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Enhanced sorption performance of MgH$_2$ doped with reduced metal-organic framework of Ni-Co-MOF-74

Atikah Kadri, Xiangdong Yao

Abstract: *Improvement has been made in hydrogen storage performance particularly in MgH$_2$ system prepared via high energy ball milling technique where bi-metal in the metal organic framework of Ni-Co-MOF-74 was introduced as catalyst. Better catalytic effect was recorded when pre-reduced Ni-Co-MOF-74 is used as catalyst for both hydrogen absorption and desorption. An apparent amount of reduced metal species in the MgH$_2$ system containing bi-metal in metal organic framework promotes more active sites which eventually improved the sorption properties and shifted the thermodynamic stability of the composite. At 280°C, hydrogen desorption was 4.7 wt% in a period of 30 minutes whereas at 200°C a remarkable hydrogen amount of 6.3 wt% was stored in 2 minutes. It is believed that the distinctive coordinate of the metal in the pre-reduced metal organic framework improved the dispersion of the catalyst thus enriched the sorption properties.*

Index Terms: *Catalysts, Hydrogen storage, Magnesium hydride, Mechanical milling, Metal organic framework.*

I. INTRODUCTION

The development of a safe and an efficient mode for storing hydrogen remains a key challenge in the realization of a widespread application in mobile devices. The most mature of the solid-state hydrogen storage options is that of the metal hydrides. Being light metals, magnesium hydride (MgH$_2$) and its alloys have been most widely investigated for hydrogen storage applications. MgH$_2$ is a highly potential hydrogen encapsulating material due to its relatively high gravimetric and volumetric capacities of 7.6 wt% and 110 kg/m^3 of hydrogen by volume respectively as well as outstanding reversibility and abundantly available thus low in cost. However, the potential of MgH$_2$ in vehicular application remained as a challenge particularly in the impractical desorption temperature beyond 300°C as well as its sluggish hydrogenation/dehydrogenation kinetics. The fact that bulk MgH$_2$ possess a very strong affinity between magnesium and hydrogen, it requires a decomposition enthalpy of about 75 kJ/mol H$_2$ which corresponds to desorption temperature of 300 °C at 1 bar [1].

There have been considerable research efforts reported in the literature on improving MgH$_2$ hydrogen storage properties, for example to reduce particle size and add additives to serve as active sites. Additives that have been investigated include transition metals [2], [3], oxides [4], [5] and metal halides [6]. Intermetallic compounds of transition metals are also considered as catalysts or destabilizing materials for magnesium (hydride). Attempts have been made to destabilize magnesium hydride by using intermetallic compounds possessing low heat of hydride formation. However, no significant change in the thermodynamics was achieved for these composites [7], [8].

It is well accepted that the use of nanocatalysts improves absorption and desorption performance in MgH$_2$ [9]. Nanoscale bimetallic of chromium and vanadium were also used as catalysts showing significant room-temperature hydrogen uptake over 10 cycles at absorption pressure as low as 2 bar [10]. Both nickel and cobalt are known catalysts that promote the hydrogen sorption kinetics in magnesium [11], although a binary catalyst as such in magnesium system has not received much research attention.

Recently, substantial progress has been made in utilizing metal-organic frameworks (MOFs) as a hydrogen storage material via physisorption [12]. Owing to the hybrid architecture it is expected to create a large pore volume and surface area to promote the capture and/or regeneration of hydrogen. Having a large density of active sites in which the constitutional metal node has free or exchangeable coordination positions and the high porosity draw a large potential for MOFs in solid catalysts application [13], [14]. The introduction of MOFs as an additive in the MgH$_2$ system is motivated by the fact that the unique structure of MOFs (which contain metals transition metals) is known to provide a catalytic effect in MgH$_2$ storage systems. Catalytic activity in the MgH$_2$ composite is expected to be enhanced by having finely dispersed bimetal catalysts originated from the MOF. To the best of our knowledge, no studies devoted to the sorption properties of MgH$_2$ have tackled the use of reduced MOFs as additives. In the present study, bi-metal MOFs of Nickel and Cobalt are prepared and introduced to MgH$_2$ as additive. Particular attention is given to the effect of reduced Ni-Co-MOF-74 on sorption performance in MgH$_2$ system.

