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Hydrogen Generation from Magnesium Hydride By Using Organic Acid

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**HYDROGEN GENERATION FROM MAGNESIUM
HYDRIDE BY USING ORGANIC ACID**

by

Yen-Hsi Ho

A Thesis Submitted in
Partial Fulfillment of the
Requirements for the Degree of
Master of Science
in Engineering

at

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August 2013

ABSTRACT

HYDROGEN GENERATION FROM MAGNESIUM HYDRIDE BY USING ORGANIC ACID

by

Yen-Hsi Ho

The University of Wisconsin-Milwaukee, 2013
Under the Supervision of Professor Tien-Chien Jen

In this paper, the hydrolysis of solid magnesium hydride has been studied with the high concentration of catalyst at the varying temperature. An organic acid (acetic acid, CH_3COOH) has been chosen as the catalyst. The study has three objectives: first, using three different weights of MgH_2 react with aqueous solution of acid for the hydrogen generation experiments. Secondly, utilizing acetic acid as the catalyst accelerates hydrogen generation. Third, emphasizing the combination of the three operating conditions (the weight of MgH_2 , the concentration of acetic acid, and the varying temperature) influence the amount of hydrogen generation. The experiments results show acetic acid truly can increase the rate of hydrogen generation and the

weight of MgH_2 can affect the amount of hydrogen generation more than the varying temperature.

Keywords: Magnesium hydride, Hydrogen generation, Hydrolysis reaction, Acetic acid

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CHAPTER 1 Introduction

1.1 Research and Background

Nowadays, human lives in an industrial world that consumes more and more energy in our daily lives. The major energy resources include petroleum, coal and natural gas. The researchers provide the statistics data of petroleum storage in the world that will deplete in next fifty years. Moreover, burning these carbon-based fuels will generate the byproduct, carbon dioxide, which can cause the serious environmental pollution: globe warming. Globe warming already increase the average temperature in whole world gradually. In recently reports, globe warming result in the climate change, the species extinct, the iceberg melt, and the sea level rise, which endanger the people whose live in the low sea level area, Finding the new green energy resources is one of the urgent issues in the world.

New green energy resources must have some advantages, such as environmental friendly, high efficiency, good stability, and outstanding performance. Hence, the suitable energy resource for above requirements is hydrogen. Hydrogen already been widely used in many applications, such as power generation, transportation, and so on. Japan's government already has a project, call World Energy Network (WE-NET) [1], to research

and develop hydrogen energy. In 2003, Japan's government cooperated with the industrial companies for demonstrating the principle of the fuel cell car. In same time, seven hydrogen stations have been set up to provide hydrogen for the fuel cell cars. In European Union, the hydrogen energy fuel cell buses joined into the driving test in nine cities. After three years, the reports showed twenty seven fuel cell buses accumulated ten millions kilogram mileages and forty millions passengers without causing any serious transportation accidents. However, hydrogen technique still has some challenges in storing method, producing procedure, and working environment. According to hydrogen applications, US Department of Energy (DOE) has set the standards [2] for on-board applications and the targets for hydrogen storage capacity, volumetric and gravimetric capacity, hydrogenation, dehydrogenation, reversibility, and safety. The best method of applying hydrogen energy is the fuel cells.

In long term investigations, energy losses always happen during transforming process so consumers merely use 30~40% energy from the total amount of energy generation. Fuel cells do not limited by Carnot cycle, which can keep higher efficiency of transforming energy than traditional heat engines. Consequently, high energy efficiency fuel cells and hydrogen storage techniques become the key point for discovering new energy resources.

1.2 Fuel Cell Introduction

In recently, many alternative energy resources show up in the world. For example, solar energy, wind power, biofuel, geothermal energy, fuel cell, and so on. The traditional power station generates electricity by burning fossil fuels that force the turbine to rotate and then generate the electricity. The generated energy could be consumed during the transmission processes. By contrast, fuel cell do not need to concern the consumed energy by the transmission processes because the principle of fuel cell is an electrochemistry reaction, which is transfer the chemical energy to the electrical energy directly. Fuel cell has higher efficiency of energy conversion than fossil fuels or the traditional power stations.

In 1838, German scientist Christian Friedrich Schonbein discovered the electrochemistry effect of the fuel cell. Next year, barrister sir William Robert Grove published the first fuel cell thesis in the Philosophical Magazine and Journal of Science. Grove used sulfuric acid solution as the electrolyte, platinum as membrane, hydrogen as a cathode, and oxygen as an anode to build the prototype of fuel cell. This design has been acknowledged for the earliest fuel cell in the world.

Fuel cell can be classified by the electrolyte into four major categories. For example, Solid Oxide Fuel Cell (SOFC) uses yttria-stabilized zirconia (YSZ) for the electrolyte;

Molten Carbonate Fuel Cell (MCFC) uses lithium potassium carbonate salt for the electrolyte; Alkaline Fuel Cell (AFC) uses aqueous alkaline solution for the electrolyte; Proton Electrolyte Membrane Fuel Cell (PEMFC) uses polymer membrane for the electrolyte. The more details show in Table 1.

In different kinds of fuel cells, PEMFC (Fig. 1) has some advantages, such as small volume, lightweight, and portability, which compare with other fuel cells. The principle of PEMFC is that uses oxygen and hydrogen as a fuel to release electron during the chemical reaction. Considering the fuel sources, oxygen can be easily obtained from air. However, there have no convenient methods to obtain hydrogen directly. How to store and release hydrogen becomes one of the most important challenge in PEMFC.

1.3 Hydrogen Storage

Hydrogen storage can be separated by the various storage technologies. For example, the portable applications need to choose the suitable hydrogen storing technique, which should be safe, lightweight, high storing capacity, and low cost. U.S. Department of Energy already set the target of hydrogen storage for the years 2010 and 2017 (Table 2). Five basic well-known hydrogen storage methods are physisorption hydrogen storage, liquid hydrogen storage, high pressure hydrogen storage technique, and chemical-bond hydrogen storage.

Physisorption hydrogen storage means Van der Waals force keep hydrogen molecules on the material's surface and without dissociation. Carbon nanotube is one kinds of physisorption hydrogen storage technology. In 1991, the Japanese scientist, Sumio Iijima, discovered carbon nanotube and published the studying report in Nature Magazine. The advantage of carbon nanotube is the high surface areas, so it can keep hydrogen molecular into the Nano-level lattice structure, which has a high capacity for hydrogen storage. However, this technique is still an immature process and the price is high. In current researches, carbon nanotube has the hydrogen storage capacity less than 1 wt% under ambient conditions. If the sample can be stable during the preparation, the hydrogen storage capacity will be up to 2.5 wt% under cryogenic conditions.

The method of liquid hydrogen storage is that hydrogen will be cooled until 20K and then stored in a cryogenic tank. This method has higher hydrogen capacity (70.5 kgHm^{-3}) than pressurized hydrogen technique (25 kgHm^{-3}), but the disadvantages are the low efficiency of the liquidizing process and need extra insulation equipment for protecting the process. For the transportation application, the cryogenic tank could cause the safety concern because it has a possibility of explosion during the usage.

High pressure hydrogen storage technique is the most common method of hydrogen storage in industry, which using high pressure technique (350 or 700 bar) to store hydrogen gas into the high pressure steel cylinder. The requirements of this technique only need a compressor and a pressure vessel, which is a simple and cheapest method. Low volumetric capacity is the major disadvantage. Under 700 bar condition, the gravimetric capacity is 4.5 wt% which has a small distance from reaching the DOE 2010 target. Another disadvantage is the cylinder could be leak or explode during the inappropriate operation. If high pressure hydrogen storage technique applies in the vehicle, the issues of the extra weight and the limited space are unfavorable to the manufactures or the consumers.

Compare chemical-bond hydrogen storage technique, also can be called “hydride”, with others, and it has more advantages such as high volumetric energy density, simply

reaction, safety and more purely hydrogen after the reaction. However, in many investigations of chemical-bond hydrides, some hydrides have hydrogen storage capacity lower than 2 wt%, such as LaNi_5 , TiFe , and TiMn_2 [3] which are not the appropriate hydrogen resource candidates. In other words, not whole hydrides can be used in hydrogen generation application. Therefore, chemical-bond hydrogen storage can use the chemical catalyst to adjust hydrogen generation rate. For example, Sodium borohydride (NaBH_4) is the first candidate of hydrogen energy resource since 2000. NaBH_4 has a 10.8 wt% hydrogen storage capacity. It has high theoretical efficiency of hydrogen generation, but the byproduct, sodium metaborate, could be re-hydrogenated back into NaBH_4 during the reacting process, which decreases the amount of hydrogen generation. It seems to be a good potential hydrogen resource for the automotive application. However, the byproduct, sodium metaborate, has a corrosive nature and other drawbacks so US Department of Energy (DOE) [1] announced No-Go for the decision that NaBH_4 is not suitable for hydrogen generation field.

1.4 Literature Review

In recent years, hydrogen energy becomes one of the top alternative energy resources, and many hydrogen generation methods have been discovered from different research areas. Uan et al. [4] used the magnesium scraps from recycling Mg products in sodium chloride (NaCl) to generate hydrogen. He mentioned that only high-grade Mg products can be processed for the hydrogen generation experiments because they have high electrochemical activity and low corrosion resistance. Moreover, the low-grade Mg products may include some metals that can increase the corrosion resistance, such as iron, nickel, and copper. In this paper, the hydrogen generation rate was 432.4 ml/min over 8100 sec. In 2009, Uan et al. [5] used the three types of the low-grade Mg scraps (LGMS) with the different ratio of the citric acid and seawater to generate hydrogen. After every 30 min for refreshing seawater and citric acid added, the total amount of producing hydrogen is 70 liter in 100 minutes. Yu et al. [6] investigated the low-grade Mg scraps with organic acid in hydrogen generation experiments. Yu used the different ratio of the citric acid and the acetic acid separately in NaCl to react with the low-grade Mg scraps.

Sodium borohydride (NaBH_4) was the first potential hydrogen energy source that caught much attention from the society in 1950s. NaBH_4 has a high hydrogen storage capacity than other materials mostly. However, if the concentration of the byproduct,

sodium metaborate, becomes higher during the reaction, it will obstruct the diffusion of NaBH_4 ion to the catalyst and then decrease the hydrogen generation amount, which the real efficiency of hydrogen generation is lower than the theoretical prediction. The liquid acids can accelerate the hydrogen generation process so Akdim et al. [7] used the two kinds of acid, hydrochloric acid (HCl) and acetic acid (CH_3COOH), for the catalyst to release hydrogen from NaBH_4 . The result showed that the CH_3COOH has the same efficiency as HCl and has more relatively green than HCl. Moreover, Yu et al. [8] had the same conclusion that although both HCl and acetic acid can accelerate the reaction, the acetic acid has the ability of the hydrolysis more powerful than HCl in the same pH value. Yu also found that the higher temperature and concentration of NaBH_4 can increase the reaction rate in the hydrogen generation experiments. Kim et al. [9] tried to use a new catalyst, sodium hydrogen carbonate (NaHCO_3), for generating hydrogen from NaBH_4 with connecting PEMFC system. In the different concentration of NaHCO_3 , the highest power density is 128 mW/cm^2 at 0.7 V. Fan et al. [10] mixed NaBH_4 with AlLi to become a solid-type mixture. The amount of hydrogen generation can be adjusted by the weight of AlLi/ NaBH_4 , the content of Li, the concentration of CoCl_2 solution and the hydrolysis temperature. The experiments showed that 1674 ml hydrogen can be generated by Al-20wt% Li/ NaBH_4 and the solution of 2.5 wt% CoCl_2 .

Huang et al. [11] hydrolyzed water to generate hydrogen with aluminum. The hydrolysis reaction used mercury and zinc amalgam as a catalyst. The experiments gained the highest hydrogen generation rate was $43.5 \text{ cm}^3/\text{h}\cdot\text{cm}^2$ at 65°C . Mercury has high activation energy (74.8 KJ/mole) than zinc amalgam (43.4 KJ/mole), which zinc amalgam can hydrolyze water with aluminum to generate hydrogen rapidly.

The previous metal hydrides have been developed for hydrogen generation in a long time, but researchers want to find more green hydrogen resource, which has better characteristics of environmental friendly and safety. Magnesium hydride (MgH_2) has been chosen for another potential hydrogen resource. In 1912, Jolibois et al. [12] was the first one who discovered it from the pyrolysis of ethyl magnesium iodide in vacuum. Magnesium hydride also has a strong ability of carrying hydrogen than most metal hydrides.

The ball milling process is one of the most common techniques to improve the hydrogen storage properties. Grosjean et al. [13] [14] produced hydrogen from using magnesium or magnesium hydride to react with water or potassium chloride (KCl) solution. Grosjean concluded that Mg+10 % Ni composite material during the ball milling process after 30 min has the best performance. Moreover, KCl solution has more effective influence to react with magnesium than magnesium hydride. Hong et al. [15]

mentioned that $\text{MgH}_2 + 5\% \text{MgO}$ powder has the highest amount of hydrogen generation, 0.7g H_2 for 3 min and 0.97g H_2 for 60 min, after milling 2 hours, which compared with other materials such as Al, TiH_2 , unmilled Mg, milled Mg, unmilled MgH_2 , and milled MgH_2 . Maddalena et al. [16] mixed the nanozised MgH_2 , which obtained by the ball milling process, with Mg, Nb_2O_5 addition, and Mg-Ni-Fe ribbons separately for enlarging the absorption and desorption kinetics.

In the previous investigations, acid solutions have been utilized as the catalyst to accelerate the reaction during the hydrogen generation experiments. Kushch et al. [17] used citric acid to increase hydrogen amount from Mg. The results indicated that 1.0846 kg of magnesium and 5.7155 kg of citric acid can generate 1 m^3 of hydrogen. Kushch mentioned that the whole hydrogen generation process is more economical and safe because citric acid is a weak organic acid, which has been used widely in the food industry for a long time. In addition, citric acid can be totally solute into water, which can steady the pH value for controlling hydrogen production.

1.5 Research Objective

From the literature review, metal hydrides become the new hydrogen resource recently. For example, sodium borohydride (NaBH_4) has been investigated in many ways before the No-Go decision made by US Department of Energy (DOE). Although the hydrogen storage capacity of NaBH_4 can reach the DOE's target, its hydrogen generation progress and regenerate process are not suitable for portable or on-board application, which the byproduct, sodium metaborate, is a corrosive material that is not safe for the regular operation. Lithium aluminum hydride (LiAlH_4) [18] is another potential hydrogen resource. However, LiAlH_4 needs the high temperature (above 200°C) environment for processing the dehydrogenation comparatively. Magnesium hydride (MgH_2) is a non-corrosive material, and can be release hydrogen in room temperature. MgH_2 has high hydrogen capacity than most hydrides. Hence, if researchers can control the weight of MgH_2 , the concentration of the catalyst, and the shape of MgH_2 , MgH_2 will become the one of the best candidates of hydrogen generation. However, MgH_2 has the low dehydrogenation with water so how to release hydrogen become the crucial problem. From the previous investigations, acids [7][17] have already been proved that can increase hydrogen generation rate. Citric acid is one of acids that also can accelerate hydrogen generation rate but its characteristic of weak acid that needs a long time to release hydrogen from MgH_2 .

The purposes of the present study are to design a safe environment for the hydrogen generation experiments and choose a catalyst which can increase hydrogen generation rate and also can protect environment.

CHAPTER 2 Theoretical Principle and Experimental

Method

2.1 Magnesium Hydride

Magnesium hydride (MgH_2) is one kinds of chemical hydride, which is a compound that bonding with metal and hydrogen. Fig. 2 shows that the metallic hydrides with the weight of hydrogen, such as Lithium borohydride (LiBH_4), Lithium hydride (LiH), Lithium aluminum hydride, and so on. For example, Lithium borohydride (LiBH_4) [18] has highest hydrogen storage than other metallic hydride. However, LiBH_4 has the disadvantages, such as high thermodynamic desorption, irreversibility, and high pressure requirement, which need more investigations to improve the reactive process.

In 1912, scientists obtained magnesium hydride combined with ethylene and magnesium halide after the pyrolysis of ethyl-magnesium halide.



From equation 1, magnesium hydride is a compound, not a pure material. Therefore, how to increase the purity of magnesium hydride becomes another question. Finally, pure magnesium hydride was produced by pyrolysis of diethyl-magnesium at 400 K in vacuum environment until 1951.



The large amount of magnesium hydride was produced by Mg to react with hydrogen at 200 bar and 850K with the MgI_2 as the catalyst.



Moreover, MgH_2 has high energy density, theoretical hydrogen capacity, low price, and Mg is third common material in nature. That is the reasons why MgH_2 become the one of the popular hydrogen candidates.

2.2 The Principle of Hydrogen Generation

Hydrogen can be produced by reacting MgH_2 with water. Equation (4) shows the hydrolysis reaction.



$$\Delta H_r = -277 \text{ kJ/mole}$$

The exothermic behavior happened during the hydrolysis reaction. When MgH_2 meet with water, it releases hydrogen slowly and forms magnesium hydroxide (Mg(OH)_2) which is a non-toxic compound and a flame retardant [22]. According to equation (4), the theoretical hydrogen capacity is 7.6 wt% after 1 mole MgH_2 react with 2 moles water to generate 2 moles H_2 . Moreover, if MgH_2 become main hydrogen resource for PEMFC, the produced water from electrochemistry reaction is recycled back to MgH_2 , which has higher theoretical hydrogen capacity (15.2 wt%).

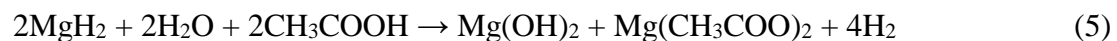
$$\text{Theoretical hydrogen capacity without water} = \frac{(\text{molecular weight of } 2\text{H}_2)}{(\text{molecular weight of } \text{MgH}_2)}$$

$$\text{Theoretical hydrogen capacity with water} = \frac{(\text{molecular weight of } 2\text{H}_2)}{(\text{molecular weight of } \text{MgH}_2) + (\text{molecular weight of } 2\text{H}_2\text{O})}$$

DOE set the targets of hydrogen storage (Table 3) that achieved hydrogen storage of 4.5 wt% in 2007 and 6 wt% in 2010. Magnesium hydride can satisfy with both requirements.

Magnesium hydride is a light gray crystalline compound that absorbs H_2 at high temperature above 320°C under normal pressure. The hydrolysis of MgH_2 is a very

slow reaction because the formation of magnesium hydroxide layer will cover on MgH₂ surface, which stop the hydrolysis reaction. Due to the concentration of the saturated solution is 1.9mg/L, magnesium hydroxide is hard be dissolved into water at room temperature. Thus, the utilization of catalysts has been used to accelerate the rate of hydrogen generation. In this paper, acetic acid has been chosen as a catalyst for increasing hydrogen generation rate. The following equation illustrates the hydrolysis reaction with using acetic acid.



Acetic acid (CH₃COOH, molar mass 60 g/mole) is a benign organic acid, which enrich the sour taste and pungent smell after adding into vinegar. Acetic acid has been used massively in the food industry [23].

In conclude, using MgH₂ with acetic acid as a catalyst for hydrogen generation has following advantages:

- ⌚ High efficiency of releasing hydrogen
- ⌚ Safe usage
- ⌚ Non-toxic byproduct
- ⌚ Highly adjustable concentration of acetic acid
- ⌚ Accelerate hydrogen generation process
- ⌚ Controllable hydrogen generation amount by injecting the requirement of

acetic acid

- ⌚ Environmental friendly for both materials

2.3 Introduction of Hydrogen Generation Apparatus

In this paper, the experimental apparatus include the three-necked round-bottom flask, the water bath, the liquid-gas separator, the flow meter and software. Fig. 3 depict the each component for the hydrogen generation experiment. The following sections will introduce the detail of the experimental apparatus and setting.

2.3.1 Hydrogen generation reactor

The drip-style three necked round-bottom flask has been used in hydrogen generation experiment. Fig. 4 shows that the left rim of the flask is a tunnel for hydrogen that generated from the reaction. The middle of the flask is a burette with a switch, which the solvent (acetic acid with water) has been filled before starting the experiment. The right rim of the flask connects with a thermometer.

2.3.2 Thermostatic Water Bath

In this paper, the function of thermostatic water bath (Onlyscience Co. Ltd) has been used for simulating the variable environmental temperature. The reason is to observe the influence of the different environmental temperature for hydrogen

generation. The temperature of thermostatic water bath (Fig. 5) has been set in 30°C, 40°C, 50°C and 60°C separately for the hydrogen generation experiments. Table 4 shows the detail of thermostatic water bath.

