

Research Article

Preparation of Mg_2FeH_6 Nanoparticles for Hydrogen Storage Properties

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Magnesium (Mg) and iron (Fe) nanoparticles are prepared by thermal decomposition of bipyridyl complexes of metals. These prepared Mg-Fe (2:1) nanoparticles are hydrogenated under 4 MPa hydrogen pressure and 673 K for 48 hours to achieve Mg_2FeH_6 . Their structural analysis was assessed by applying manifold techniques. The hydrogen storage properties of prepared compound were measured by Sieverts type apparatus. The desorption kinetics were measured by high pressure thermal desorption spectrometer (HP-TDS). More than 5 wt% hydrogen released was obtained by the Mg_2FeH_6 within 5 min, and during rehydrogenation very effective hydrogen absorption rate was observed by the compound.

1. Introduction

The demand for an efficient and clean alternative fuel has increased in the recent years due to energy crisis and growing environmental concerns [1]. The use of hydrogen as an alternate fuel is a forward step to give up noninvasive and nontoxic emission, which is spoiling our surroundings [2]. The increasing relevance of hydrogen economy has generated interest in finding a safe way to transport hydrogen. Magnesium (Mg) is considered as attractive material for hydrogen storage economy due to light weight, low cost, and high storage capacity (7.6 wt%) [3, 4]. But slow kinetics and high temperature limit its practical use. Many efforts have been made to improve its absorption rate by reducing size or by adding transition metals [5, 6], creating defects by ball milling [7–10], synthesizing composite of metal hydrides [11, 12], and adding catalyst [13–18]. Mg-based 3d transition metal hydrides have attracted much interest for the future hydrogen economy, due to their great abundance, high reversible storage capacity, and better kinetics [19–21]. Mg_2FeH_6 is considered as attractive/gorgeous materials in particular due to its high gravimetric capacity 5.4 wt% and superior volumetric density $150 \text{ kg H}_2 \text{ m}^{-3}$. However, Mg and

Fe do not form stable binary compound; therefore synthesis of Mg_2FeH_6 is very difficult [22, 23].

First report for the preparation of Mg_2FeH_6 compound was published by Didishein et al. [24], by sintering process of Mg and Fe powder under high hydrogen pressure. By applying high hydrogen pressure (12 MPa), elevated temperature (793 K) and after several days, the yield of complex hydride was only 50 wt. (%). To improve the yield (%), a processing route that is, mechanical alloying of precursory materials before sintering was employed [25]. Another approach for the synthesis of Mg_2FeH_6 compound was also performed by reactive milling (RM) under high hydrogen pressure [26]. This mechanically activated method can reduce the particle size to nanolevel and enhance the hydrogen sorption kinetics of the hydrides. However, the obtained compound still contained unreacted MgH_2 and Fe. Alternatively, Raman et al. [23] milled the $2\text{Mg} + \text{Fe}$ mixture under 1 MPa hydrogen pressure. Moreover, the compounds often oxidized and got polluted by sintering, mechanical alloying, and reactive milling methods.

Progress regarding the yield has been made by using optimized sintering or RMA conditions, and more recently, by hydriding combustion synthesis [27] or sintering of metal

nanoparticles produced by a hydrogen plasma metal reaction [28]. However, one drawback of the processing methods (with yields over 90%) is the long processing time involved, which is usually 10 h. Additionally, the Fe is usually retained in the synthesized material; for this reason, a purification process is necessary.

In this work, Mg and Fe pure nanoparticles are prepared by the thermal decomposition of bipyridyl complex of Mg and Fe. This is an alternate approach for the preparation of pure Mg and Fe nanoparticles. (2:1) Mg-Fe nanoparticles were transferred into reactor to form Mg_2FeH_6 under 4 MPa hydrogen pressure. Hydrogen storage behavior of the Mg_2FeH_6 compound having yield more than 90% was also investigated.

2. Experimental

The reagents and chemicals used are magnesium chloride (Sigma Aldrich), iron chloride (Sigma Aldrich), 2,2' bipyridine (Fluka), 1-propanol, and diethyl ether. Scheme 1 is give brief illustration for the preparation of pure metal nanoparticles.