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II. EXPERIMENTAL

A. Synthesis of Ni-Co-MOF-74

The preparation of Ni-Co-MOF-74, denoted NiCof, was based on a literature procedure [15]. Typically, a mixture of 1.5 g of $Ni(NO_3)_2.6H_2O$, 1.5 g of $Co(NO_3)_2.6H_2O$ and 1.0 g of 2,5 dihydroxyterephthalic acid (DHTA) was dissolved in 140 ml of dimethylformamide (DMF), 140 ml of ethanol and 140 ml of water under stirring. The mixture was heated in teflon-lined steel autoclave at 100 ^{o}C for 66 hours. After cooling to room temperature, the mother liquor was decanted and the products were washed with methanol before being immersed in methanol. The methanol solvent was decanted and replaced once each day over the next three days. The resulting solid was heated under vacuum at 150°C. After 24 hours the powder was cooled to room temperature before being kept in an argon-filled glove box (MBraun) to protect against air and moisture. The preparation of reduced Ni-Co-MOF-74 was performed by using hydrogen as the reduction agent at 300 °C and atmospheric pressure. Two reduction times of 30 minutes and 5 hours were used to prepare 30mNiCof and 5hNiCof respectively in order to investigate the reduction effect of the two extreme of reduction times.

B. Synthesis of MgH$_2$ storage composite

The MgH$_2$ composites were prepared by pre-milling MgH$_2$ (hydrogen storage grade - purchased from Sigma-Aldrich) in a sealed vial of high energy SPEX 8000 vibration ball mill with argon as the working ambience for 2 hours. Following this, 5 wt% of NiCof, 30mNiCof and 5hNiCof were introduced and milled for another 2 hours. The composites are denoted MNiCof, M30m-NiCof and M5h-NiCof, respectively. The ball to powder ratio was kept at 40:1 in all sample preparations. For comparison purposes MgH$_2$ was milled for a similar total milling time of 4 hours, denoted M4. In order to avoid oxidation, all sample handling of the storage material was performed in an Argon-filled glove box (MBraun), in which the water and oxygen level were kept below 1 ppm.

C. Characterization

The microstructure of the samples was characterized by X-ray diffraction (XRD, Rigaku) with Co Kα radiation at a scanning rate of 2°/min. The morphology and composition of the samples were further analyzed by employing a transmission electron microscope (TEM, FEI F20) equipped with energy dispersive X-ray spectrometry (EDS). The samples were prepared by dipping a TEM substrate (consisting of a holey carbon film on a copper grid) in pre-sonicated octane sample mixture. Kratos Axis ULTRA X-ray photoelectron spectrometer with a 165 mm hemispherical electron energy analyzer is used to generate XPS data where the survey scans were taken at 160 eV. As the amount of nickel and cobalt in the composition were too small, the multiplex narrow scans of Ni 2p, Co 2p, O1s and C 1s were taken at pass energy of 80 eV with at least 6 sweeps. Survey scans were carried out over a 1200 – 0 eV binding energy range with 1.0 eV steps and a dwell time of 100ms. Narrow scans were run with 0.1eV steps and 250 ms dwell time. Base pressure in the analysis chamber was 1.0 x 10^{-9} torr and during sample analysis 1.0 x 10^{-8} torr.

All hydrogenation measurements of the milled powders were evaluated using an automated Sieverts apparatus (Suzuki Shokan PCT H$_2$ absorption rig). The absorption and desorption tests were performed under an initial pressure of 2.0 MPa and 1 KPa at various temperatures respectively after a complete dehydrogenation and hydrogenation cycle. Non-isothermal hydrogen desorption of the samples was investigated by temperature programmed desorption (TPD) equipped with a thermal conduction detector. Argon was used as the carrier gas and all samples were subjected to a 5 °C/min heating rate with 50 ml/min flowing rate during the analysis.