2.3.3 Liquid-Gas Separator

The flow from the left rim of three necked round-bottom flask includes vapor and hydrogen from the reaction. Therefore, in this paper, the purpose of the hydrogen generation experiment is to obtain hydrogen without vapor, so the function of chemical separator is that eliminate vapor before hydrogen entered into the flow meter. Fig. 6 (A) shows that the separator can condense vapor to become the liquid type of water. Fig. 6 (B) shows that the chemical separator has been put between the first separator and the flow meter. The chemical separator can absorb remaining vapor after hydrogen pass through the first separator. The two types of separator can guarantee that hydrogen without vapor before entering into the flow meter.

2.3.4 Flow Meter

The most important purpose of the experiment is that how much hydrogen has

been generated from magnesium hydride with acetic acid. In this paper, the flow meter made by Fujikin Co. has been used to measure the amount of hydrogen generation. The principle of the flow meter uses the characteristic of different gas molecule has a different heat capacity to estimate hydrogen amount. For example, the heat capacity of hydrogen is $28.82 \text{ J/mole}\cdot\text{k}$ and Argon is $20.79 \text{ J/mole}\cdot\text{k}$. When hydrogen goes through the heat sensor in the flow meter, the software will use the temperature gradient to measure the mass flow rate and then collect and record the data every 10 data per second. Fig. 7, 8, and 9 show the flow meter, the principle of the flow meter, and the operating software. Table 5 is the specification of the flow meter.

2.3.5 Portable pH Meter and Precision Balance

Fig. 10 shows the portable pH meter (Milwaukee Co.) has been used after finishing every experiment. Magnesium hydride is a weak alkaline compound, which the weight of magnesium hydride can affect the pH value. In this paper, three different weight of magnesium hydride have been used for measuring hydrogen generation amount so the purpose of the portable pH meter is to record pH value for every experimental operating condition. Table 6 is the specification for the portable PH

meter.

The weight of magnesium hydride is the one of most critical parameters in hydrogen generation experiment. The digital readability of the precision balance (Fig. 11) that manufacture by Ohaus co is one in a thousand, which can precisely control the weight of magnesium hydride to decrease the experimental error. Table 7 is the specification of the precision balance.

2.4 Experiment Procedure

In this paper, every experiment should be following the procedure for maintaining the experimental accuracy. On the other hand, standard experiment procedure not only can reduce the experimental error but also increase the consistence of experimental results. Fig. 12 shows magnesium hydride sample (95.8 % MgH_2 , Biocoke Lab). In this paper, three different weight of MgH_2 (0.4g, 0.8, and 1.2g) has been used in the hydrogen generation experiments. When the heavy weight of MgH_2 contact with the high concentration of acetic acid (99.85% Acetic acid, DudaDiesel co., Fig. 13), the strong exothermic reaction could break the hydrogen reactor to endanger the operator. Hence, that is the reason why the small weight of MgH_2 has been chose in the experiments. Table 8 shows the experimental conditions for the hydrogen generation experiments. The experimental steps list in the following.

1. Fill water into the water bath and set up the temperature
2. Open the computer to connect with the flow meter
3. Connect the flow tunnel to each experimental apparatus
4. Turn off the switches on the three necked round-bottom flask and the separators
5. Follow the experimental requirement to measure the weight of magnesium hydride by the digital balance

6. Put magnesium hydride into the three necked round-bottom flask and connect the flow tunnel
7. Follow the experimental requirement to adjust the concentration of acetic acid and the ultra-clean water by the digital balance
8. Pour the acetic acid into the middle tunnel of the three necked round-bottom flask
9. Check up every connecting part.
10. Use camera to record the change of the reaction temperature
11. Click the recording button on the software and turn on the switch on the middle of the three necked round-bottom flask simultaneously
12. After one hour, click the stop button on the software and disconnect every experimental part
13. Put pH meter detector in the three round-bottom flask for 5 minutes
14. Pour the solvent into the waste container
15. Clean the three necked round-bottom flask
16. Repeat step 1 to step 15 for the different experimental conditions

Before starting the experiment, the operator needs to notice the temperature of the water bath and thermometer should be reached the setting temperature. The inside of the three necked round-bottom flask should be a dry environment, which

prevent the magnesium hydride react with the residual liquid before starting the experiment. Fig. 14 shows the vigorously reaction, which is the exothermic reaction or hydrogen generation phenomenon.

CHAPTER 3 Results and Discussions

3.1 Hydrogen Generation Experiments

The amount of hydrogen generation would be affected by many parameters. In this chapter, the results of hydrogen generation experiment will be discussed with the weight of MgH_2 , the concentration of acetic acid, and the effects of external temperature. Finally, the repeats of hydrogen generation experiments will be made for improving the experimental appearance.

Before starting the experiments, magnesium hydride has been examined by x-ray scattering (XRD) and energy dispersive x-ray spectroscopy (EDS). Fig. 15 shows the XRD detail of MgH_2 in $20\mu\text{m}$ and $5\mu\text{m}$. Fig. 16 shows the MgH_2 sample included magnesium hydride, magnesium, and magnesium hydroxide. Fig. 17 is that EDS detected the MgH_2 sample in different probe points. Fig. 17(a) shows that when the probe point put on the surface of MgH_2 sample, the compounds include magnesium hydride, magnesium, and magnesium hydroxide. Moreover, after the probe point put on the crake of MgH_2 sample (Fig. 17(b)), the compounds only include magnesium hydride and magnesium. The reason is that MgH_2 surface can form magnesium hydroxide after connecting with air.

3.1.1 The Effects of Different Concentration of Acetic Acid and Different Weight of MgH₂

According equation 4, the hydrolysis reaction is an exothermic reaction ($\Delta H_r = -277$ kJ/mole), which released high energy during the reacting process. Because of the high concentration of acetic acid with the high weight of MgH₂ would generated the hydrolysis reaction vigorously, the ratio of the concentration of acetic acid to the weight of MgH₂ should be decided carefully before starting the hydrogen generation experiments. In this paper, the concentration of acetic acid has been decided from 10 to 100 wt% (interval of 10 wt% per operating condition). The low weight of MgH₂ (0.4g, 0.8g, and 1.2g) has been used for compromising the high concentration of acetic acid.

Every operating condition for hydrogen generation experiment has been recorded in 60 minutes. The experimental results have been marked in different time range for the understanding easily. Fig. 18~21 show that the results of 0.4g MgH₂ with the different concentration of acetic acid in 30~60°C. The reason for the little different total amount of hydrogen generation with the various concentration of acetic acid is caused by the operating errors, such as the weight of MgH₂, the weight of the solution, or so on. However, these operating errors did not affect the hydrogen generation experiment a lot based on the results. In Fig. 18 (a), the amount of hydrogen

generation is approximately 0.07 liter with the different concentration of acetic acid.

In 60 wt% concentration of acetic acid, the experiment shows the fastest hydrogen generation rate. After 60 wt% concentration of acetic acid, the hydrogen generation rate will be slowed down. Fig. 18 (b) illustrates that the reactions of hydrogen generation have been finished in 10 minutes except the 100 wt% concentration of acetic acid, which completed the reaction at 30 minutes. According to the experimental results (Fig. 19, 20, and 21), 0.4gMgH₂ generated almost 0.07 liter hydrogen with the different temperature in 60 minutes. Fig. 22~25 show that 0.125 liter hydrogen has been produced by 0.8gMgH₂ with different concentration of acetic acid. 1.2g MgH₂ (Fig. 26~29) with different concentration of acetic acid has largest amount of hydrogen generation (0.185 liter) than 0.4gMgH₂ and 0.8gMgH₂.

From the above results, the similar trends have been observed, which the 60 wt% concentration of acetic acid has the fastest hydrogen generation rate in the different weights of MgH₂. In other words, the high concentration of acetic acid did not represent to get the better results of hydrogen generation, which MgH₂ needed to spend the longest time for producing hydrogen in 100 wt% concentration of acetic acid.

3.1.2 The Effects of Different External Temperature

The function of the water bath is to simulate the variable environment for hydrogen generation. In this paper, the operating temperature has been set up from 30~60°C. Fig. 30 (a)~(j) show the results of 0.4gMgH₂ reacted with the different concentration of acetic acid at 30°C, 40°C, 50°C, and 60°C. In general, the amount of hydrogen generation did not influenced explicitly by the external temperature except when 0.4gMgH₂ reacted with 100 wt% concentration of acetic acid. However, some experimental results might not have the same trends of hydrogen generation but the most results (not included 100 wt%) show the amount of hydrogen generation or the rate of hydrogen generation has the same curve from the four different temperatures. For example, Fig. 30 (a) show the 0.4MgH₂ with 10 wt% concentration of acetic acid at 60°C has the lowest amount of hydrogen generation and the fastest rate of hydrogen generation to compare with the same weight of MgH₂ with 10 wt% concentration of acetic acid at the different temperature. The reason can be ascribed to the operating errors (the weight of MgH₂) because the most results provided the evidences that the external temperature did not influenced easily the experimental results, which Fig. 30 (d) is the one of the best illustrations.

As mentioned earlier, the same experimental results can be obtained from 0.8gMgH₂ and 1.2gMgH₂ with the same concentration of acetic acid at the different

external temperature (Fig. 31 (a)~(j) and Fig. 32 (a)~(j)). If the operating errors can be reduced effectively from every experiment steps, the experimental results have the consistency of amount of hydrogen generation, such as Fig. 31 (a), (c), and so on.

The hydrogen generation is a standard hydrolysis process so hydrogen will be discontinued or delayed to generate from MgH_2 under the water deficient condition. Hence, when the solubility of MgH_2 reached the limitation in the high concentration of acetic acid, the results show the unstable phenomenon. For instance, that is the reason that three different weight of MgH_2 with 100% concentration of acetic acid (Fig. 30 (j), Fig. 31 (j), and Fig. 32 (j)) show the particular “S” shape of the curve in the results. Moreover, the same explanation can illustrate the hydrogen generation rate slowed down by increasing the concentration of acetic acid to 60 wt%.

3.2 Operating Error

The weight of MgH_2 has an influencing ability in the hydrogen generation experiments more than the concentration of acetic acid or the affection of external temperature. In Fig. 33~35, "RE" has been added behind the external temperature, which mean this operating condition has been redone, such as 50°C -RE, 40°C -RE, and so on. Fig. 33 (a) shows after finishing four kinds of the external temperature as an operating condition, 0.4gMgH_2 with 20 wt% acetic acid at 50°C only got 0.06 liter hydrogen from the experiment. However, other operating condition can get 0.07 liter hydrogen from the experiments. Based on the experimental accuracy, 0.4gMgH_2 at 50°C has been redone for providing the demonstration. From following every experimental step carefully, the amount of hydrogen generation can be corrected near to 0.7 liter. Fig. 33 (b) shows the results of 0.4gMgH_2 with 70 wt% at 40°C and 50°C also been improved by redoing the experiments.

During the repeating experiments, the importance of MgH_2 weight has been discovered. If the weight of MgH_2 can be controlled in 1/100 precisely, the amounts of hydrogen generation have the same value in different external temperature. In Fig. 34 and Fig. 35, the repeating experiments improved the results by adjusting the weight of MgH_2 in the same range.

CHAPTER 4 Conclusions

4.1 Conclusion

In this paper, the major purpose is to measure the amount of hydrogen generation from using magnesium hydride with the different concentration of acetic acid at the varying temperature. The hydrogen generation experiments has been set up with the three operating conditions, three different weights of MgH_2 , ten kinds of concentration of acetic acid, and the four individual temperature, within 60 minutes. The concentration of acetic acid can accelerate the hydrogen generation rate. From the results, when the concentration of acetic acid increased to 60 wt%, three different weights of MgH_2 have the highest hydrogen generation rate. However, after the solubility of MgH_2 reached a limitation, the experimental results showed the unstable phenomenon of hydrogen generation, which was the reactions have been slowed down or delayed. Moreover, the results of hydrogen generation did not significant affected in the varying temperature environment. Finally, the repeating hydrogen generation experiments provided the evidence that the weight of MgH_2 played an important role more than the concentration of acetic acid or the varying temperature in this series experiments.

4.2 Future Works

This paper investigates the solid type of magnesium hydride to react with acetic acid as the catalyst for the hydrogen generation experiments. The conclusions show the external temperature did not affect the results of hydrogen generation so this operating condition can be ignored in the future work. Therefore, there have some suggestions for the future work, such as milling MgH_2 , increasing the weight of MgH_2 , and testing the new acid as the catalyst. First, the amount of hydrogen generation will increase after the contact surface of MgH_2 increased by utilizing the milling process. Secondly, the large amount of hydrogen can be obtained from increasing the weight of MgH_2 . Third, trying to find the new catalyst to improve the rate of hydrogen generation. Furthermore, the exit of hydrogen generation can connect with proton electrolyte membrane fuel cell for testing the possibility of the portable application.

CHAPTER 5 References

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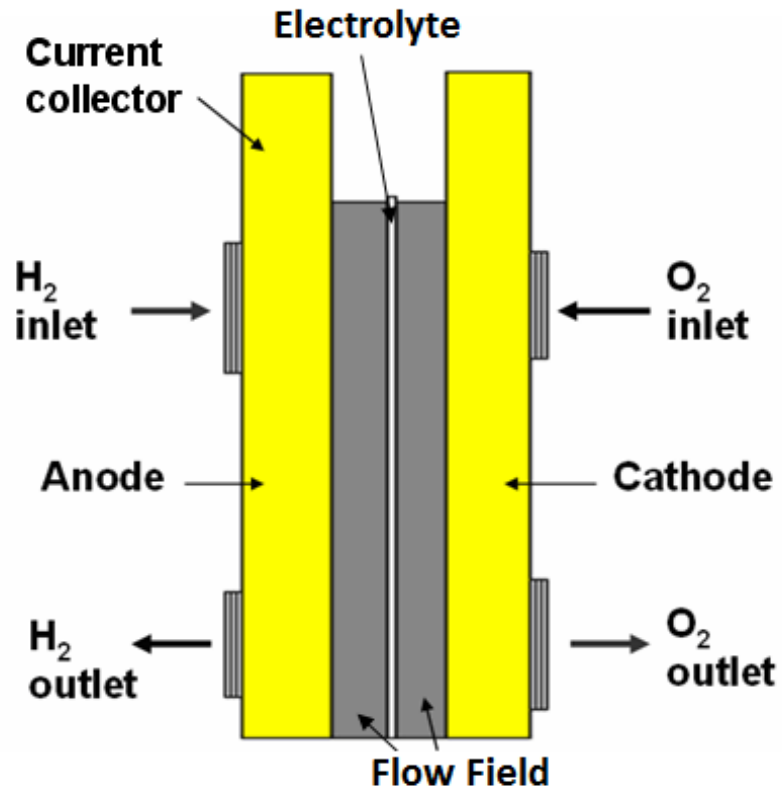


Fig. 1 Scheme of a Proton Electrolyte Membrane Fuel Cell (PEMFC)

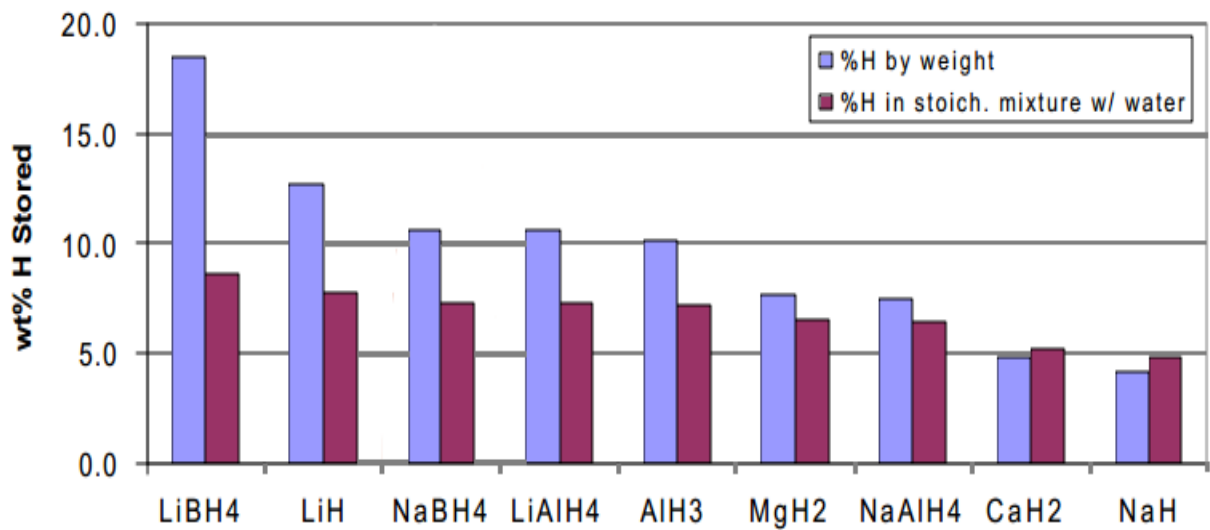


Fig. 2 Comparison of chemical hydride [20]