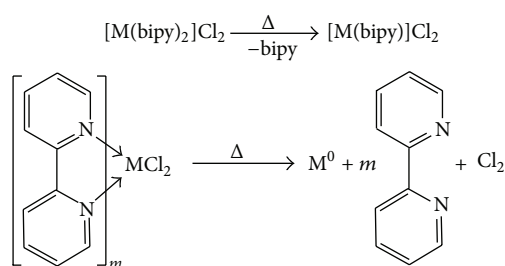
2.1. Preparation of $[\text{Mg}(\text{Bipy})_3]\text{Cl}_2$. 2.56 gm (15 mmol) solution of 2,2' bipyridine and 4.75 g (50 mmol) solution of magnesium chloride were prepared in 1-propanol. Both solutions were mixed through dropping funnel in a conical flask with constant stirring at a temperature of 323–333 K. Bipyridyl complex of magnesium did not precipitate by adding the bipyridine solution to the magnesium chloride solution, but the color of the mixture changed from transparent to orange. The solution was concentrated to two-third of the original through rotary evaporator and then kept for crystallization. Crystallization occurred very slowly and took six days for completion. The crystals of the bipyridyl complex were separated through filtration, washed with diethyl ether, and dried under IR lamp.

Melting point, IR, NMR, and TGA results of the compound are given below.

442 K, IR (KBr, cm^{-1}): ν (C–H_{arom}) 3050, 2927; ν (C–N_{arom}) 1600; ν (C–C_{arom}) 1501, 1453, 900–600; ν (Mg–N) 415. HNMR (DMSO, ppm): 8.687 (dd, 2H, bipyH_{2,2'}); 8.384 (dd, 2H, bipyH_{5,5'}); 7.95 (ddd, 2H, bipyH_{4,4'}); 7.458 (ddd, 2H, bipyH_{3,3'}). TGA: 460–580 K (80.32% weight loss), 606–722 K (18.22% residual mass).

2.2. Preparation of $\text{Fe}[(\text{Bipy})_2]\text{Cl}_2$. 2.97 g (15 mmol) tetrahydrated ferrous chloride ($\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$) and 7.05 g (45 mmol) 2,2'-bipyridine dissolved in solvent (2-propanal) separately. Both solutions were mixed with HPLC pump in flask with constant stirring at room temperature, keeping flow rate very slow (0.02 ml/sec) to get the homogenous mixture. Yellow precipitates were abstained, which were washed with tetrahydroformide to remove the impurities. The complex was crystallized from methanol solution in open air.

Melting point, IR, HNMR, and TGA results of the compound are given below.



General scheme for the preparation of nanoparticles

SCHEME 1

432 K, IR (KBr, cm^{-1}): ν (C–H_{arom}) 3059, 2905; ν (C–N_{arom}) 1593; ν (C–C_{arom}) 1502, 1442, 900–600; ν (Fe–N) 413. ¹HNMR (DMSO, ppm): 7.798 (s, 6H, bipyH_{4,4'}); 7.301 (s, 6H, bipyH_{5,5'}); 8.441 (s, 6H, bipyH_{6,6'}); 8.224 (s, 6H, bipyH_{3,3'}). TGA: 469–510 K (28.12% weight loss) 606–718 K, (38.41% weight loss), residual mass 25.13%.

Keeping in view all these observations, following pathway has been proposed for thermal degradation of $\text{M}_x[\text{bipy}]_y\text{Cl}$ to metal. Bipyridine was removed from the metal complex in two or three steps. This shows coordination on metal centre paving the way for H-elimination exclusion form pyridine ring followed by the reduction, elimination, and finally formation of metal nanoparticles.

2.3. Preparation of Mg-Fe Hydride Nanoparticles. 1.5 gm of as-prepared pure (2Mg:Fe) nanoparticles was minced in gloves box to get the homogeneous mixture. This mixture was then transferred into sample tube of the chamber. First the chamber was flushed with argon to remove the oxygen contents and evacuated up to 10^{-3} Pa. In the second step the chamber was heated at 673 K with the heating rate of 5 K/min under 4 MPa hydrogen pressure. After 24–26 hours Mg was converted into MgH_2 , but no reaction with Fe nanoparticles occurred. After prolonging hydrogenation for 48–50 hours MgH_2 reacted with Fe and converted into Mg_2FeH_6 . The structural properties of the compound were analyzed by PANalytical X'Pert Pro powder X-rays diffractometer (XRD). The details of apparatus are given in our previous report [18].