III. RESULT AND DISCUSSION

A. Characterization of prepared Ni-Co-MOF-74

A typical 3-dimensional scheme of Ni-Co-MOF-74 is shown in Fig. 1a. With Ni and Co coordinated in the MOF structure it is expected that this will improve the dispersion of both Ni and Co species in the composites. XRD patterns of the as-prepared NiCof and simulated NiCof are shown in Fig. 1b. Both patterns show similar peaks thus providing strong evidence that NiCof was correctly synthesized.
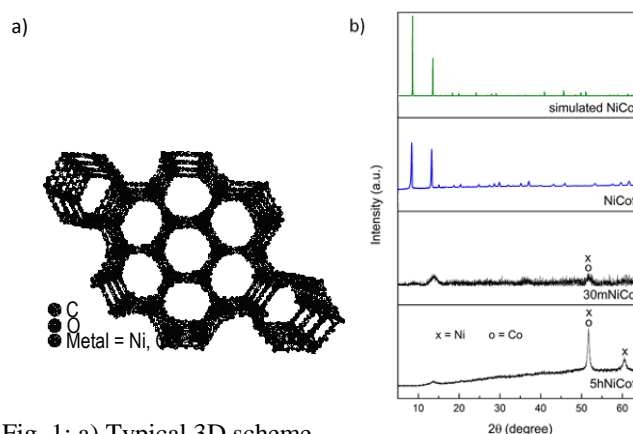

Fig. 1: a) Typical 3D scheme of Ni-Co-MOF-74 and b) XRD profile for simulated NiCof, NiCof, 30mNiCof & 5hNiCof.

The chemical states of the as-prepared NiCof were identified using XPS and are shown in Fig. 2a & 2b. The Ni 2p spectrum exhibits a 3/2 spin orbit of an oxidation state of 2+ at 856.5 and 862 eV [16]. Meanwhile the Co 2p spectrum reveals a binding energy of 781, 783 and 786 eV [17], representing an oxidation state of 2+. It is worth to note that there is no 0+ state present in the as-prepared samples. As expected, the reduced NiCof shows an extra peak for 0+ state of nickel and cobalt (Figure 2c & 2d) with a binding energy of 852.5 [16] and 778.4 eV [18] respectively. The mixed state of the metals in the reduced samples indicates that the reduction did not take place completely. Longer reduction time allows more Ni and Co to be reduced as shown in the XRD profiles in Fig. 1a. The Ni and Co metal intensities were more obvious in 5 hour compared to the 30 minutes reduction time.

Looking at the morphology of the 5hNiCof displayed in Fig. 3a, it provides another unique characteristic of this prepared catalyst where the particle size is estimated to be in the range of 5 nm. The energy-dispersive x-ray (EDX) analysis shown in Fig. 3b confirmed the presence of Ni and Co in 5hNiCof.
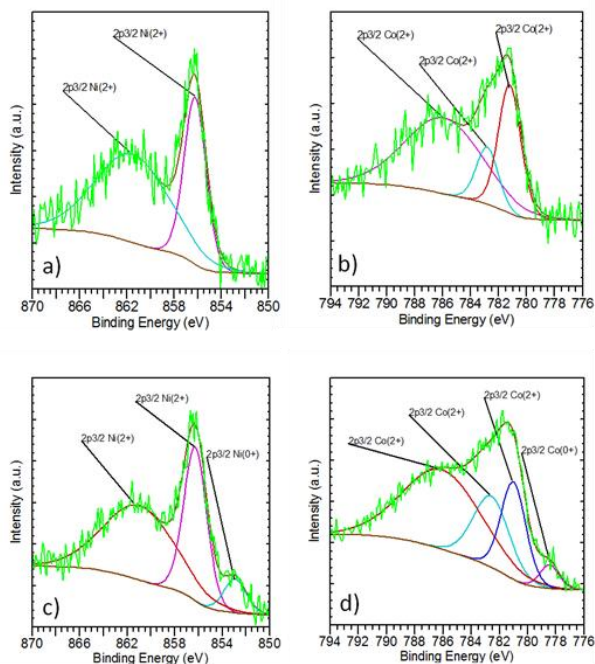