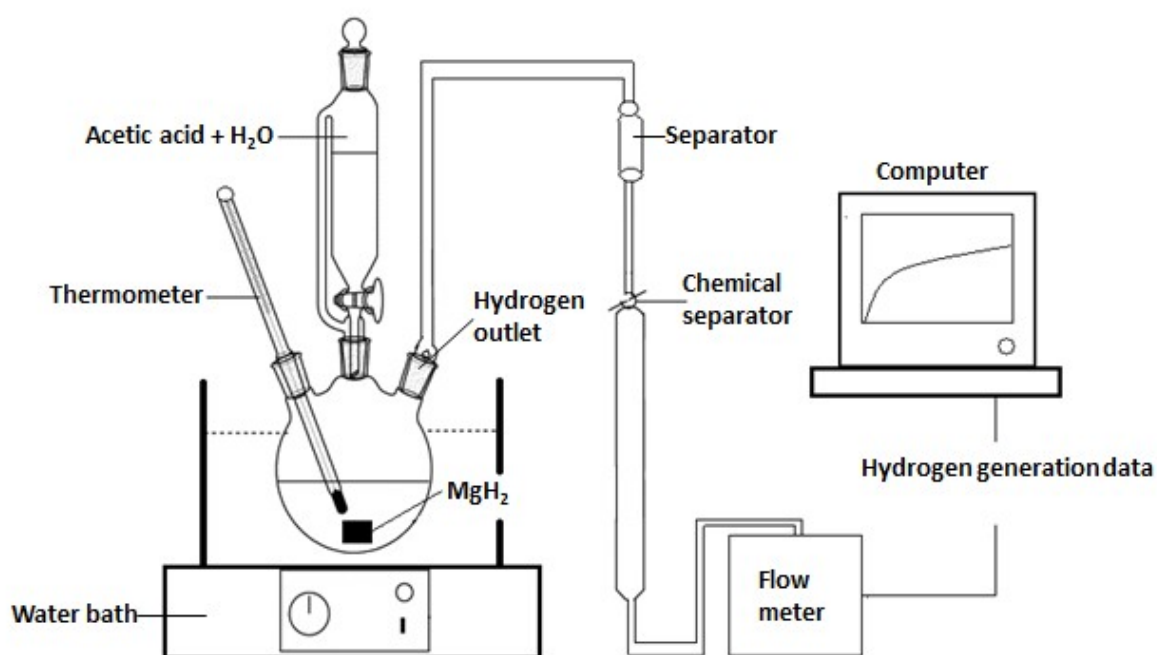


Fig. 3 Experiment apparatus

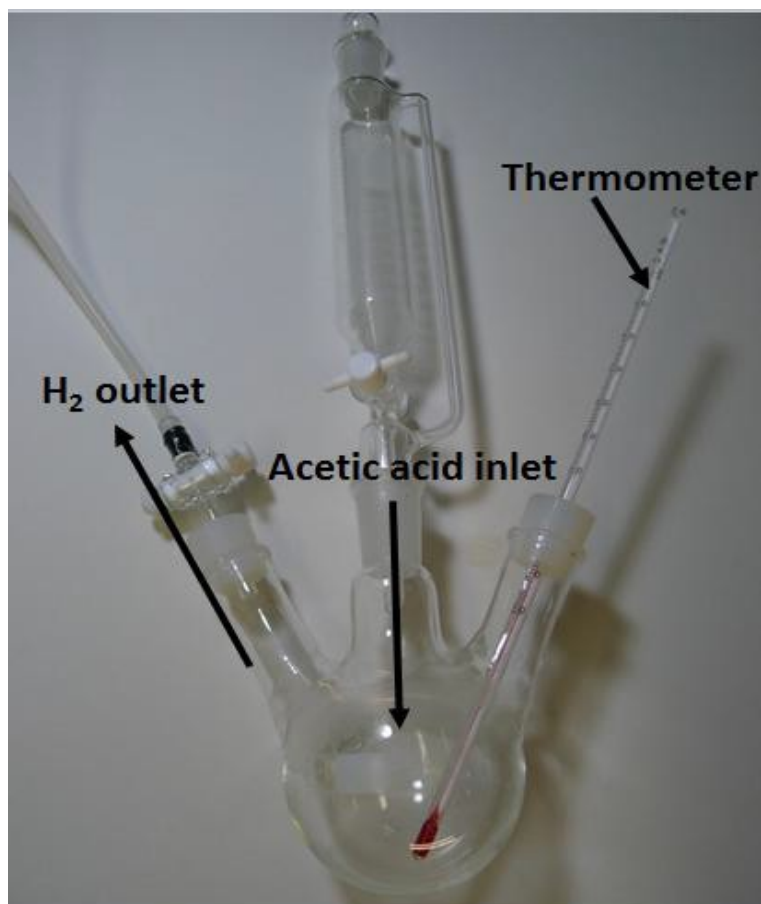


Fig. 4 The drip-style three necked round-bottom flask



Fig. 5 Thermostatic water bath

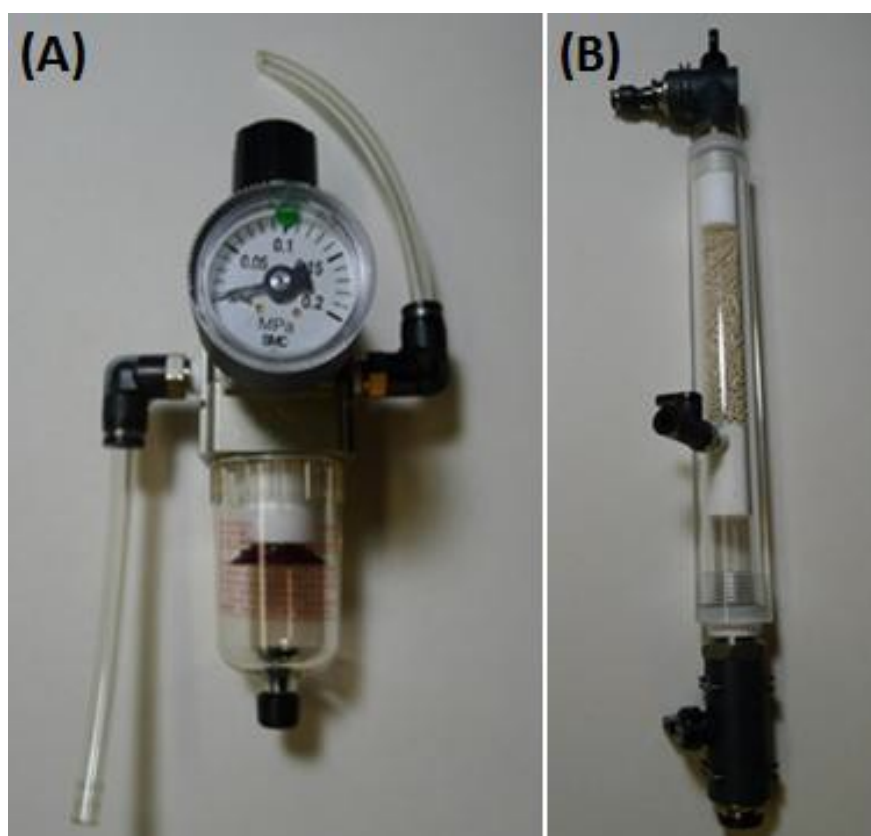


Fig. 6 (A) Separator (B) Chemical Separator



Fig. 7 Flow mete

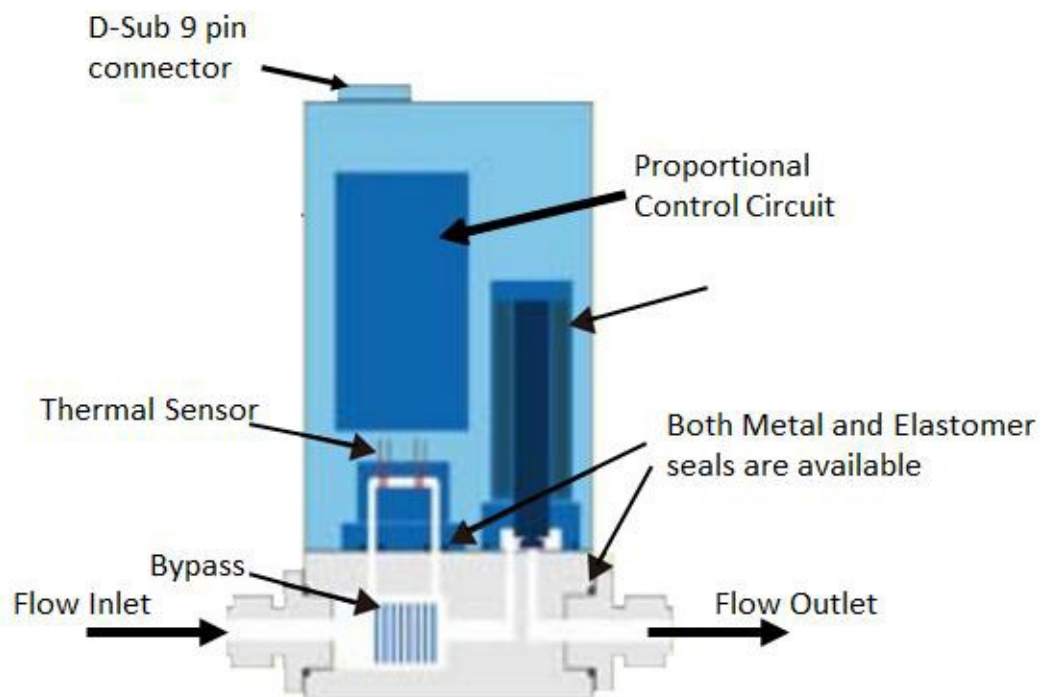


Fig. 8 The principle of flow meter

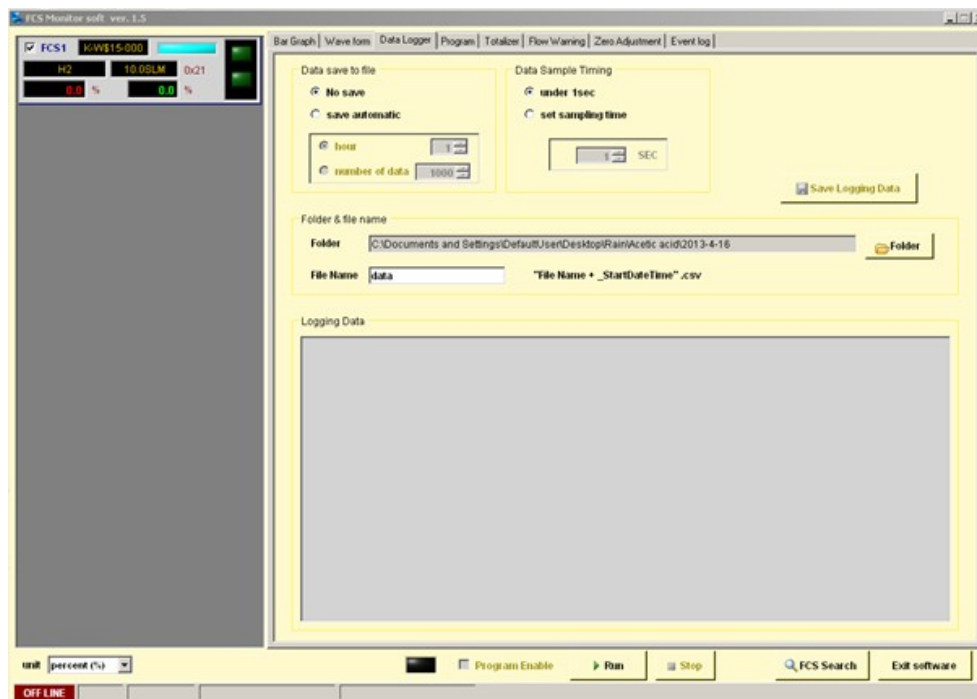


Fig. 9 Operating software

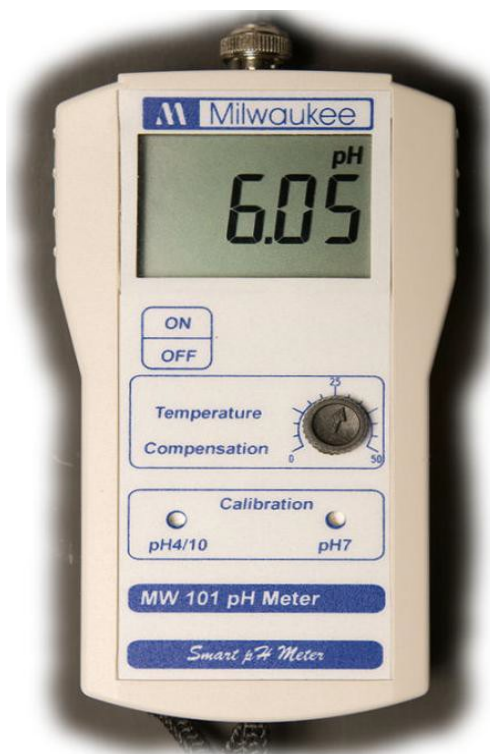


Fig. 10 The portable pH meter



Fig. 11 Precision balance



Fig. 12 Magnesium hydride sample



Fig. 13 Acetic acid

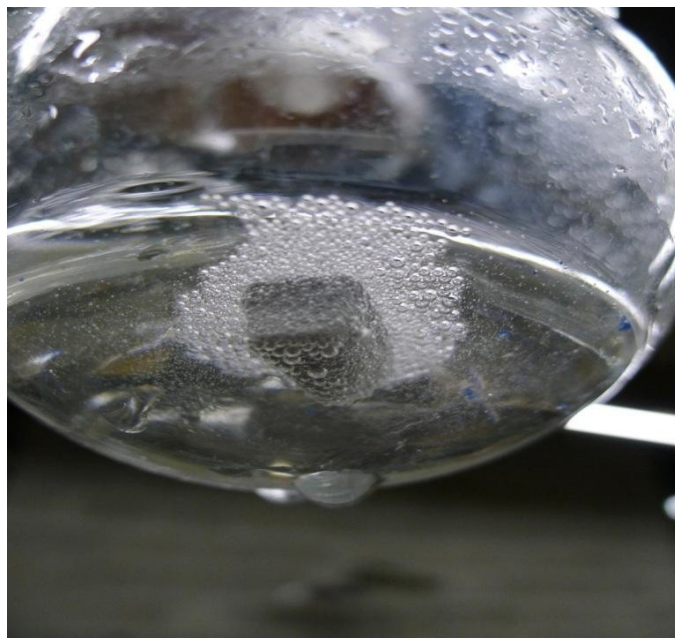


Fig. 14 Hydrogen generation phenomenon

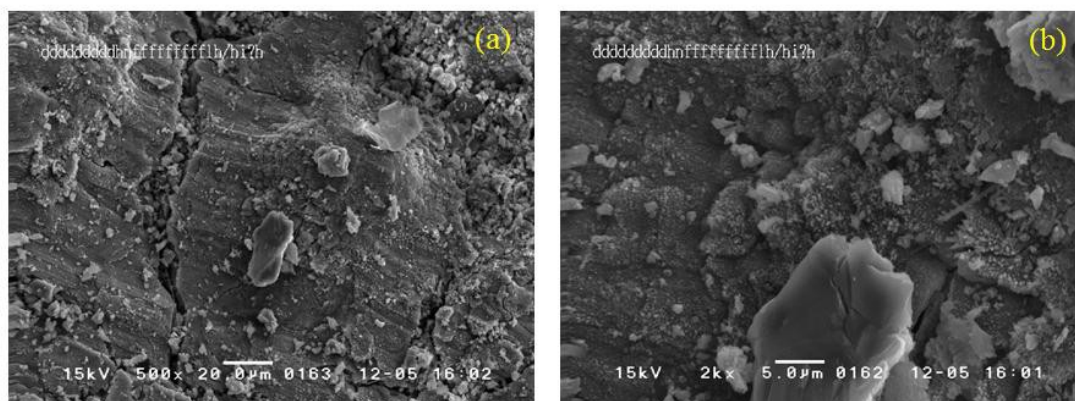


Fig. 15 XRD for MgH₂ (a) 20μm (b) 5μm

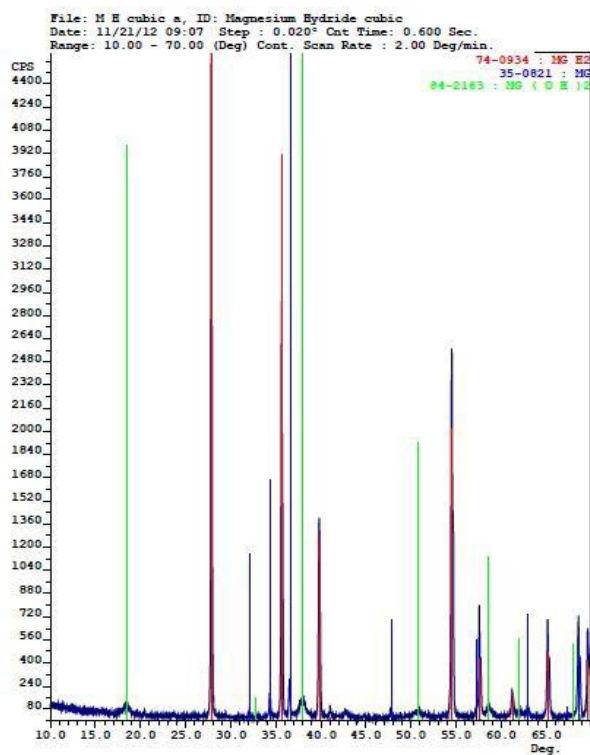


Fig. 16 XRD for MgH₂

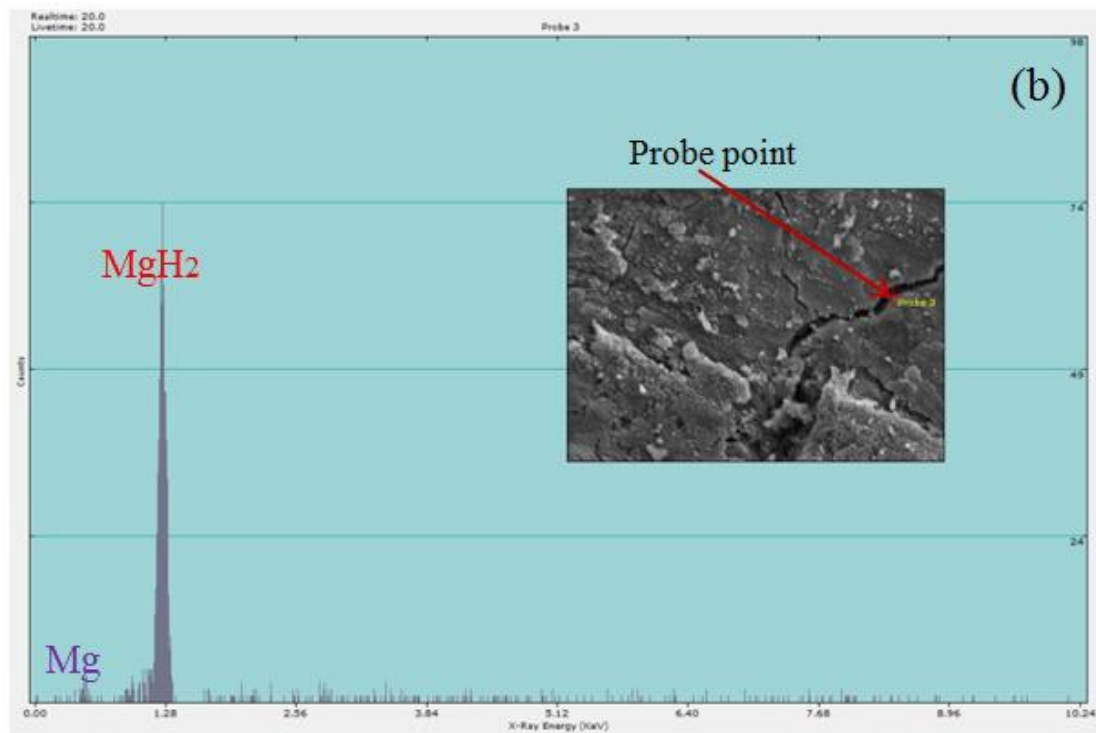
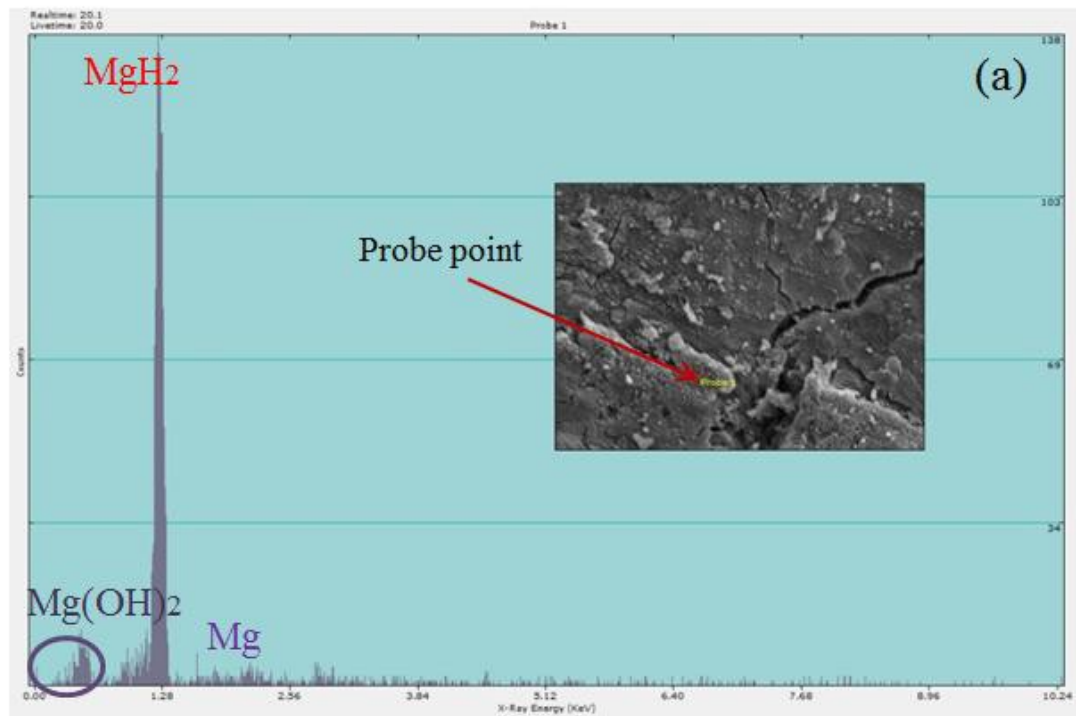


Fig.17 EDS for MgH₂ in the different probe points

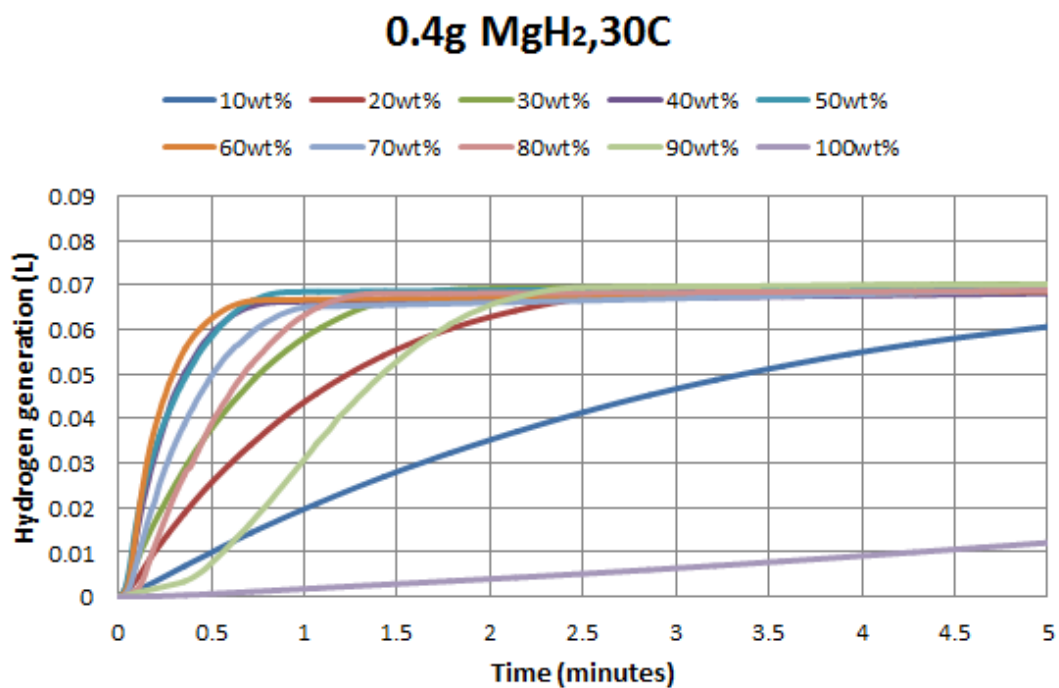


Fig. 18 (a) Different concentration of acetic acid with 0.4gMgH₂ at 30°C in 5 minutes

