the spillover of hydrogen [25]. However, at higher pressures and room temperature, this effect of spillover was considered to be very low. In modified material (5% Pd/DUT-5), hydrogen sorption is achieved through the synergistic effect of two mechanisms: physisorption and chemisorption. This effect was previously studied with the different content of Pd particles deposited on the surface of COF-102 and Zr-BTC. The presented data have shown that hydrogen storage capacity changes with the metal content and that the higher Pd content is more favorable for hydrogen storage [26, 27].

4. Conclusions

Modification of DUT-5 material using Pd nanoparticles was successfully prepared. However, the modification using acid treatment leads to structural destruction. The embedded Pd nanoparticles against DUT-5 causes partial decomposition of the pristine structure of DUT-5 which lead to low hydrogen adsorption capacity, however in Pd/DUT-5 material the increasing Pd concentration cause an increase of hydrogen adsorption capacity which might be due to a spillover effect. The measurement of hydrogen adsorption capacity at 50 °C has
a lower value than at room temperature due to the decrease of hydrogen adsorption capability through physisorption.

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