Fig. 2: Narrow scans XPS spectra for NiCof a) Ni $2p_{3/2}$, b) Co $2p_{3/2}$ and 5hNiCof c) Ni $2p_{3/2}$ and d) Co $2p_{3/2}$.
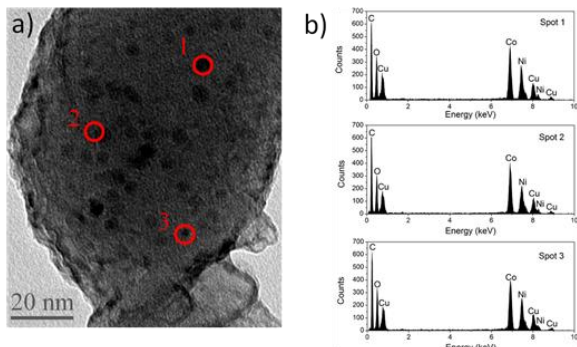

Fig. 3: Images for 5hNiCof of a) TEM bright field and b) EDX element analysis correspond to spots on a).

B. Characterization of MgH$_2$ composites

Fig. 4 shows the XRD patterns of all the MgH$_2$ composites in as-milled and M5h-NiCof at dehydrogenated states. In the case of the as-milled sample, the most intense Bragg peaks were identified as tetragonal β-MgH$_2$ (PDF 12-0697) and a meta-stable orthorhombic phase α-MgH$_2$ (PDF 35-1184) also can be observed, upon milling MgH$_2$, which is consistent with previously reported work [15], [20] . In the dehydrogenated sample, the most observed peaks are from Mg (PDF 41-1443) and a small peak of MgO (PDF 45-0946) was detected in all samples, which was likely due to the slight oxygen contamination during the XRD test.

After 4 hours milling, all the as-milled MgH$_2$ composites showed broadened diffraction peaks. The observed effects result from plastic deformation, re-crystallization, fracturing and cold welding of material [19]. The grain size of host MgH$_2$ in all as-milled samples was estimated to be about 12 nm from the modified Scherrer equation, thus suggesting that the additives do not affect the diffraction patterns in any appreciable way.

Fig. 4: XRD profiles for as-milled M5h-NiCof, dehydrogenated M5h-NiCof, M30m-NiCof, MNiCof and M4.

The TEM dark field image of as-milled M5h-NiCof is shown in Fig. 5a and it was estimated that the grain size of MgH$_2$ was in the range of less than 15 nm, which in accordance to the XRD estimation. This also clearly indicates that these additives do not accelerate the sorption kinetics by changing the MgH$_2$ microstructure. The EDX element analyses were performed on M5h-NiCof to identify the presence of Ni and Co species, as well as to observe their dispersion in the synthesized composites. Fig. 5b shows the EDX spectrums corresponding to selected spots shown in Fig. 5a, respectively. It is clear that the distributions of Ni and Co species are evenly dispersed in the synthesized composite and this suggests their intimate contact with the MgH$_2$ crystallites.
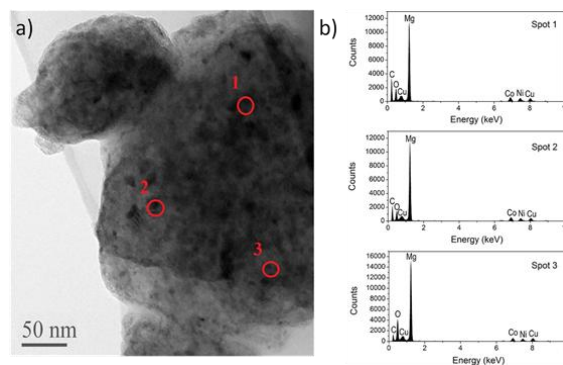

Fig. 5: Images for M5h-NiCof of a) TEM bright field and b) EDX element analysis correspond to spots on a).