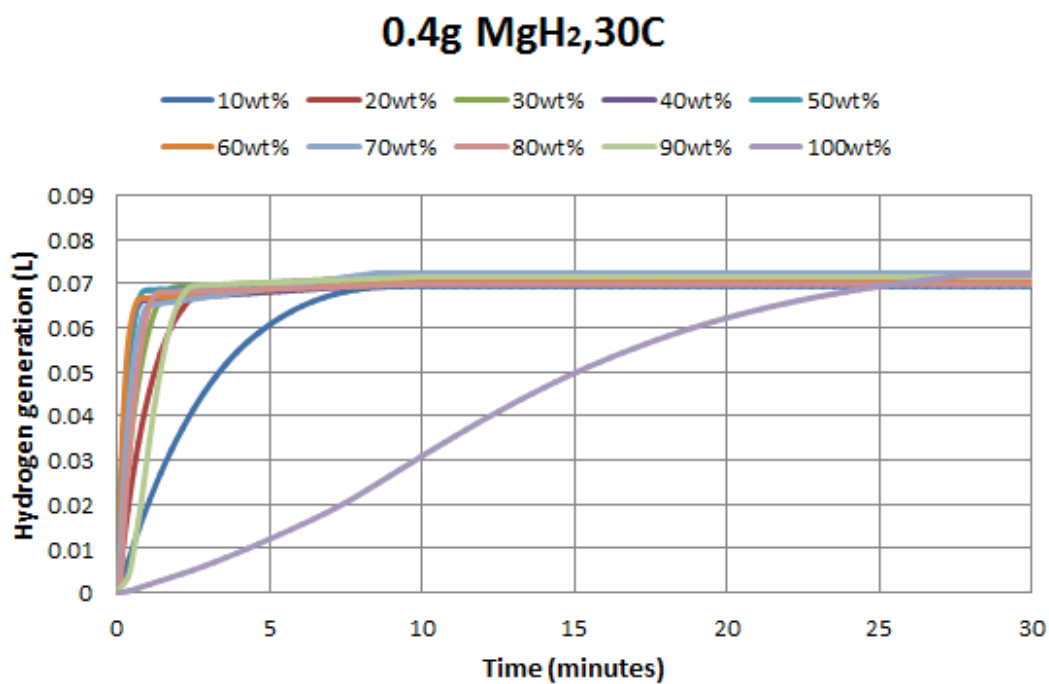


Fig. 18 (b) Different concentration of acetic acid with 0.4gMgH₂ at 30°C in 30 minutes

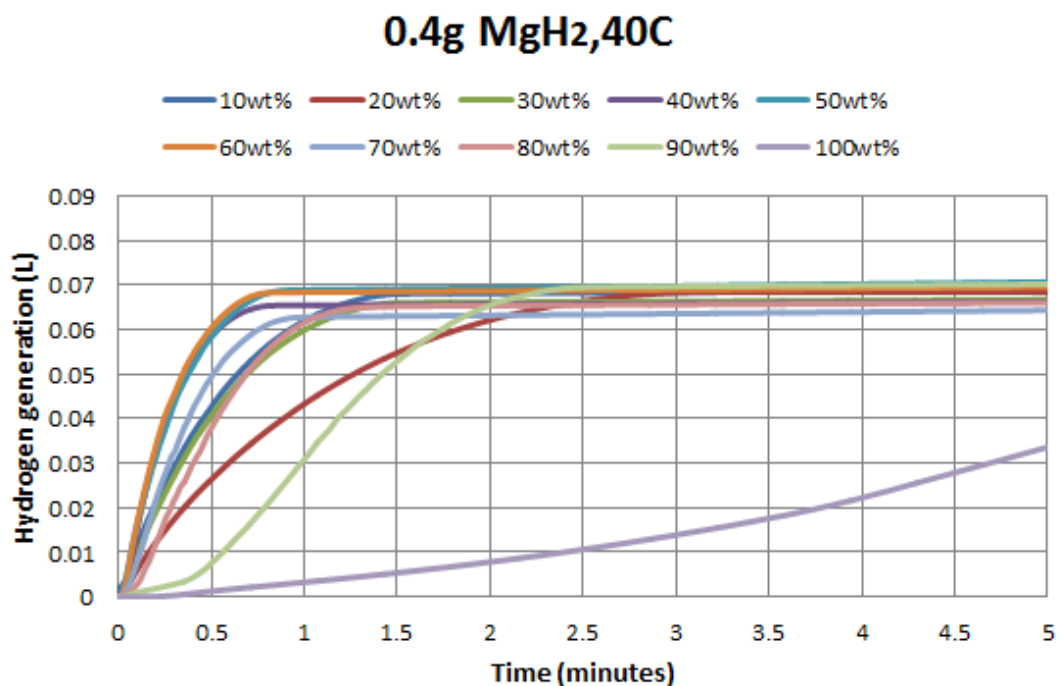


Fig. 19 (a) Different concentration of acetic acid with 0.4gMgH₂ at 40°C in 5 minutes

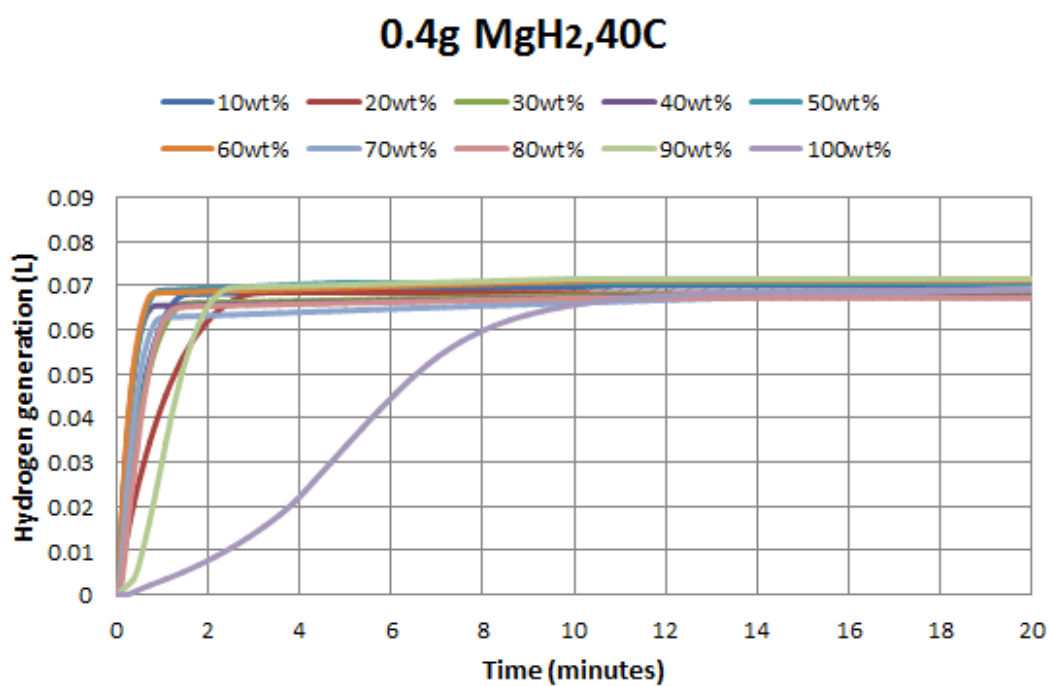


Fig. 19 (b) Different concentration of acetic acid with 0.4gMgH₂ at 40°C in 20 minutes

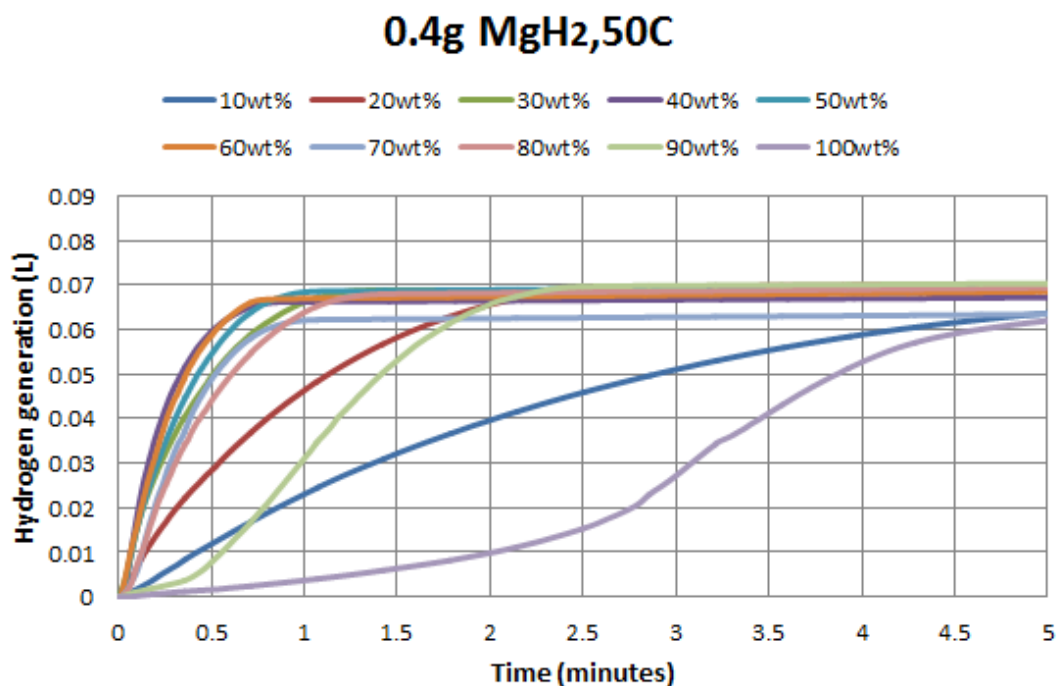


Fig. 20 (a) Different concentration of acetic acid with 0.4gMgH₂ at 50°C in 5 minutes

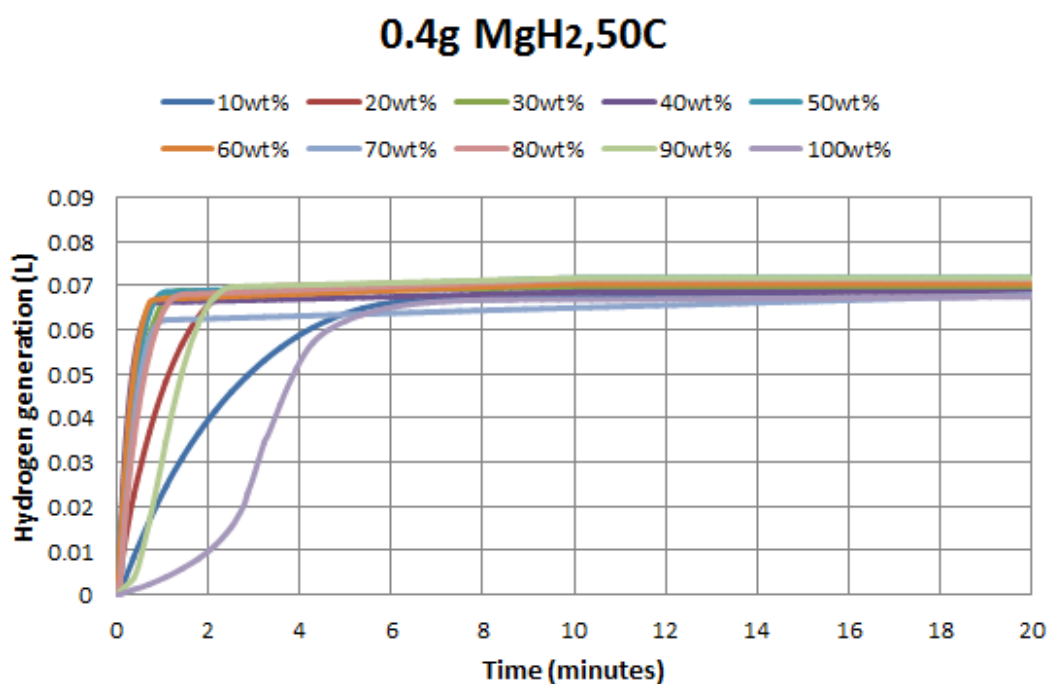


Fig. 20 (b) Different concentration of acetic acid with 0.4gMgH₂ at 50°C in 20 minutes

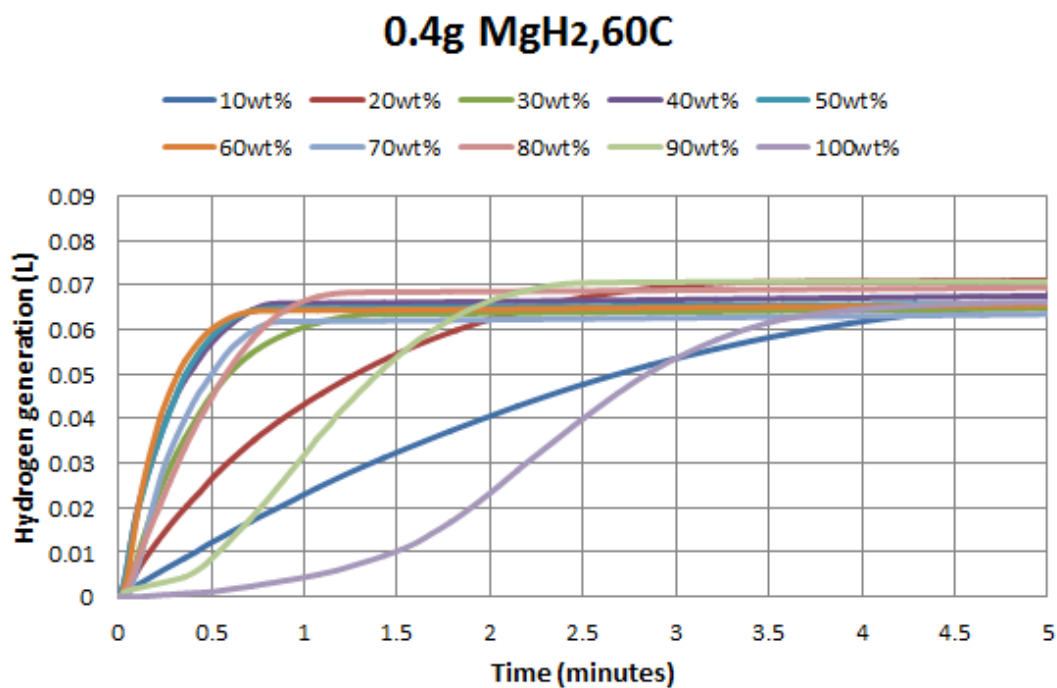


Fig. 21 (a) Different concentration of acetic acid with 0.4gMgH₂ at 60°C in 5 minutes

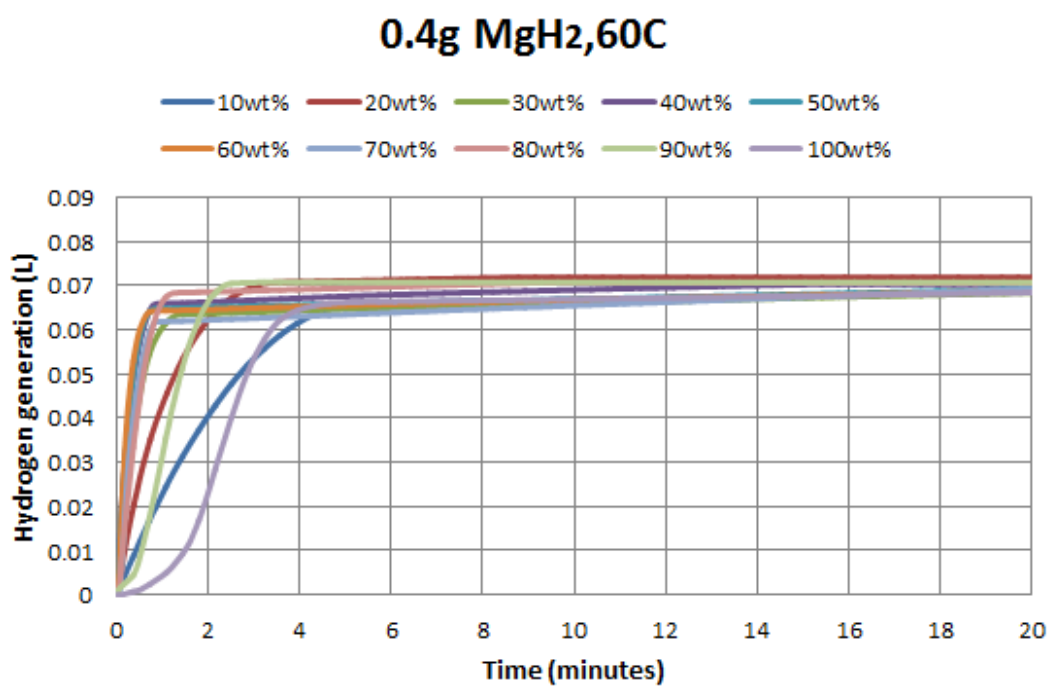


Fig. 21 (b) Different concentration of acetic acid with 0.4gMgH₂ at 60°C in 20 minutes

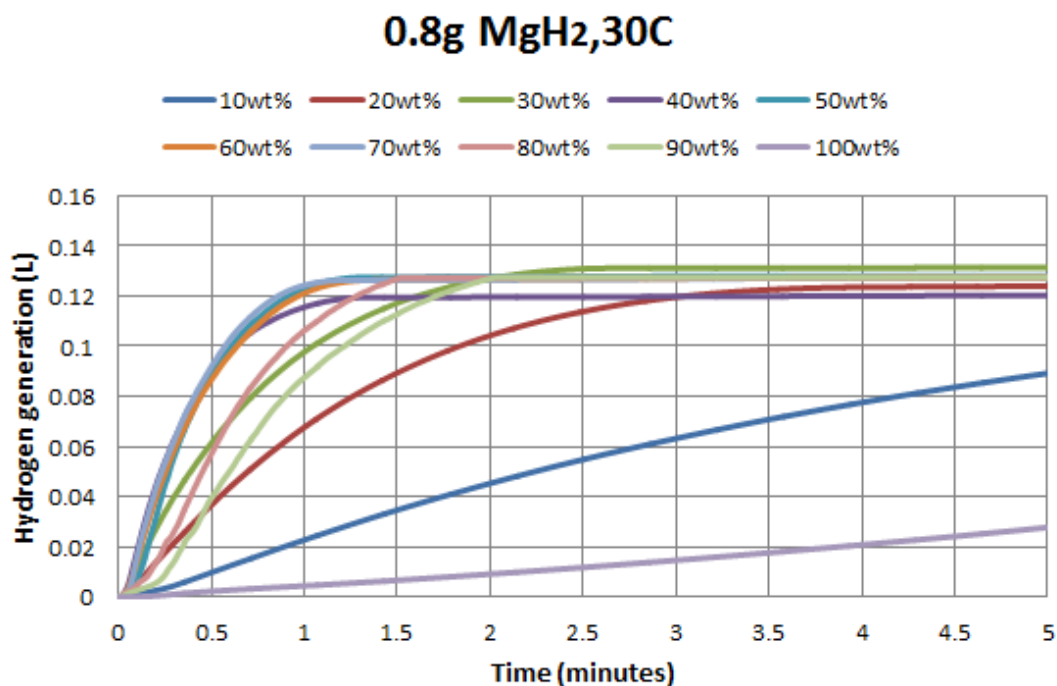


Fig. 22 (a) Different concentration of acetic acid with 0.8gMgH₂ at 30°C in 5 minutes