Morphology and particle size of compound were determined by Amray-1910FE field emission scanning electron microscope (FESEM). Thermal desorption spectra were measured by Setaram Labsys apparatus under an argon flow in an Al_2O_3 crucible in the range of 300–673 K at heating rates 2, 5, 10, and 15 K/min. Additionally, hydrogen absorption and desorption behavior was studied by using Sieverts type apparatus. The results show that very promising hydrogen storage behavior was observed by the Mg_2FeH_6 nanoparticles.

3. Results and Discussions

XRD patterns of the Mg-Fe compound are shown in Figure 1. The pattern in Figure 1(a) belongs to pure Mg (PDF-04-0770) and pure Fe (PDF-34-0529) nanoparticles. Figure 1(b) shows

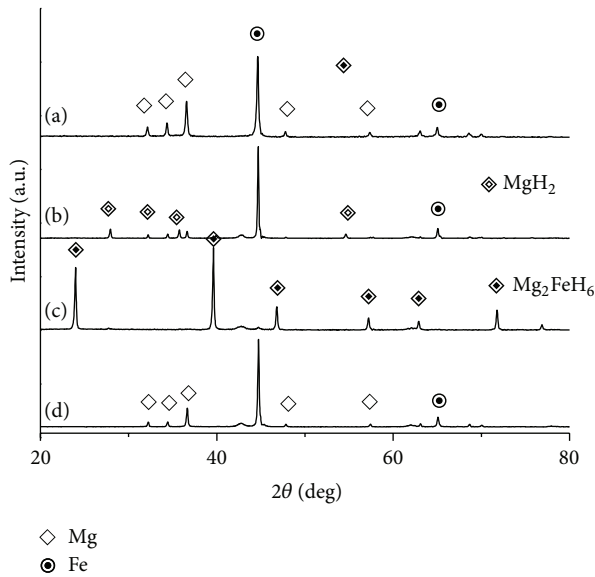


FIGURE 1: XRD patterns of Mg-Fe compound; (a) mixture of Mg and Fe, (b) after 24-hour hydrogenation, (c) after 48-hour hydrogenation, and (d) after dehydrogenation under initial 200 Pa hydrogen pressure.

the mixed pattern of MgH_2 (PDF-12-0697) and Fe after 24-hour hydrogenation. Figure 1(c) confirms the pure phase of Mg-Fe hydride (PDF-38-0843) where Figure 1(d) represents again the pure elemental phase of Mg and Fe nanoparticles after dehydrogenation.

The XRD results also show that, after hydrogenation sharpness of the Mg-Fe hydride peaks increased, with the insertion of hydrogen atom, the surface lattice volume of the compound is increased. This expansion and contraction causes cracks in the particles, resulting in the decrease of particle size [33].

Figure 2 shows the morphology and particle size of Mg-Fe compound after hydrogen absorption and desorption. Figure 2(a) shows the mixed phase of Mg and Fe nanoparticles. It also shows that particles are homogeneously dispersed, but the mixture has different particle sizes. The larger particles are surrounded by the smaller particles. Broadness of the peak of Fe from XRD pattern illustrates that smaller particles are of iron, where larger particles belong to magnesium.

Figure 2(b) again shows the mixed phase of MgH_2 and Fe after 24-hour hydrogenation under 4 MPa pressure at 673 K. SEM results also show some aggregation effect due to higher temperature. After 48 hours of hydrogenation, the compound completely converted into Mg-Fe hydrides as conformed from Figure 1(c). Figure 2(c) shows that the compound is elongated. This elongation might be due to the distortion of lattice after the insertion of hydrogen atom [34].