The XPS Ni 2p and Co 2p spectrums of 3/2 spin orbital of re-hydrogenated MgH$_2$ composite (MNiCof and M5h-NiCof) show that there are two peaks at 852.5 and 857.5 eV, corresponding to Ni (Ni^{0+}) and NiO (Ni^{2+}), and three peaks at 778 eV, corresponding to Co (Co^{0+}); 782.1 and 786.5 eV both correspond to CoO (Co^{2+}) (Fig. 6a,b,c & d). The ratio between 0+ and 2+ states for both Ni and Co was more obvious in M5h-NiCof compared to MNiCof. This suggests that more reduced metal present in M5h-NiCof which act as catalyst thus expected to boost the hydrogen sorption performance. The milling process also allows the metal species in 5h-NiCof to be well distributed, while having intimate contact with MgH$_2$ crystallites.
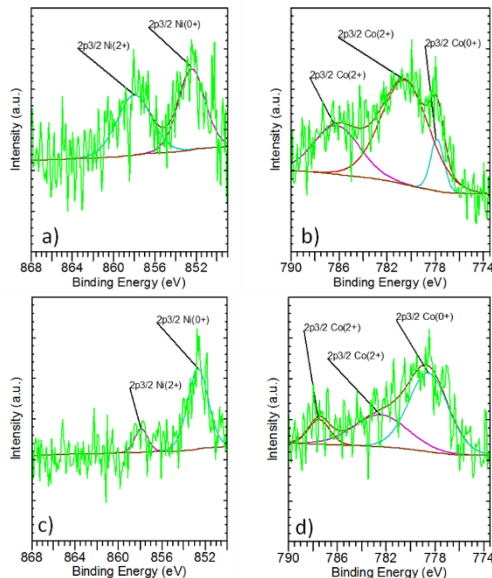
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Fig. 6: Narrow scans XPS spectra for MNiCof a) Ni $2p_{3/2}$ & b) Co $2p_{3/2}$ and M5h-NiCof c) Ni $2p_{3/2}$ and d) Co $2p_{3/2}$

C. Absorption

As expected, improved absorption kinetics were observed in samples containing additives compared to M4, as shown in Fig. 7a. In 2 minutes at 200° C, M5h-NiCof displayed the best absorption amount of 7.1 wt% of hydrogen based on magnesium in the composite. Meanwhile, for the same absorption time, 6.8 and 6.6 wt% of hydrogen was absorbed by M30m-NiCof and MNiCof, respectively. This suggests that the existence of reduced NiCof contributes to the enhanced absorption kinetics of the composites. Several authors have postulated that the presence of metal clusters and metal ions of Ni and Co in different valence states are responsible for the improved sorption performance of magnesium composites [20]. The longer reduction time promotes more metal species to be reduced thus with intimate contact between reduced metal species and magnesium provides active sites for fast hydrogen dissociation and possible sites for hydride nucleation and growth as proposed by Schimmel et al [21].

To investigate further, the absorption rate of M5h-NiCof based on the composite was studied at different temperatures of 150 and 100 °C and is shown in Fig. 7b. It is clearly shown that having 5h-NiCof in the composite enhanced the absorption kinetic, even at a lower absorption temperature of 150 °C. In two (2) minutes the composite absorbed 6.3 wt% of hydrogen at 200 °C. At lower temperatures of 150 °C, the composite absorbed 5.5 wt% within the same time limit, and even at as low as 100 °C hydrogen may be absorbed as much as 3.0 wt% in 10 minutes. This superior low temperature absorption performance is very significant for practical applications.
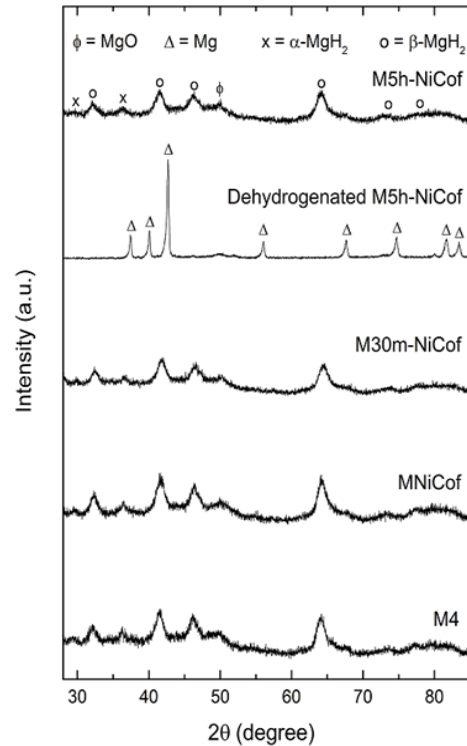