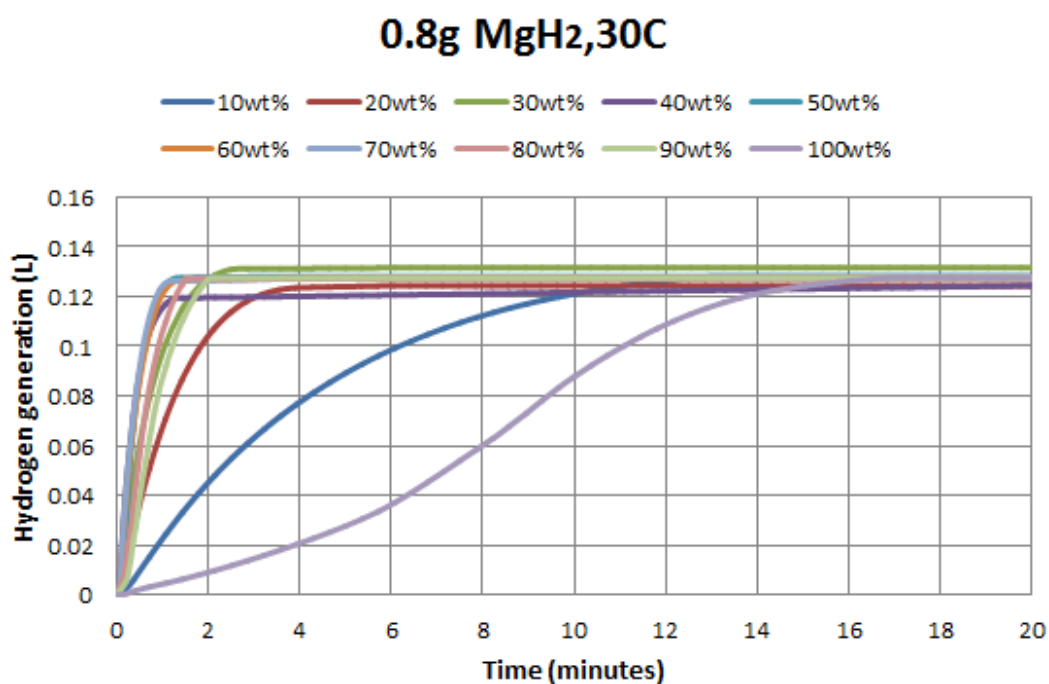


Fig. 22 (b) Different concentration of acetic acid with 0.8gMgH₂ at 30°C in 20 minutes

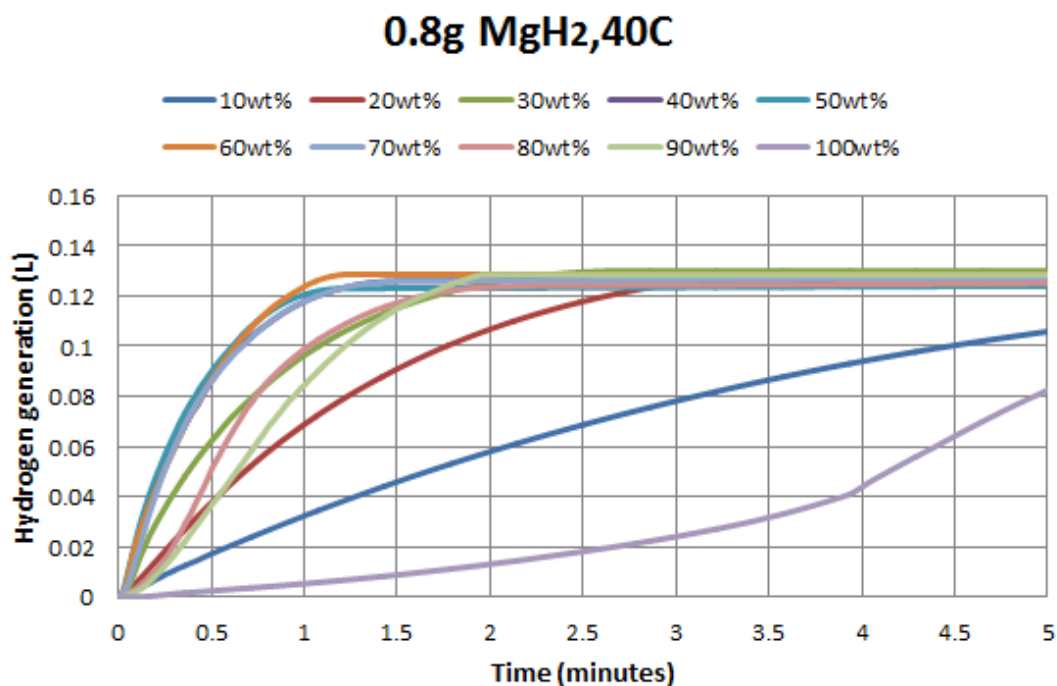


Fig. 23 (a) Different concentration of acetic acid with 0.8gMgH₂ at 40°C in 5 minutes

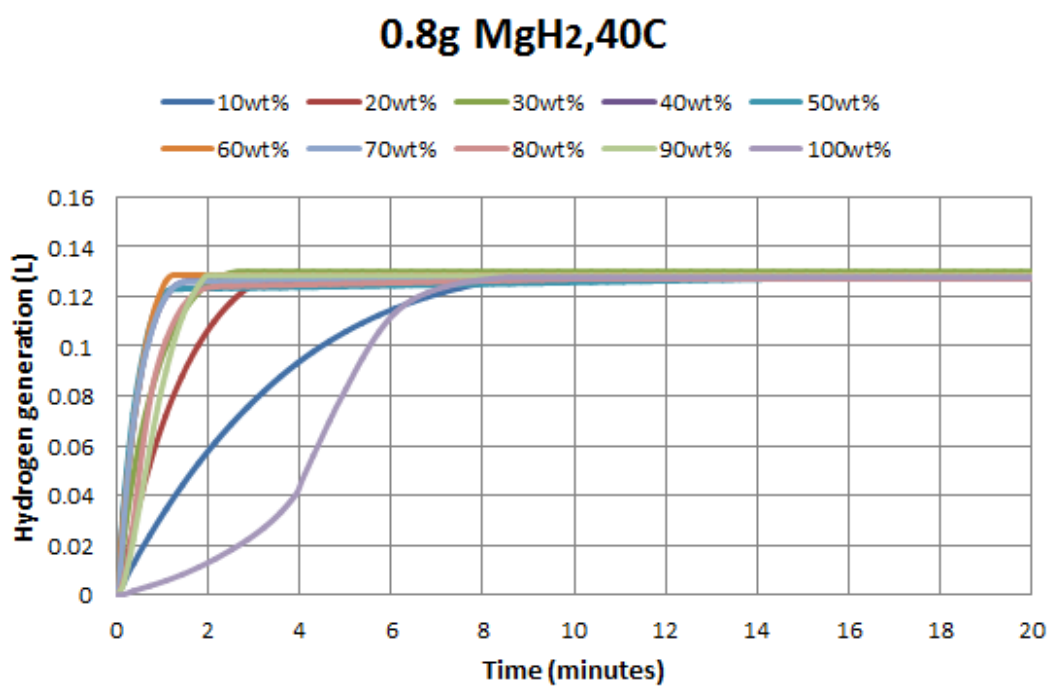


Fig. 23 (b) Different concentration of acetic acid with 0.8gMgH₂ at 40°C in 20 minutes

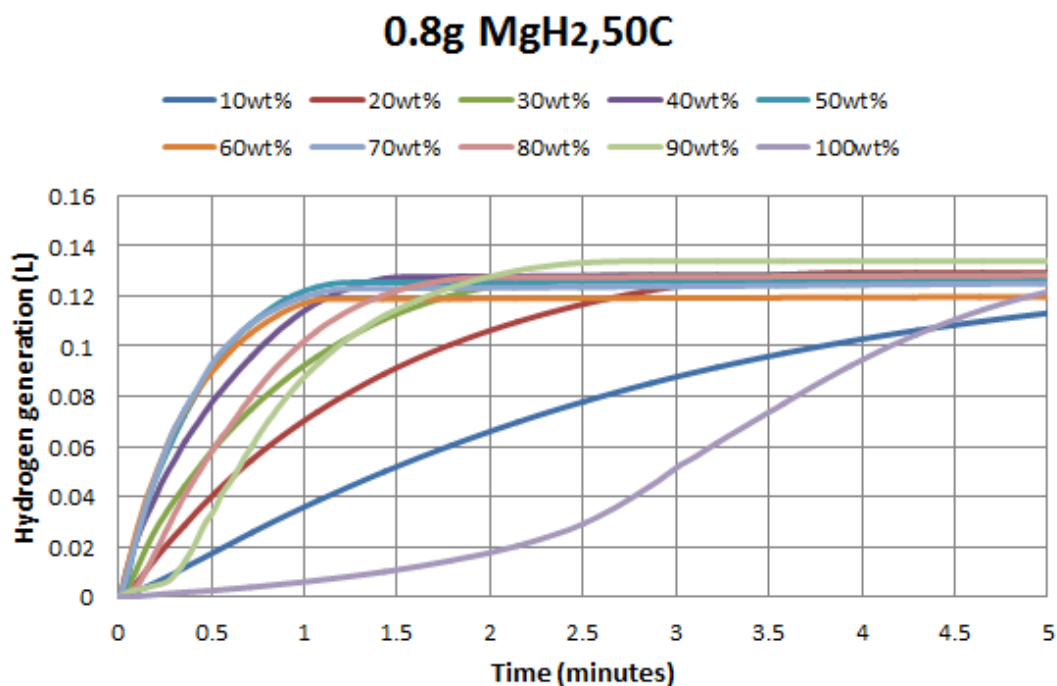


Fig. 24 (a) Different concentration of acetic acid with 0.8gMgH₂ at 50°C in 5 minutes

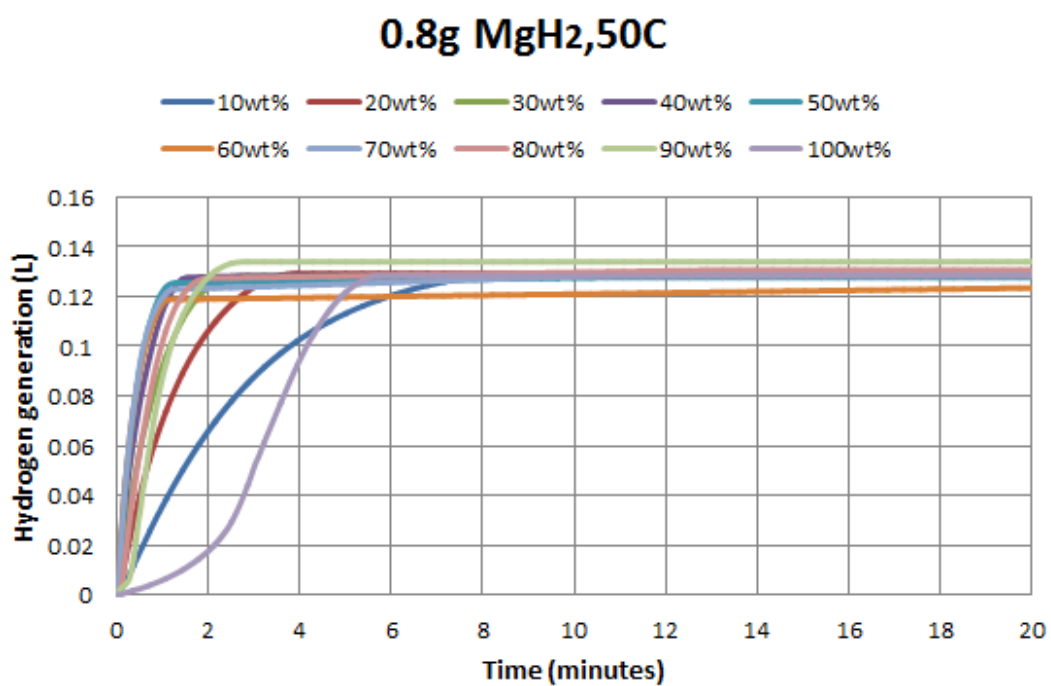


Fig. 24 (b) Different concentration of acetic acid with 0.8gMgH₂ at 50°C in 20 minutes

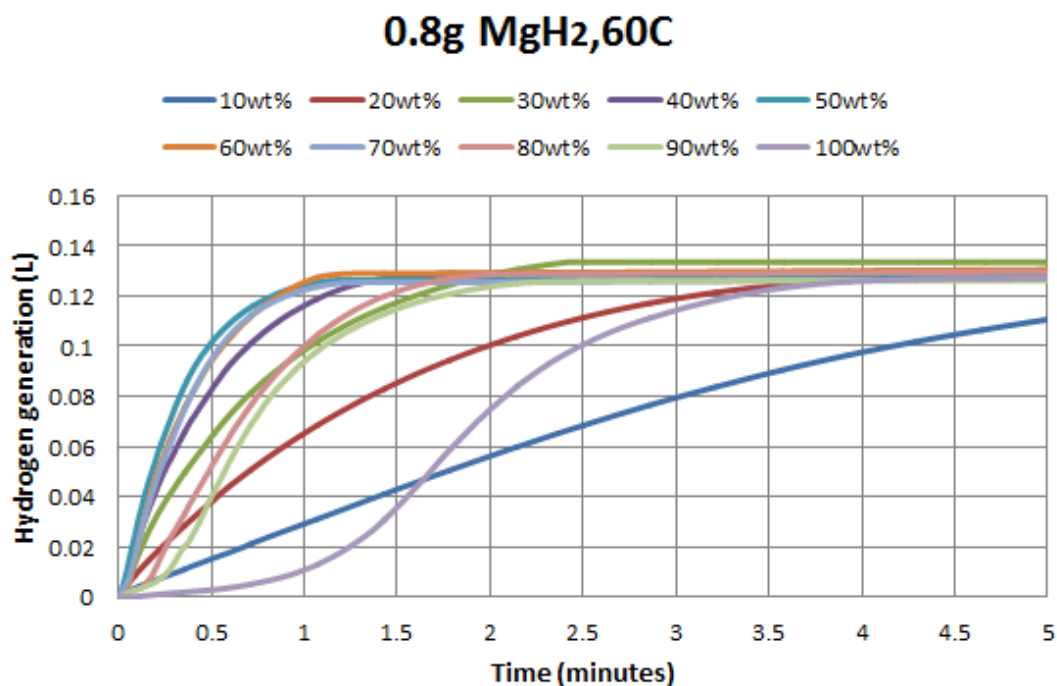


Fig. 25 (a) Different concentration of acetic acid with 0.8gMgH₂ at 60°C in 5 minutes

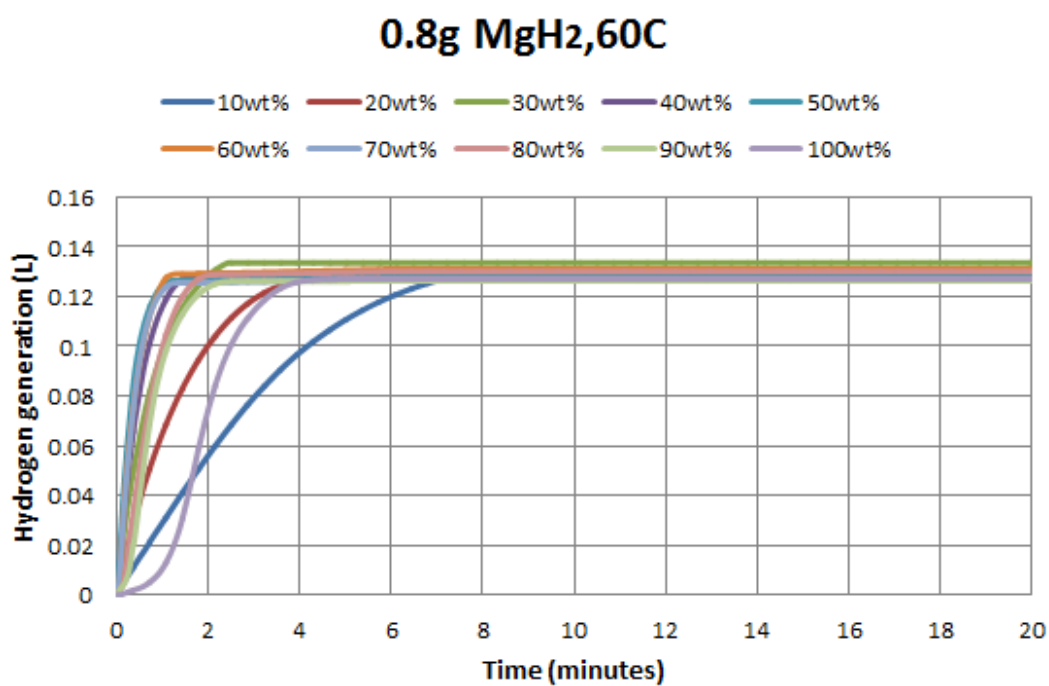


Fig. 25 (b) Different concentration of acetic acid with 0.8gMgH₂ at 60°C in 20 minutes

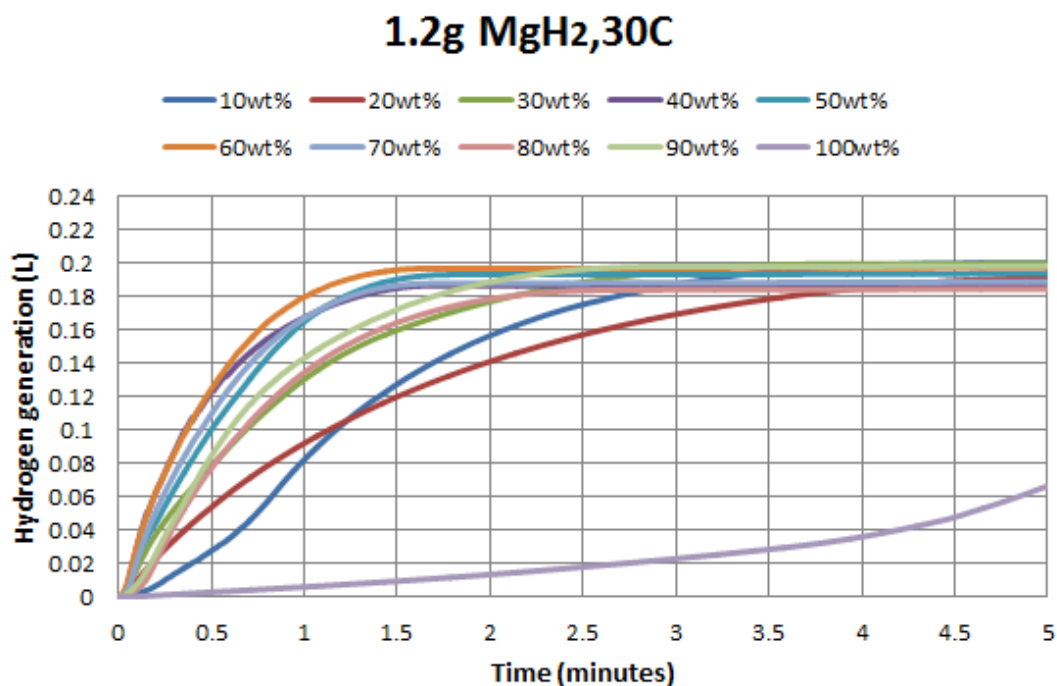


Fig. 26 (a) Different concentration of acetic acid with 1.2gMgH₂ at 30°C in 5 minutes

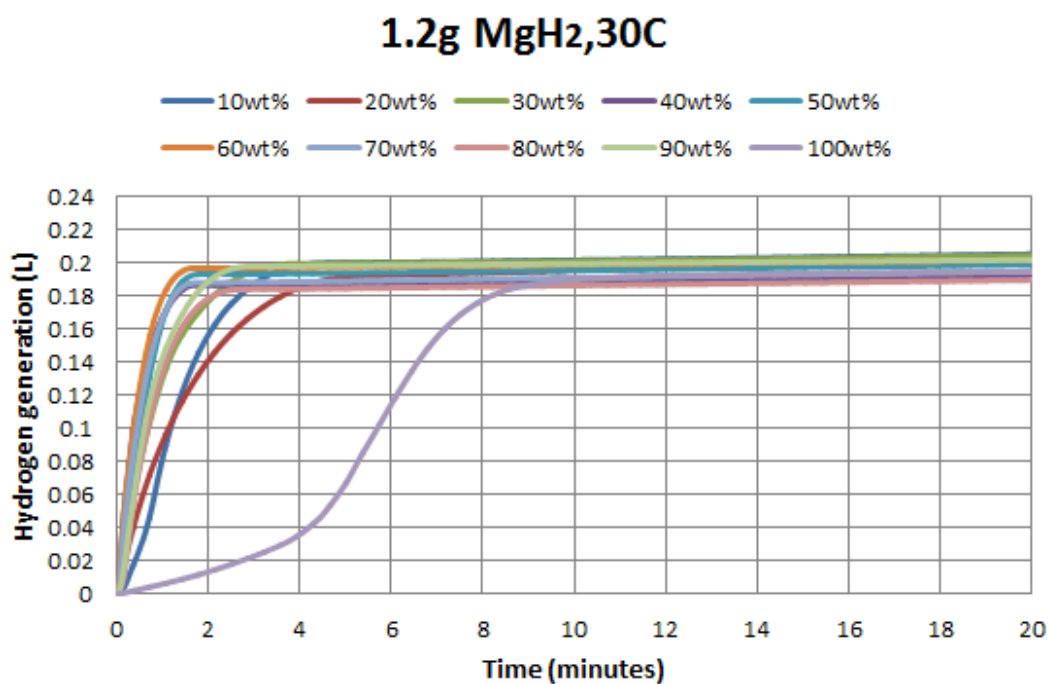


Fig. 26 (b) Different concentration of acetic acid with 1.2gMgH₂ at 30°C in 20 minutes