Figure 2(d) shows the SEM micrograph of regenerated Mg and Fe nanoparticles after dehydrogenation under 200 Pa hydrogen atmosphere. Significant decrease in particle size was observed after dehydrogenation, due to the elimination of hydrogen from the compound. Hydrogen desorption results are illustrated in Figure 3 at different temperatures

under initial 200 Pa hydrogen pressure, which indicates that up to 5 wt% hydrogen desorption occurs within 3–5 min. Results also show that the rate of desorption is effectively increased by increasing the temperature. At 573 K, more than 5 wt% hydrogen was released by the system and 4.4 wt% and 3.5 wt% hydrogen desorption occurred within 5–10 min at temperatures of 523 and 473 K, respectively.

More than 5 wt% release of hydrogen also justifies the purity of Mg_2FeH_6 nanoparticles.

The hydrogen absorption of 2Mg:Fe compound at different temperatures, under 4 MPa hydrogen, is presented in Figure 4. Within 30–45 sec, 4.5% hydrogen was absorbed by the system at 573 K. This high absorption rate is due to the catalytic effect of Fe nanoparticles. The absorption rate becomes slow due to the reaction of MgH_2 with H_2 and Fe, resulting in the formation of Mg_2FeH_6 . Very promising hydrogen absorption 3.7 wt% and 4 wt% was also observed by the compound at 473 K and 523 K, respectively. This high absorption rate is also due to the decrease in particle size after dehydrogenation and optimal morphology, which provide the shorter diffusion distance for hydrogen atoms during the solid-solid and gas-solid reactions compared with microsize compound to diffuse into the surface lattice [35].

Figure 5 represents the thermal desorption spectrum of Mg_2FeH_6 compound with the different heating rates of 2 K/min, 5 K/min, 10 K/min, and 15 K/min. Desorption peaks at 648 K, 630 K, 625 K, and 610 K corresponds to heating rates of 15 K/min, 10 K/min, 5 K/min, and 2 K/min. The desorption activation energy of the compound can be estimated by using Kissinger equation [36]:

$$\frac{(\ln(\beta/T_p^2))}{d(1/T_p)} = -\frac{E}{R}, \quad (1)$$

where T_p is the peak desorption temperature, β is the heating rate, E is activation energy, and R is the gas constant.

The activation energy of pure Mg-Fe hydrides (Mg_2FeH_6) nanoparticles compound calculated from Kissinger plot as shown in the inset of Figure 5 is 159.8 ± 6.7 kJ/mol. These results are closely matched to the 165.7 ± 8.4 kJ/mol and 174 ± 36 kJ/mol demonstrated by Gennari et al. and Zhang et al. [8, 29], respectively, whereas their synthesis procedure is quite different as compared to this report. In this process, some contents of Fe remained unreacted, which played as catalyst, due to the lower activation energy which they have reported.

The pressure-composition-temperature (P-C-T) curves shown in Figure 6 were obtained during the first hydrogen desorption/absorption cycle without any activation process at 473, 523, and 573 K. Only one plateau is observed during dehydrogenation and rehydrogenation of compound. Hydrogen desorption contents are 3.8 wt% at 473 K, 4.3 wt% at 523 K, and 4.95 wt% at 573 K. Whereas during rehydrogenation 4.7 wt% at 573 K, 4.13 wt% at 523 K, and 3.4 wt% at 473 K were absorbed by the compound. Desorption and absorption plateau pressure of hydrogen is given in Table 1. The hydrogen storage capacity and the equilibrium pressure in both absorption and desorption processes decrease as the temperature falls. This fact is due to the metal atom