Fig. 7: Hydrogen absorption at a) 200 °C based on Mg of M5h-NiCof, M30m-NiCof, MNiCof & M4 and b) 200, 150 & 100 °C for M5h-NiCof.

D. Desorption

Temperature programmed desorption (TPD) spectra of hydrogen from the prepared samples are shown in Fig. 8. All samples display a single desorption peak except for MNiCof, with multiple peaks at 285, 345 and 390 °C. The possible reason for the observed multiple peaks in MNiCof is the catalytic effect of various chemical states of the metal species of the additive in the composite as shown in Fig. 6a & 6b.

Composites with reduced NiCof display a single peak, in which the catalytic effect is dominated by zero state (0+) of the metal additives. A longer reduction time of NiCof produced more zero state (0+) of the metal and the effect can be observed by narrower peak shown in the evolution of M30m-NiCof to M5h-NiCof. M4 exhibits the highest peak at 370 °C, while MNiCof M5h-NiCof and M30m-NiCof shows a drop of 80 and 45 °C respectively. This suggests that the catalytic effect of reduced NiCof enhanced hydrogen release of the composite. With respect to the onset temperature, M5h-NiCof displays the lowest of 220 °C compared to 327, 230 and 235 °C for M4, M30m-NiCof and MNiCof respectively, implying that reduced metal species promotes lower temperature hydrogen release.

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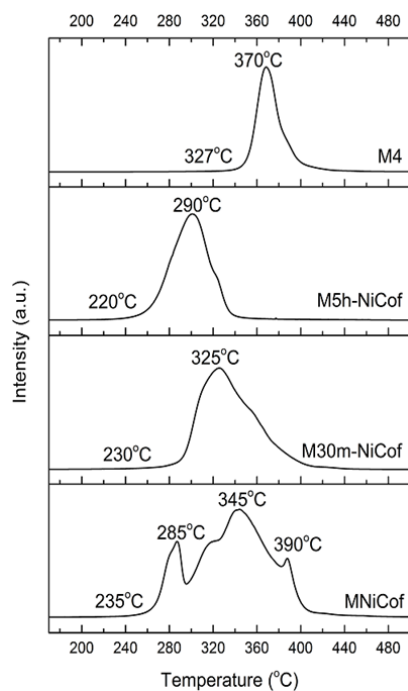*& Sciences Publication*

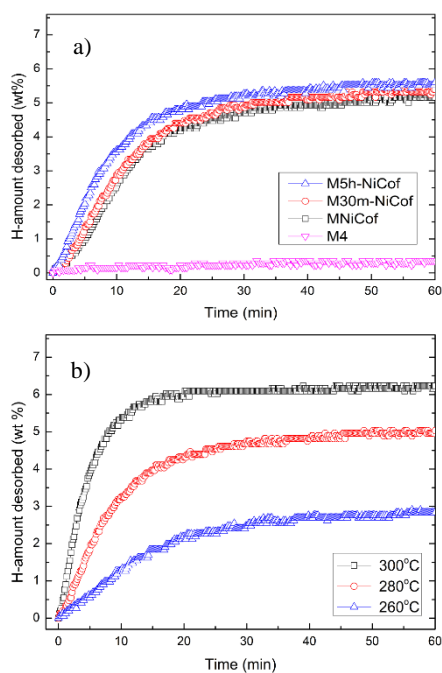Fig. 8: TPD-MS profiles of M4, M5h-NiCof, M30m-NiCof and MNiCof.