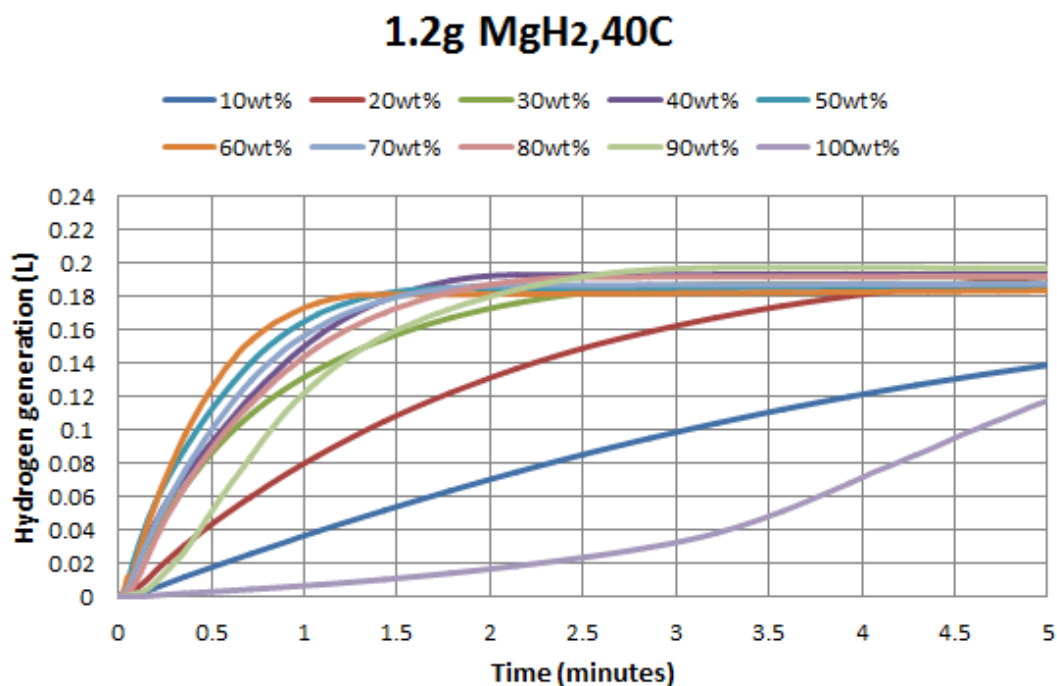


Fig. 27 (a) Different concentration of acetic acid with 1.2gMgH₂ at 40°C in 5 minutes

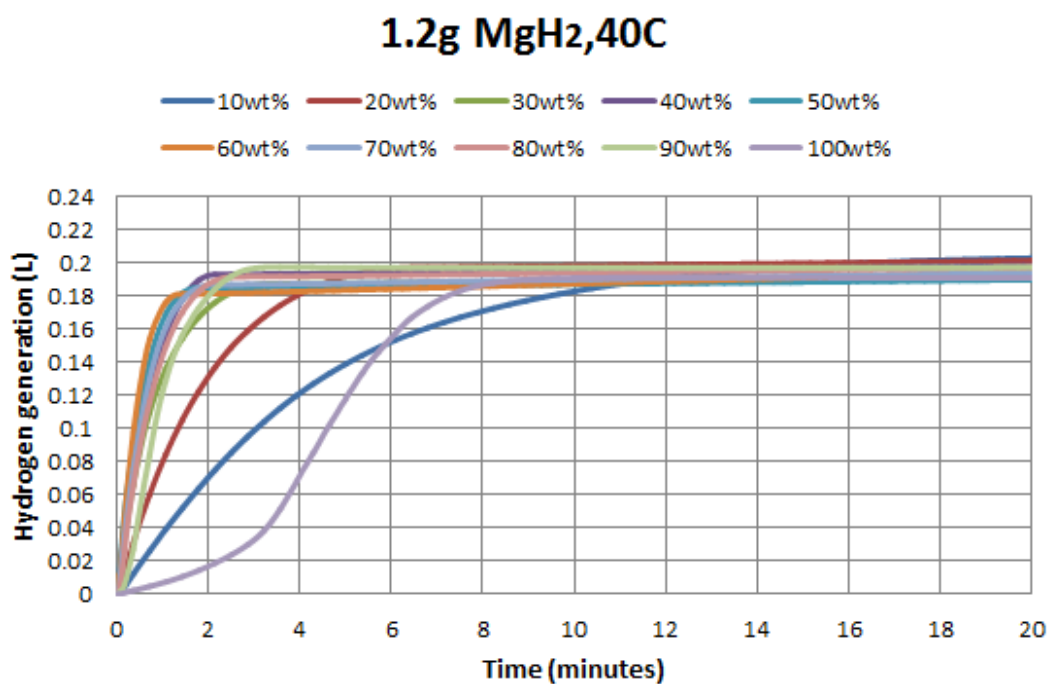


Fig. 27 (b) Different concentration of acetic acid with 1.2gMgH₂ at 40°C in 20 minutes

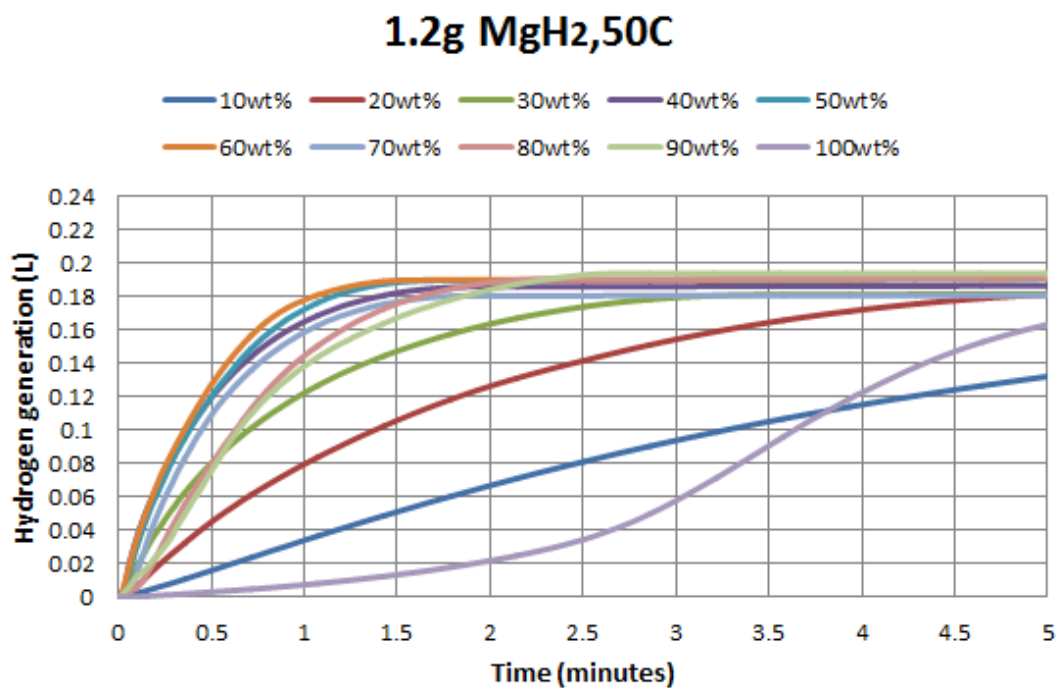


Fig. 28 (a) Different concentration of acetic acid with 1.2gMgH₂ at 50°C in 5 minutes

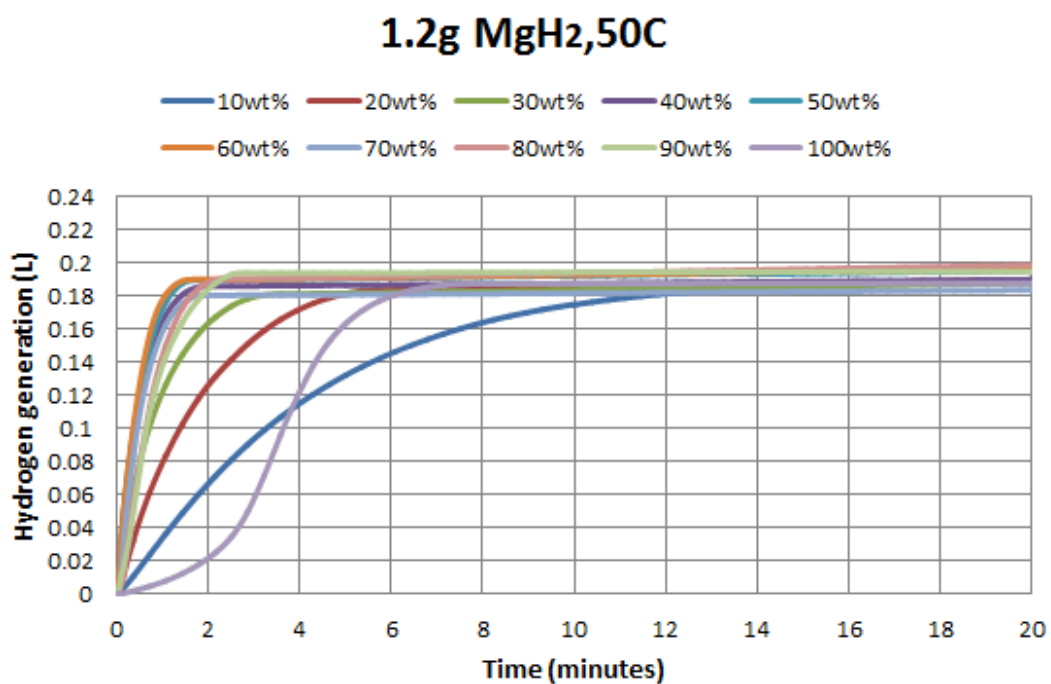


Fig. 28 (b) Different concentration of acetic acid with 1.2gMgH₂ at 50°C in 20 minutes

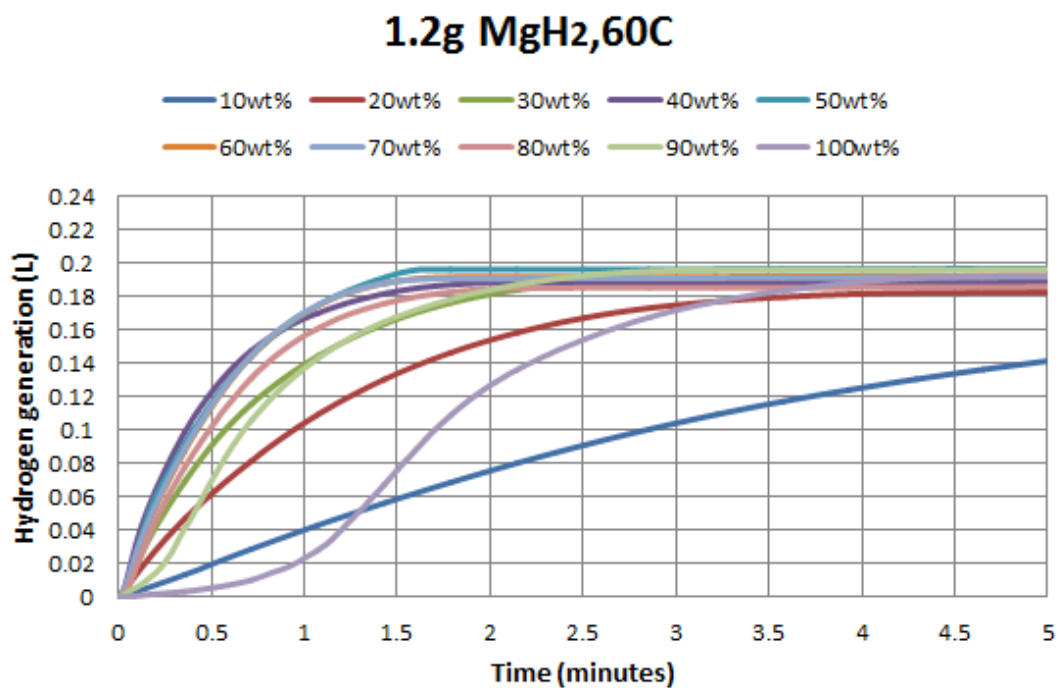


Fig. 29 (a) Different concentration of acetic acid with 1.2gMgH₂ at 60°C in 5 minutes

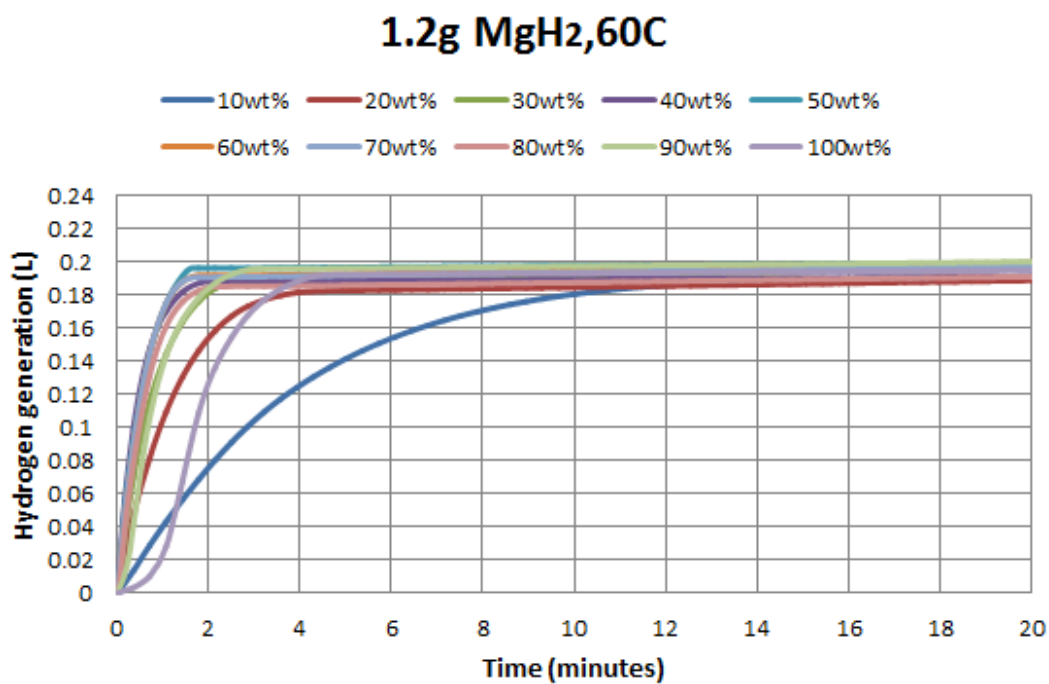


Fig. 29 (b) Different concentration of acetic acid with 1.2gMgH₂ at 60°C in 20 minutes

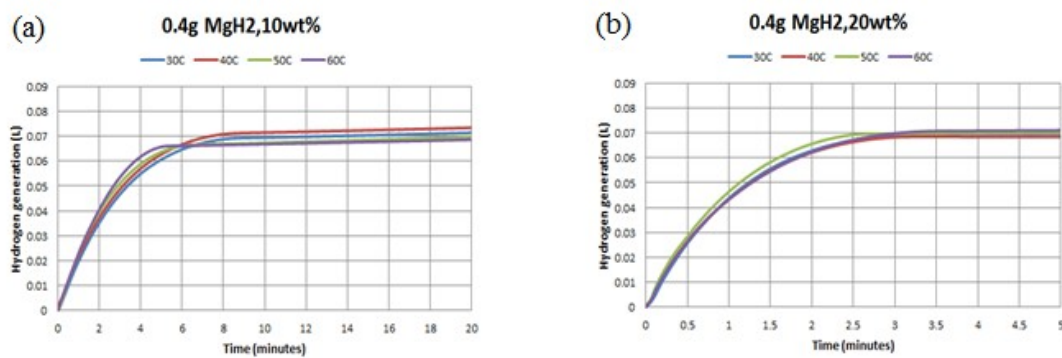


Fig. 30 Different temperature with 0.4g MgH₂ (a) 10 wt% in 20 minutes (b) 20 wt% in 5 minutes

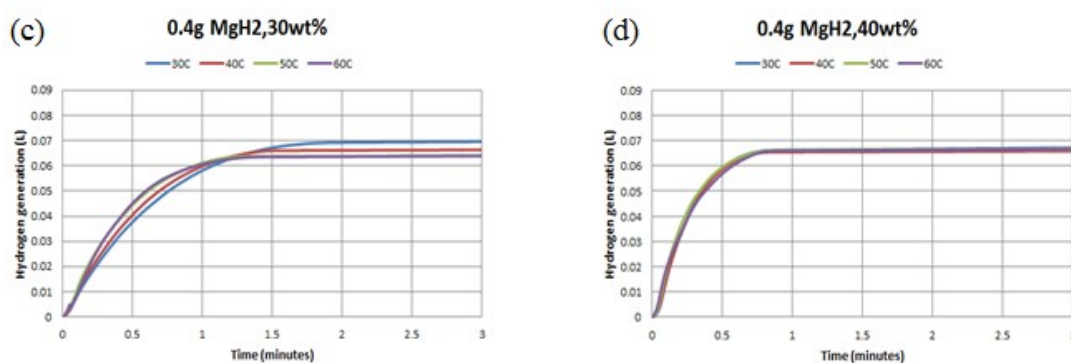


Fig. 30 Different temperature with 0.4g MgH₂ (c) 30 wt% in 3 minutes (d) 40 wt% in 3 minutes

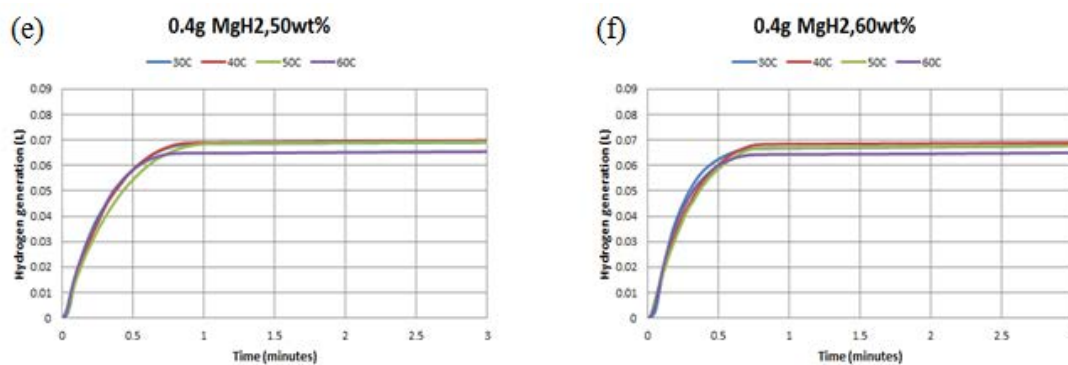


Fig. 30 Different temperature with 0.4g MgH₂ (e) 50 wt% in 3 minutes (f) 60 wt% in 3 minutes

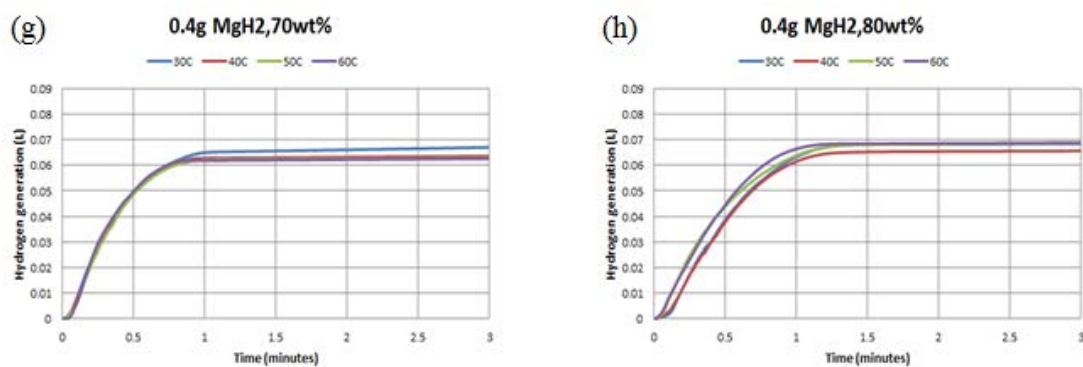


Fig. 30 Different temperature with 0.4g MgH₂ (g) 70 wt% in 3 minutes (h) 80 wt% in 3 minutes