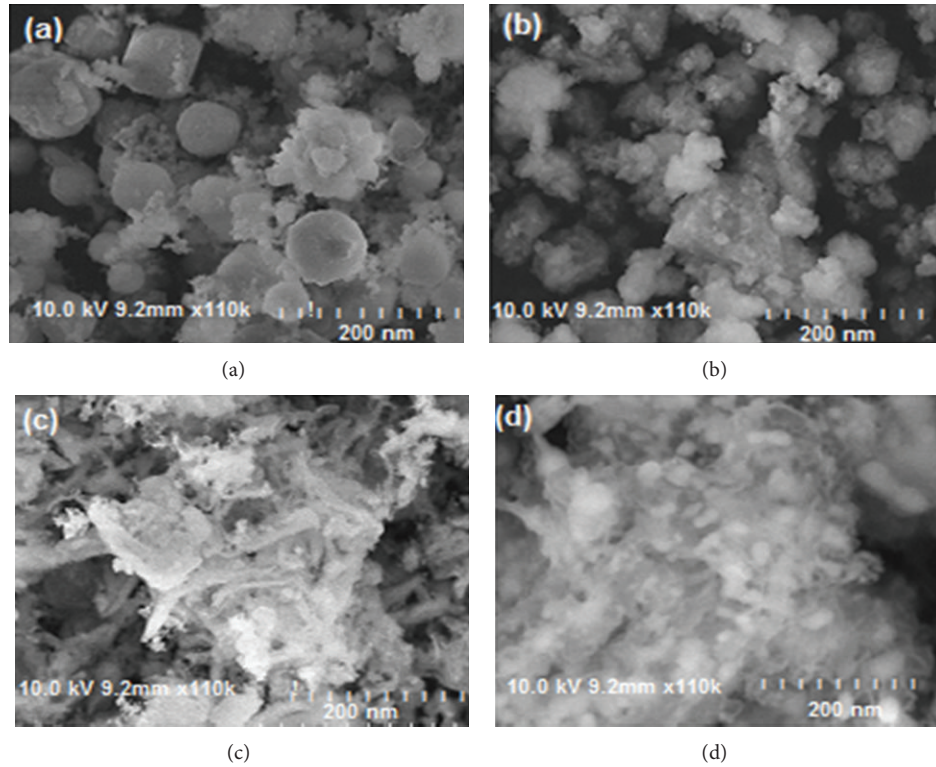


FIGURE 2: SEM images of Mg-Fe system: (a) mixture of Mg and Fe nanoparticles, (b) after 24 hr hydrogenation, (c) after 48 hr hydrogenation, and (d) after dehydrogenation.

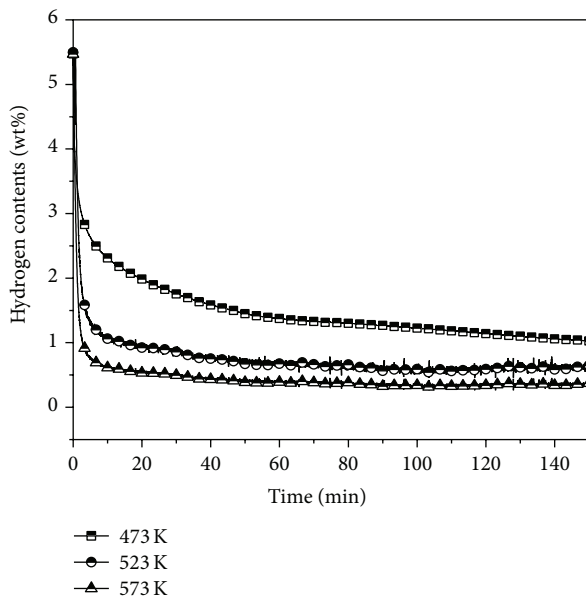


FIGURE 3: Hydrogen desorption properties of Mg_2FeH_6 nanoparticles at different temperatures under initial 200 Pa hydrogen pressure.

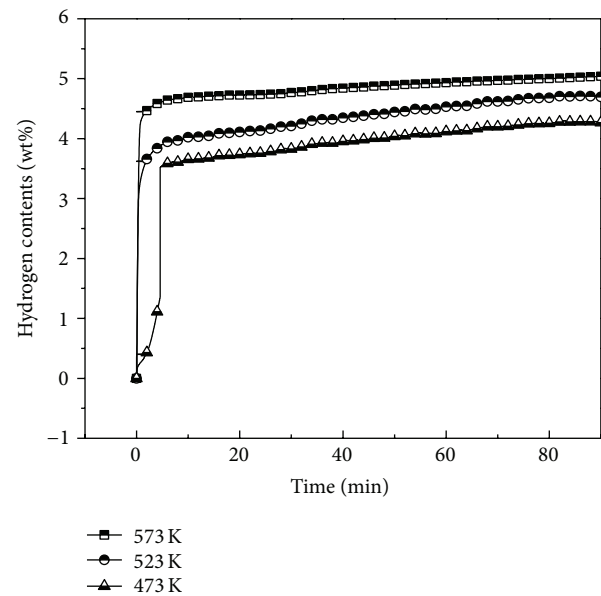


FIGURE 4: Hydrogen absorption properties of Mg-Fe hydride nanoparticles at different temperatures.