Fig. 9: Hydrogen desorption a) at 280 °C based on Mg of M5h-NiCof, M30m-NiCof, MNiCof & M4 b) at 300, 280 and 260 °C, respectively based on composite of M5h-NiCof.

The rate of hydrogen released in the storage material at a reasonable temperature is a critical factor in determining the practicality in mobile application. Dehydrogenation rate measurements were characterized for all samples at 280 °C. Composites with additives show improved rate of desorption compared to non-catalyzed MgH_2 (M4) as shown in Fig. 9a. Comparison of desorption rate between all samples was made based on the mass of magnesium in the composite. In 20

minutes M5h-NiCof released 4.8 wt% of hydrogen, while M30m-NiCof and MNiCof only desorbed 4.4 and 4.2 wt% of hydrogen, respectively. However, M4 barely released any hydrogen at 280 °C. This trend was in accordance to the observation made by TPD suggesting that reduced NiCof was responsible for the enhanced desorption kinetic. The advantage of employing MOF into the system was benefited by having the metal species assembled such that they are already well coordinated. With the effect of high energy ball milling, the distribution of the metal species of both Ni and Co can further enhanced as shown in the previous section. With more reduced Ni and Co being well distributed, more intimate contact between the catalysts and MgH_2 and thus more active sites will be available. Furthermore, the composites displayed reasonably good kinetics even at lower temperature as shown in Fig. 9b. In 30 minutes the hydrogen desorbed amount in M5h-NiCof is 4.7 and 2.5 wt% (calculated based on the mass of the composite) at 280 °C and 260 °C respectively.

The desorption thermodynamic properties were investigated according to technique adopted from Paskevicius et al. [22], where equilibrium desorption pressure is measured at a very long collection time ($\geq$ 25 hours) to ensure true Mg-H equilibrium was reached. The insert in Fig. 10 shows the equilibrium desorption pressure curves of M5h-NiCof at 300, 280 and 260 °C, respectively. It is assumed that the fugacity is equal to the pressure by considering relatively low equilibrium pressures (less than 100 KPa) were measured in all samples. This consequently allows the van't Hoff plot to be constructed (Fig.10).
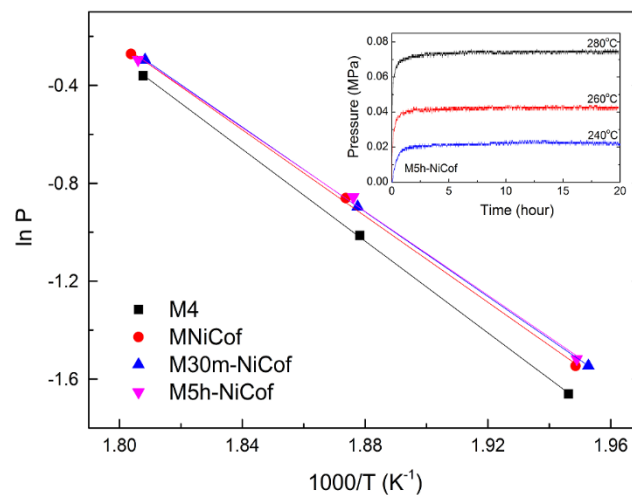

Fig. 10: van't Hoff plot for desorption of M4, MNiCof, M30m-NiCof and M5h-NiCof respectively. Inset: Kinetics desorption plots of M5h-NiCof showing that hydrogen desorption equilibrium was reached at all temperatures.

The decomposition thermodynamic properties were extracted from the plot and tabulated in Table I. All samples with additives show a drop in enthalpy (ΔH) and entropy (ΔS) compared to M4. Both MNiCof and M30m-NiCof show a similar effect towards thermodynamic stability having decomposition enthalpy (ΔH) of 72.09 ± 1.50 and   72.00 ± 1.25 kJ/mol H_2 respectively and entropy (ΔS) of 127.88.71

± 2.74 and 127.71± 2.33 J/mol H$_2$·K respectively.