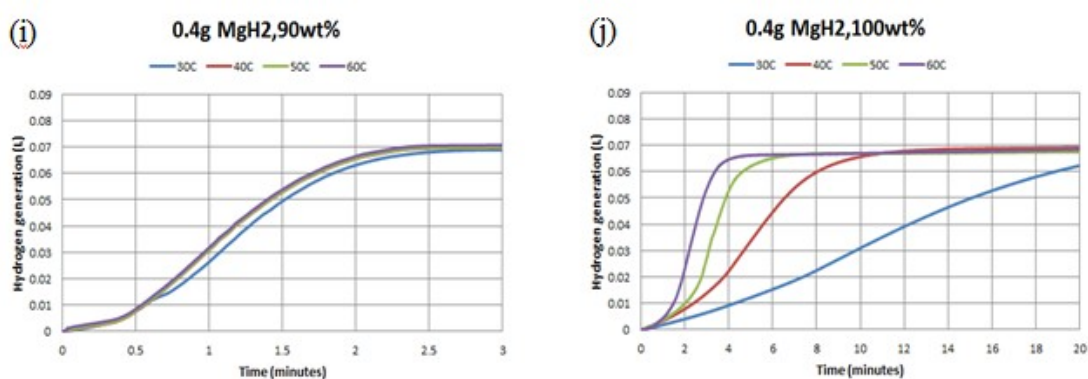


Fig. 30 Different temperature with 0.4g MgH₂ (i) 90 wt% in 3 minutes (j) 100 wt% in 20 minutes

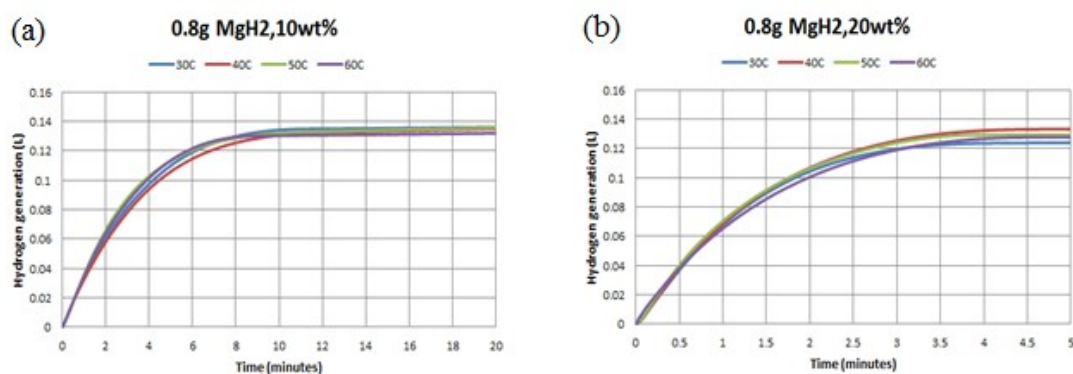


Fig. 31 Different temperature with 0.8g MgH₂ (a) 10 wt% in 20 minutes (b) 20 wt% in 5 minutes

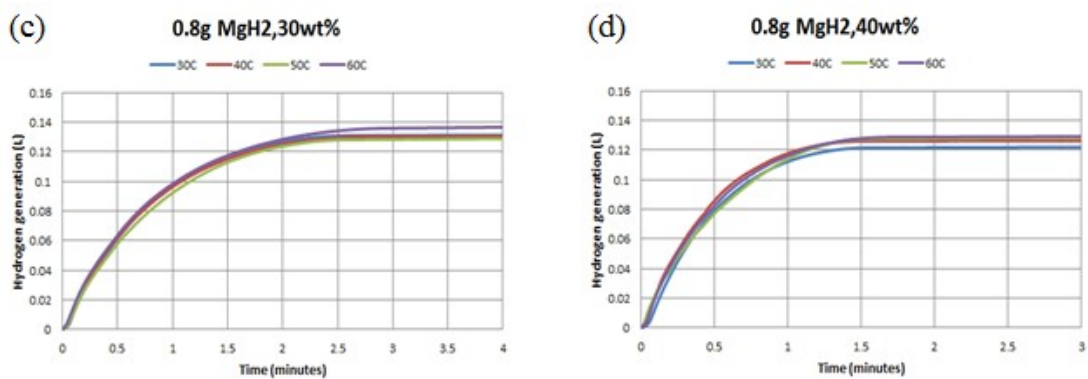


Fig. 31 Different temperature with 0.8g MgH₂ (c) 30 wt% in 3 minutes (d) 40 wt% in 3 minutes

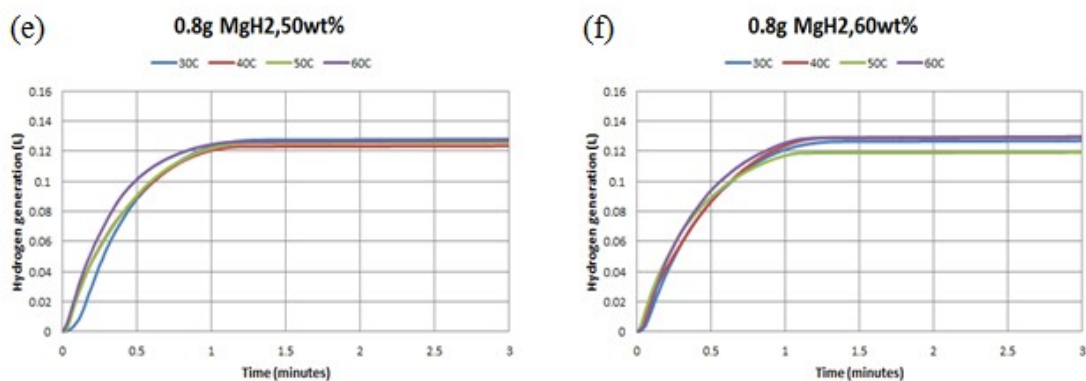


Fig. 31 Different temperature with 0.8g MgH₂ (e) 50 wt% in 3 minutes (f) 60 wt% in 3 minutes

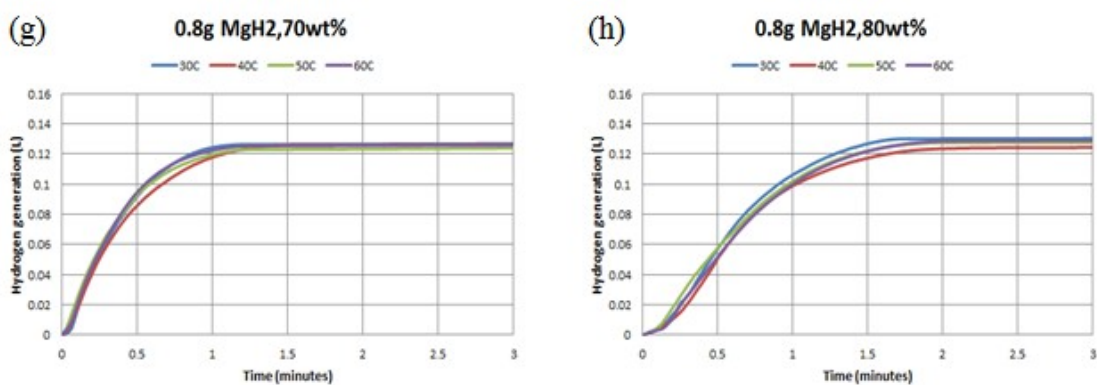


Fig. 31 Different temperature with 0.8g MgH₂ (g) 70 wt% in 3 minutes (h) 80 wt% in 3 minutes

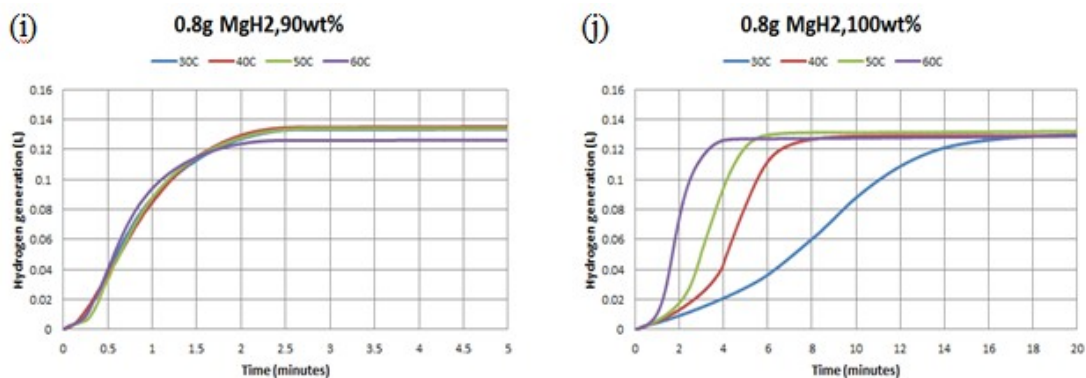


Fig. 31 Different temperature with 0.8g MgH₂ (i) 90 wt% in 3 minutes (j) 100 wt% in 20 minutes

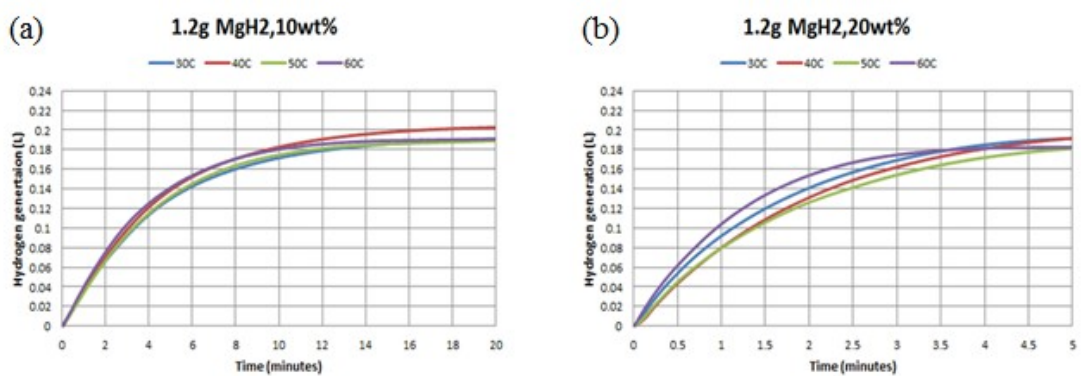


Fig. 32 Different temperature with 1.2g MgH₂ (a) 10 wt% in 20 minutes (b) 20 wt% in 5 minutes

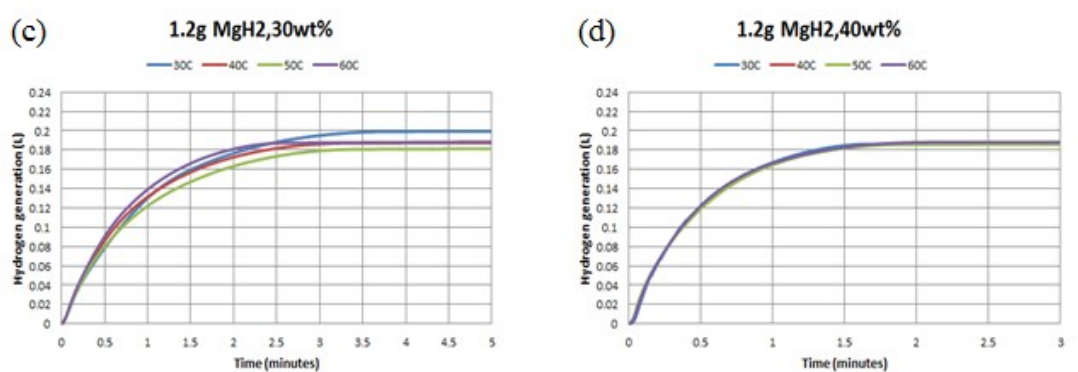


Fig. 32 Different temperature with 1.2g MgH₂ (c) 30 wt% in 3 minutes (d) 40 wt% in 3 minutes

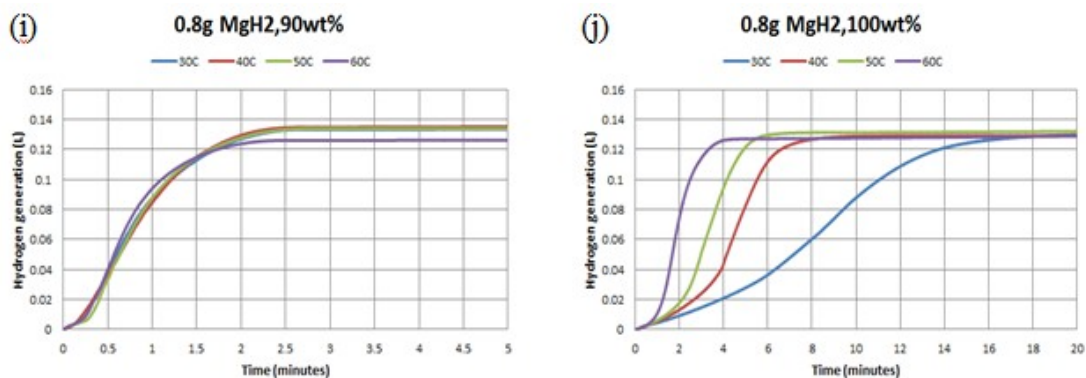


Fig. 32 Different temperature with 1.2g MgH₂ (e) 50 wt% in 3 minutes (f) 60 wt% in 3 minutes

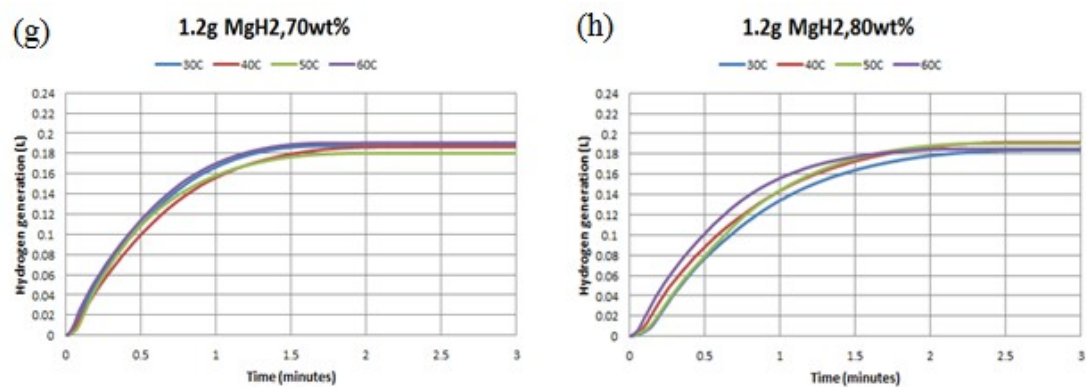


Fig. 32 Different temperature with 1.2g MgH₂ (g) 70 wt% in 3 minutes (h) 80 wt% in 3 minutes

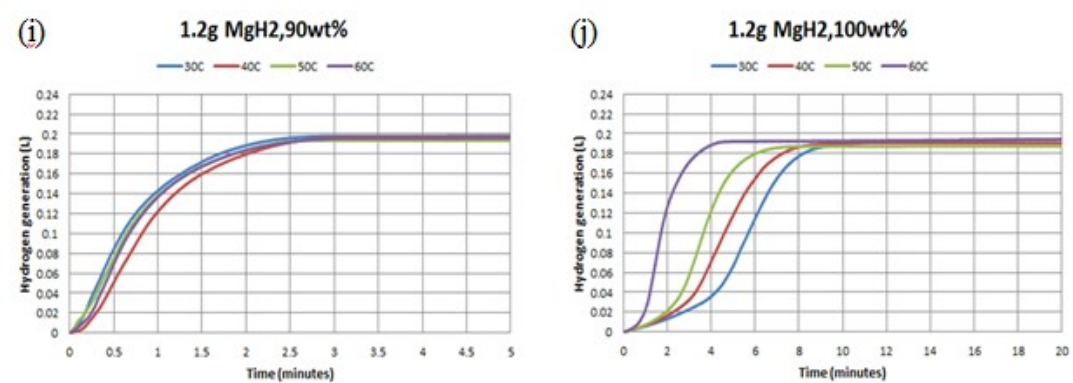


Fig. 32 Different temperature with 1.2g MgH₂ (i) 90 wt% in 3 minutes (j) 100 wt% in 20 minutes

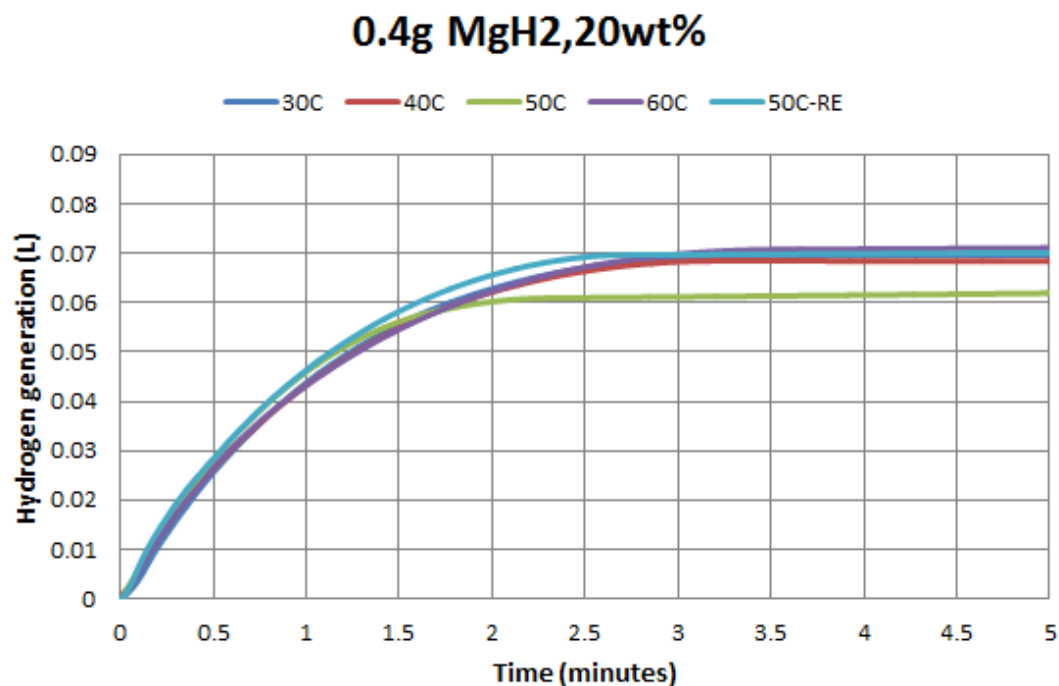


Fig. 33 (a) Repeating 0.4gMgH₂ and 20 wt% acetic acid with different temperature in 5 minutes

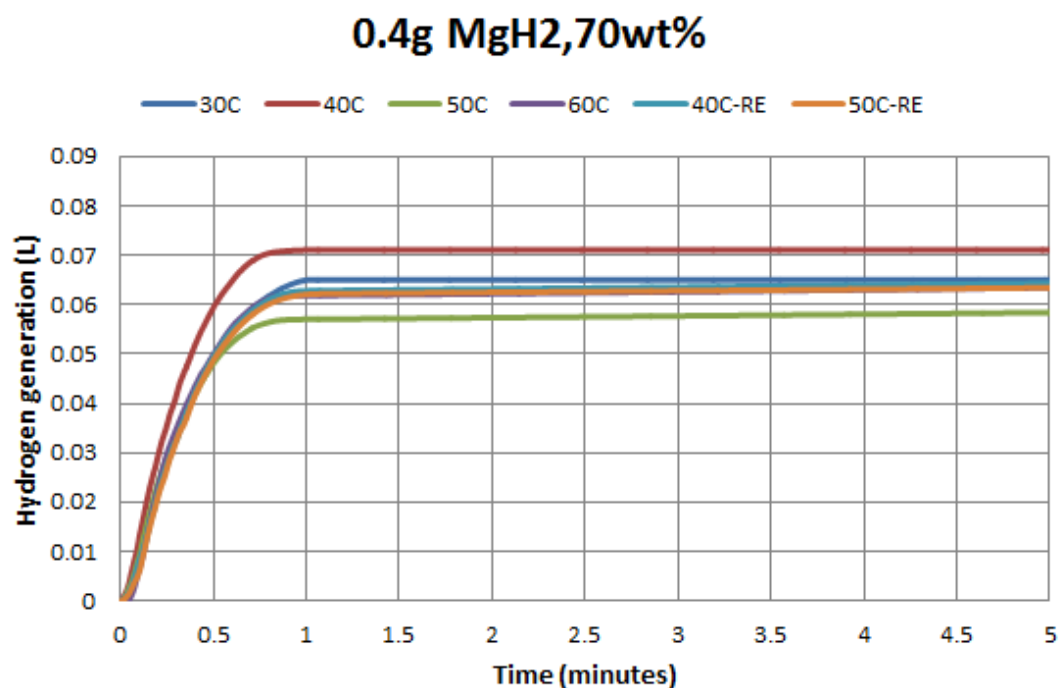


Fig. 33 (b) Repeating 0.4gMgH₂ and 70 wt% acetic acid with different temperature in 5 minutes