diffusion mechanism involved in absorption process, which is enhanced by rise in temperature [22]. Both P-C-T curves of absorption and desorption can reach more than 4 wt% hydrogen content, which suggests the excellent reversibility.

The thermodynamic information for Mg_2FeH_6 hydriding/dehydriding was determined by plotting the logarithmic dissociation pressures against the reciprocal temperature (van't Hoff plot) as shown in Figure 6. The entropy (ΔS) and

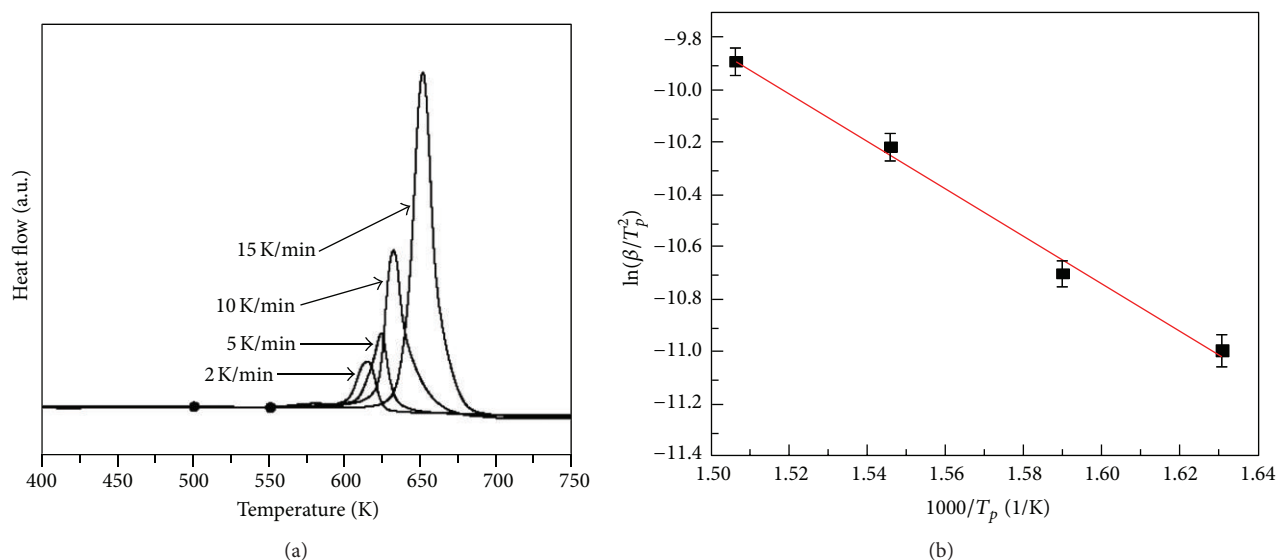


FIGURE 5: (a) Thermal desorption spectrum of Mg-Fe hydride nanoparticles with different heating rates. (b) Represents the Kissinger plot for activation energy.