Further reductions in the thermodynamic properties are observed when a longer reduction time of NiCof (5hNiCof) is introduced to the system. Displaying ΔH and ΔS of 69.68 ± 1.16 kJ/mol H$_2$ and 123.39 ± 2.24 J/mol H$_2$·K suggests that reduced metal species of the MOF enable hydrogen release at lower temperature. Thus it is in accordance to the observation made in the TPD analysis (Fig. 8), confirming that the thermodynamic properties of MgH$_2$ composites could be improved by adding reduced NiCof.

Table I: Thermodynamic properties of MgH$_2$ and MgH$_2$ composites.

Sample	Desorption	
	Enthalpy (ΔH, kJ/mol H$_2$)	Entropy (ΔS, J/mol H$_2$·K)
M4	77.41 ± 2.08	136.86 ± 3.49
MNiCof	72.09 ± 1.50	127.88 ± 2.74
M30m-NiCof	72.00 ± 1.25	127.71 ± 2.33
M5h-NiCof	69.68 ± 1.16	123.39 ± 2.24

IV. CONCLUSION

Improvement has been made in hydrogen storage performance particularly in MgH$_2$ system prepared via high energy ball milling technique where bi-metal in the metal organic framework of Ni-Co-MOF-74 was introduced as catalyst. Better catalytic effect was recorded when pre-reduced Ni-Co-MOF-74 is used as catalyst for both hydrogen absorption and desorption. An apparent amount of reduced metal species in the MgH$_2$ system containing bi-metal in metal organic framework promotes more active sites which eventually improved the sorption properties and reduced the thermodynamic stability of the composite by having the desorption enthalpy (ΔH) and desorption entropy (ΔS) of 69.68 ± 1.16 kJ/mol H$_2$ and 123.39 ± 2.24 J/mol H$_2$·K, respectively . The distinctive coordinate of the metal in the pre-reduced metal organic framework improved the dispersion of the catalyst thus enriched the sorption properties and this can be clearly presented by having hydrogen desorption of 4.7 wt% in a period of 30 minutes at a relatively low desorption temperature of 280°C, whereas at 200°C, ultrafast hydrogen absorption with the amount of 6.3 wt% was stored in 2 minutes. At the lower temperature of 100 °C the composite absorbed 3.0 wt% of hydrogen in 10 minutes, thus showing the potential of reduced MOF as a catalyst in hydrogen storage material.

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AUTHORS PROFILE

Atikah Kadri started her career as an academician in the Faculty of Chemical Engineering, Universiti Teknologi MARA after earning MSc. Engineering from University Malaya in 2003. Prior to that she attended The University of Sheffield, UK where she graduated in B. Eng. (Hons.) Chemical and Process Engineering in 1998. Her passion in nanotechnology brought her to The University of Queensland Australia where she was attached to Australian Institute for Bioengineering and Nanotechnology (AIBN) and soon awarded PhD in Advanced Materials Engineering in 2013. Her research areas are hydrides hydrogen storage, nano-catalysts and microwave extraction technique.

Xiangdong Yao is a full Professor of Energy Materials at Griffith University. He obtained his BEng at Northeastern University in 1989 and MEng at Northwestern Polytechnical University in 1992 respectively for Materials Science and Engineering. From 1992 to 2000, he was employed by the Institute of Metal Research, Chinese Academy of Science as a Research Associate (1992), Assistant Professor (1995) and Associate Professor (1998). In 2000, he went to The University of Queensland where he was granted a PhD degree in Materials Engineering in 2005, working on the computational modeling for microstructure formation in light metals. From November 2003, he joined the ARC Centre of Excellence for Functional Nanomaterials at the University of Queensland. Since November 2009, he joined Griffith University as an Associate Professor and the group leader of Advanced Energy Materials, and was promoted to full Professor in late 2012. Dr. Yao's current research focuses on Energy Materials, especially hydrogen-related materials.