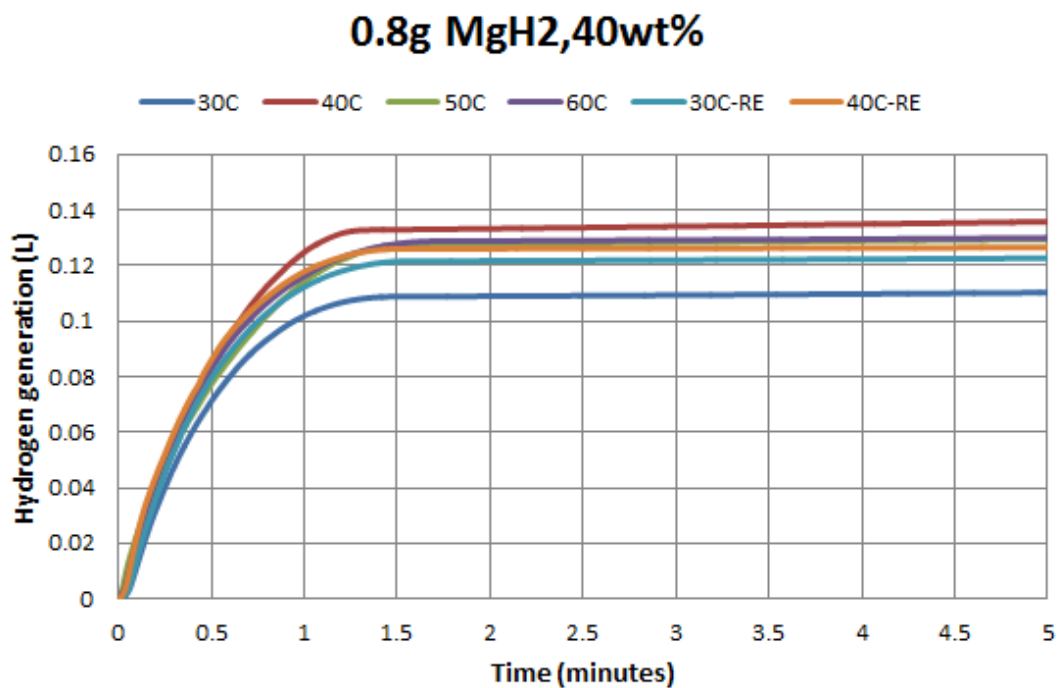


Fig. 34 (a) Repeating 0.8gMgH₂ and 40 wt% acetic acid with different temperature in 5 minutes

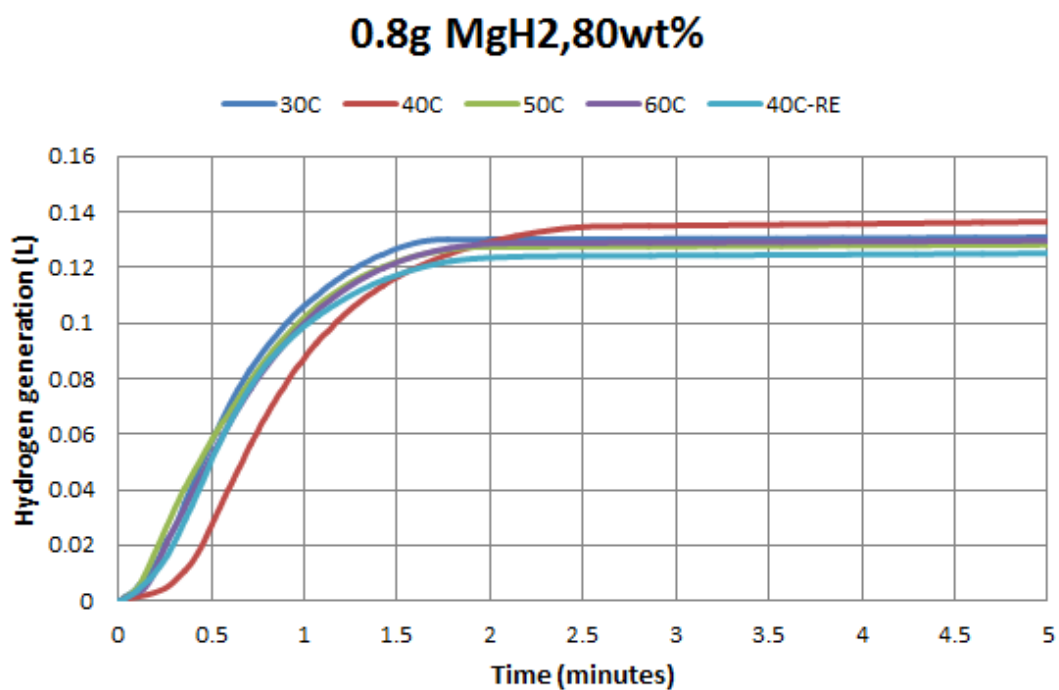


Fig. 34 (b) Repeating 0.8gMgH₂ and 80 wt% acetic acid with different temperature in 5 minutes

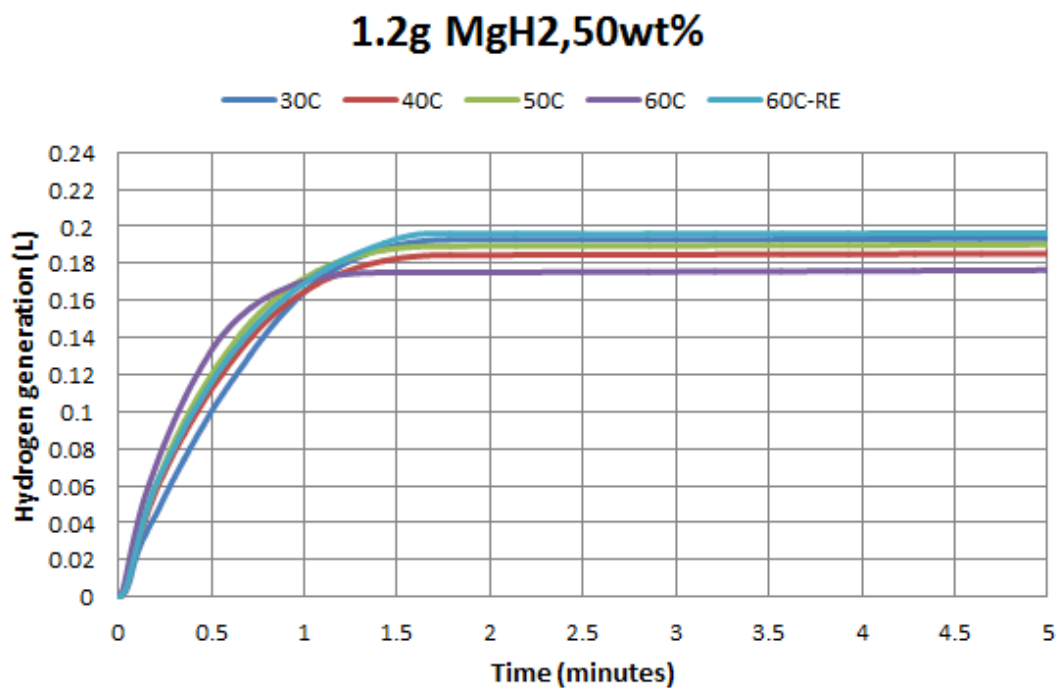


Fig. 35 (a) Repeating 1.2gMgH₂ and 50 wt% acetic acid with different temperature in 5 minutes

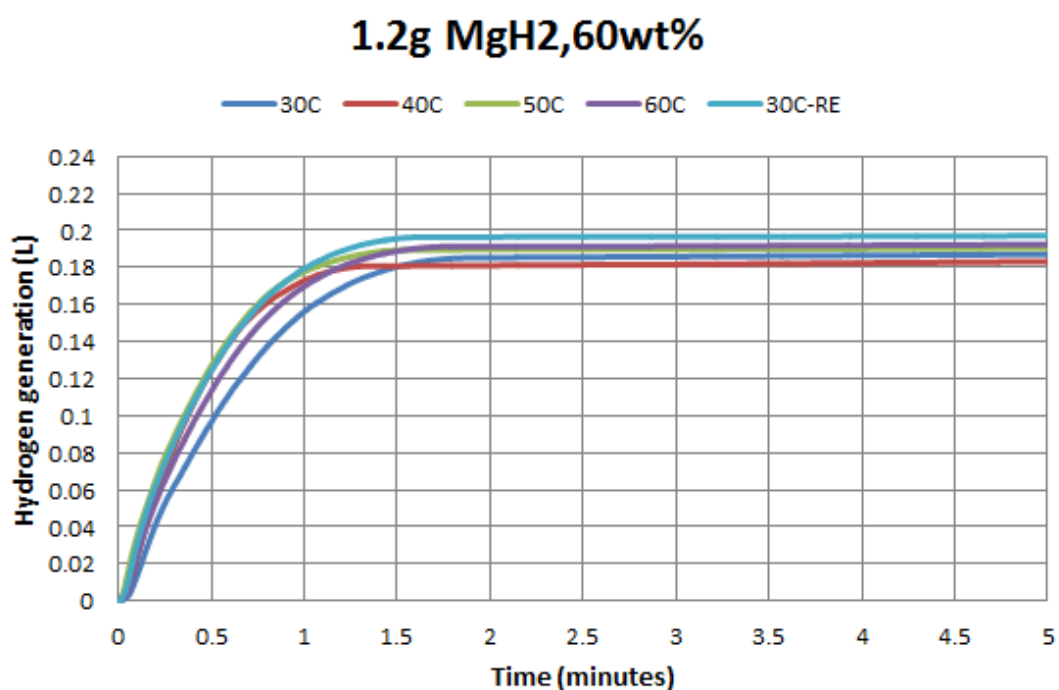


Fig. 35 (b) Repeating 1.2gMgH₂ and 60 wt% acetic acid with different temperature in 5 minutes

Table 1 Categories of Fuel Cell [18].

Fuel Cell Type	Proton Electrolyte Membrane Fuel Cell (PEMFC)	Alkaline Fuel Cell (AFC)	Molten Carbonate Fuel Cell (MCFC)	Solid Oxide Fuel Cell (SOFC)
Operating Temperature	60°C~200°C	60°C~200°C	600°C~1000°C	600°C~1000°C
Reaction Equation	Anode $\text{H}_2 \rightarrow 2\text{H}^+ + 2\text{e}^-$ $\frac{1}{2}\text{O}_2 + 2\text{H}^+ + 2\text{e}^- \rightarrow \text{H}_2\text{O}$	$\text{H}_2 + 2\text{OH}^- \rightarrow 2\text{H}_2\text{O} + 2\text{e}^-$ $\frac{1}{2}\text{O}_2 + \text{H}_2\text{O} + 2\text{e}^- \rightarrow 2\text{OH}^-$	$\text{H}_2 + \frac{1}{2}\text{O}_2 + \text{CO}_2 \rightarrow \text{H}_2\text{O} + \text{CO}$ $\frac{1}{2}\text{O}_2 + \text{CO}_2 + 2\text{e}^- \rightarrow \text{CO} + \text{O}^{2-}$	$\text{H}_2 + \text{O}^{2-} \rightarrow \text{H}_2\text{O} + 2\text{e}^-$ $\text{CO} + \text{O}^{2-} \rightarrow \text{CO}_2 + 2\text{e}^-$
Efficiency	43~58%	60%	45~50%	60%
Applications	<ul style="list-style-type: none"> . Transportation . Portal power . UPS system 	<ul style="list-style-type: none"> . Space . Military . Transportation 	<ul style="list-style-type: none"> . Composite power station 	<ul style="list-style-type: none"> . Composite power station
Advantages	<ul style="list-style-type: none"> . Quick start-up . Low pollution . Low noise 	<ul style="list-style-type: none"> . Low pollution . High efficiency . Low cost 	<ul style="list-style-type: none"> . High efficiency . Low noise . Flexibility with using different catalyst 	<ul style="list-style-type: none"> . High efficiency . Flexibility with using different catalyst
Disadvantages	<ul style="list-style-type: none"> . High cost in catalyst . Pure fuel requirement 	<ul style="list-style-type: none"> . High cost in manufacture 	<ul style="list-style-type: none"> . Long start-up . Corrosive electrolyte 	<ul style="list-style-type: none"> . Long start-up . Corrosive

Table 2 DOE Targets for Onboard Hydrogen Storage Systems for Light-Duty Vehicles [19]

Storage Parameter	Units	2010	2017	Ultimate
System Gravimetric Capacity:	kWh/kg	1.5	1.8	2.5
Usable, specific-energy from H₂ (net useful energy/max system mass)	(kg H ₂ /kg system)	(0.045)	(0.055)	(0.075)
System Volumetric Capacity: Usable	kWh/L	0.9	1.3	2.3
energy density from H₂ (net useful energy/max system volume)	(kg H ₂ /L system)	(0.028)	(0.040)	(0.070)
Storage System Cost	\$/kWh net	TBD	TBD	TBD
(included fuel cost)	(\$/kg H ₂)	(TBD)	(TBD)	(TBD)
	\$/gge at pump	3-7	2-4	2-4
Durability/Operability				
● Operating ambient temperature	°C	-30/50 (sun)	-40/60 (sun)	-40/60 (sun)
● Min/max delivery temperature	°C	-40/85	-40/85	-40/85
● Operational cycle life (1/4 tank to full)	Cycles	1000	1500	1500
● Min delivery pressure from storage system; FC=fuel cell, ICE=internal combustion engine	bar (abs)	5 FC/35 ICE	5 FC/35 ICE	5 FC/35 ICE
● Max delivery pressure from storage system	bar (abs)	12 FC/100 ICE	12 FC/100 ICE	12 FC/100 ICE
● Onboard Efficiency	%	90	90	90
● "Well" to Powerplant Efficiency	%	60	60	60
Charging/ Discharging Rates:				
● System fill time (5 kg)	Min	4.2 (1.2)	3.3 (1.5)	2.5 (2.0)
● Minimum full flow rate	(kg H ₂ /min)	0.02	0.02	0.02
● Start time to full flow (20°C) [§]	(g/s)/kW	5	5	5
● Start time to full flow (-20°C) [§]	S	15	15	15
● Transient response 10%-90% and 90%-0% ^h	S	0.75	0.75	0.75
Fuel Purity (H ₂ from storage)	% H ₂	SAE J2719 and ISO/PDTS 1487-2 (99.97% dry basis)		
Environmental health & Safety:				
● Permeation & leakage	Scch/h	Meets or exceeds applicable standards		
● Toxicity	-			
● Safety	-			
● Loss of useable H ₂	(g/h)kg H ₂ stored	0.1	0.05	0.05

Table 3 US DOE Hydrogen Storage Technical Targets [1].

Storage Parameter	Units	Year	
		2007	2010
Gravimetric Capacity (net useful energy/max system mass)	kWh/kg	1.5	2
	(kg H ₂ /L system)	(0.045)	(0.06)
Volumetric Capacity	kWh/L	1.2	1.5
	(kg H ₂ /L system)	(0.036)	(0.045)
Storage System Cost (and hydrogen cost)	\$/kWh net	6	4
	(\$/kg H ₂)	(200)	(133)
	\$/gge at pump	No Available Target	2-3

Table 4 The specification of the thermostatic water bath [23].

Model	B101 (10 Liter)
Temperature Range	Ambient(°C)+5~100°C
Operation Range	-20~100°C
Temperature Stability	±0.3°C
Display	Digital Number Display
Power Output	600W
Inside Dimensions (cm)	W29.5 x D23 x H15
Outside Dimensions (cm)	W34.5 x D28 x H27

Table 5 The specification of the flow meter [24].

Model	FCST 1030
Flow Range	10 SLM
Control Range	2~100% Full Scale
Flow Accuracy	±1% Full Scale
Ambient Temperature	5~50°C (Flow Accuracy Guaranteed 15~35°C)
Response Time	≤ 1 Sec
Linearity	±0.5% Full Scale
Repeatability	±0.2% Full Scale
Differential Pressure Range	100~300 kPa
Maximum Operating Pressure	40 kPa
Proof Pressure	1 MPa
External Leak Integrity	1×10^{-7} Pa.m ² /sec (He)
Wetted Material	SUS316, Magnetic Stainless, Chloroprene Rubber
Power Consumption	+15V:50mA, -15V:200mA (24V:DeviceNet)

Table 6 The specification of the portable pH meter [25]

Model	MW101 pH Meter
Range PH/ORP Temp	0.00 to 14.00 pH
Resolution PH/ORP Temp	0.01 pH
Accuracy	±0.02 pH
Temperature Compensation	Manual, 0 to 50°C
Calibration	Manual, 2-point through offset and slop trimmers
pH Electrode	MA911B/1 (included)
Environment	0 to 50°C, max RH 95%
Battery Type	1 x 9V alkaline (included)
Battery Life	Approximant 300 hours of use
Dimensions	145 x 80 x 40 mm
Weight	220 g (with battery)

Table 7 The specification of the precision balance [26].

Model	AV53
Capacity	51g
Readability	1 mg
Linearity	±0.002 g
Stabilization Time	2.5 sec
Power Requirement	AC Adapter (included)
Display Type	2-Line LCD with Backlight
Display Size	10 x 2.5 cm
Platform Size	10 cm
Net Weight	1.4 kg

Table 8 Hydrogen generation experimental condition

Environmental temperature	Weight of MgH₂	Concentration of acetic acid (total solution amount is 100g)
30°C	0.4g	10 wt%, 20 wt%, 30 wt%, 40 wt%, 50 wt%, 60 wt%, 70 wt%, 80 wt%, 90 wt%, 100 wt%
	0.8g	
	1.2g	
40°C	0.4g	10 wt%, 20 wt%, 30 wt%, 40 wt%, 50 wt%, 60 wt%, 70 wt%, 80 wt%, 90 wt%, 100 wt%
	0.8g	
	1.2g	
50°C	0.4g	10 wt%, 20 wt%, 30 wt%, 40 wt%, 50 wt%, 60 wt%, 70 wt%, 80 wt%, 90 wt%, 100 wt%
	0.8g	
	1.2g	
60°C	0.4g	10 wt%, 20 wt%, 30 wt%, 40 wt%, 50 wt%, 60 wt%, 70 wt%, 80 wt%, 90 wt%, 100 wt%
	0.8g	
	1.2 g	

Table 9 pH value for 0.4g MgH₂ with different temperature

	0.4g MgH₂,30C	0.4g MgH₂,40C	0.4g MgH₂,50C
Concentration of acetic acid	pH	pH	pH
10wt%	3.92	3.92	3.98
20wt%	3.70	3.69	3.63
30wt%	3.46	3.47	3.34
40wt%	3.38	3.32	3.13
50wt%	3.37	3.11	2.95
60wt%	3.13	2.94	2.81
70wt%	2.96	2.88	2.64
80wt%	2.86	2.73	2.37
90wt%	2.68	2.41	2.01
100wt%	1.93	1.69	1.60

Table 10 pH value for 0.8g MgH₂ with different temperature

	0.8g MgH₂,30C	0.8g MgH₂,40C	0.8g MgH₂,50C
Concentration of acetic acid	pH	pH	pH
10wt%	4.30	4.25	4.36
20wt%	3.89	3.86	3.97
30wt%	3.72	3.66	3.65
40wt%	3.51	3.51	3.61
50wt%	3.42	3.45	3.53
60wt%	3.40	3.37	3.33
70wt%	3.35	3.15	3.23
80wt%	3.10	3.11	3.05
90wt%	2.92	2.99	2.80
100wt%	2.31	2.33	2.24

Table 11 pH value for 1.2g MgH₂ with different temperature

	1.2g MgH ₂ ,30C	1.2g MgH ₂ ,40C	1.2g MgH ₂ ,50C
Concentration of acetic acid	pH	pH	pH
10wt%	4.48	4.48	4.51
20wt%	4.13	4.03	4.00
30wt%	3.83	3.74	3.79
40wt%	3.60	3.57	3.67
50wt%	3.53	3.41	3.53
60wt%	3.42	3.24	3.33
70wt%	3.36	3.09	3.21
80wt%	3.21	2.93	3.00
90wt%	3.15	2.72	2.68
100wt%	2.66	2.58	2.27