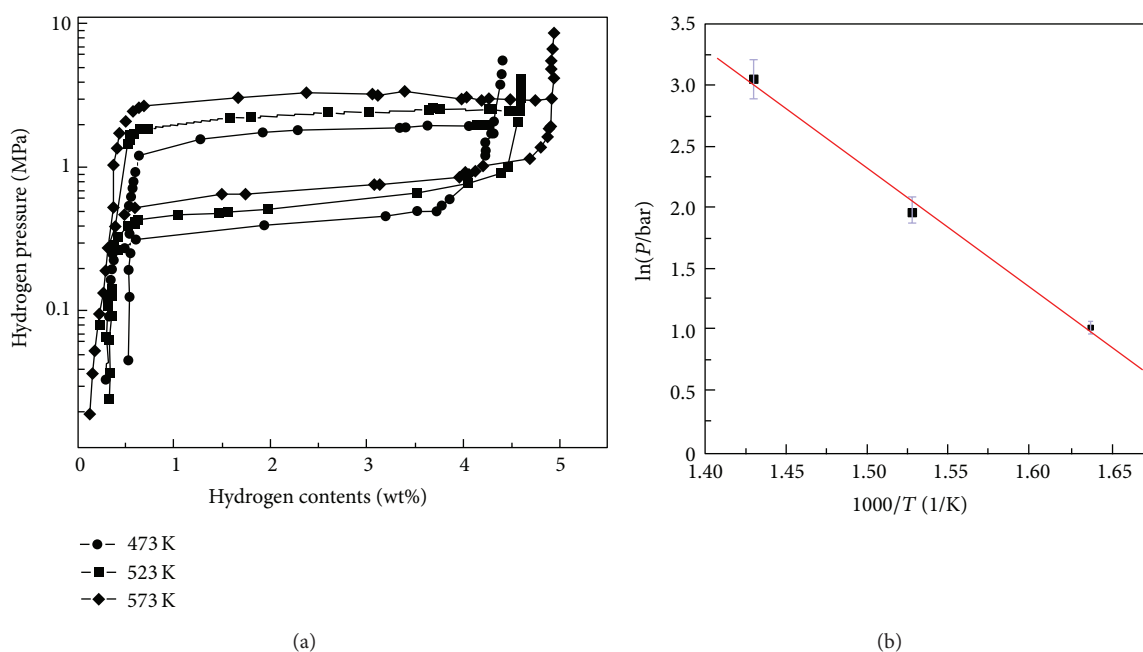


FIGURE 6: (a) Pressure-composition-temperature curves of hydrogen desorption and absorption of the samples at 473, 523, and 573 K, (b) van't Hoff plot of Mg_2FeH_6 compound constructed from desorption isotherms at 473, 523, and 573 K.

enthalpy (ΔH) values per mole of hydrogen of Mg_2FeH_6 compound were calculated by the data given in Table 1. Comparison of thermodynamic properties of this work with previous reports is given in Table 2.

4. Conclusions

In this work, pure Mg_2FeH_6 with the yield more than 90% was successfully prepared after the hydrogenation of (2:1)

Mg-Fe nanoparticles under 4 MPa hydrogen pressure. The synthesized compound has grain size about 60–70 nm. More than 5 wt% hydrogen released was observed within 5 min under 200 Pa hydrogen pressure at 573 K.

Results show that hydrogen absorption was achieved in two parts. In first step, within 1 min more than 4 wt% hydrogen absorption is attributed to the catalytic effect of Fe nanoparticles. In second step absorption rate is slow due to reaction of Fe with MgH_2 to form Mg_2FeH_6 . This effective dehydrogenation/hydrogenation rate is also due to effective

TABLE 1: Pressure-composition-isotherm (PCI) characteristics of Mg-Fe hydride nanoparticles at different temperatures.

Temperature (K)	Hydrogen desorption (wt%)	Plateau pressure desorption (MPa)	Hydrogen absorption (wt%)	Plateau pressure absorption (MPa)
473	3.8	0.32	3.4	1.8
523	4.3	0.41	4.1	2.2
573	4.95	0.72	4.7	3.1

TABLE 2: Entropy and enthalpy values for formation and decomposition of Mg_2FeH_6 compound calculated from van't Hoff plot.

No.	Entropy (ΔS)	Enthalpy (ΔH)	Reference
(1)	-145 ± 9	-89 ± 6	This work
(2)	-140.2 ± 8.7	-82.4 ± 5.8	[29]
(3)	147 ± 15	-87 ± 3	[30]
(4)	-137 ± 13	-80 ± 28	[31]
(5)	-147 ± 9	-86 ± 6	[32]

decrease in activation energy which was calculated from Kissinger plot. High absorption and desorption behavior of Mg_2FeH_6 compound at nanoscale is a forward step for the hydrogen storage materials for mobile applications.

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