



Catalytic effect of PdCl₂ on the performance of Mg-based hydride hydrolysis

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Received 10 02 2023; accepted 03 08 2024

Available 08 31 2024

Abstract: Magnesium hydride (MgH₂) is a promising hydrogen storage material and has a theoretical hydrogen generation of up to 1703 mL/g when it combines with water. A study was done to understand the effects of PdCl₂ on the kinetics of Mg-based hydride hydrolysis. The preparation of the sample was done by mechanical ball milling technique in 1 hour. The addition of 10 wt% PdCl₂ was found to significantly enhance the hydrogen production via the MgH₂ hydrolysis at room temperature. At room temperature, the MgH₂-10 wt% PdCl₂ system showed the fastest hydrolysis rate where it produced 1153 mL/g of hydrogen after 250 min, which is 131% higher than the 1-hour milled MgH₂. In addition, Mg(OH)₂, MgH₂ and Pd are the byproducts found from the hydrolysis process of the mixture. The hydrolysis kinetic outcomes showed a novel approach to enhance the hydrolysis of MgH₂, suggesting that PdCl₂ is a potential reagent for boosting the hydrolysis of MgH₂ for green hydrogen generation systems.

Keywords: Magnesium hydride, hydrolysis, hydrogen production, palladium (II) chloride, catalyst

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Peer Review under the responsibility of Universidad Nacional Autónoma de México.

1. Introduction

As for July 2023, the world population stands at 8 billion (United Nations, 2023). As populations rise with the expectation of decent living conditions, global energy consumption will continue to increase which is attributable to developed nations. The U.S. Energy Information Administration (EIA) predicts that the world's primary energy usage will increase from 601.5 to 886.3 quadrillion BTU from 2020 to 2050 (U.S Energy Information Administration, 2021). Additionally, the EIA expects the global usage of petroleum and other liquid fuels to increase by 33.8 quadrillion BTU during the same period. As a result, there is a global focus on creating dependable power systems that utilize renewable energy, such as solar PV systems, wind power systems, and hydropower systems. According to the Renewable Energy Policy Network for the 21st Century (REN21), around 28% of the energy used for electricity production in 2019 was derived from renewable sources (REN21, 2022).

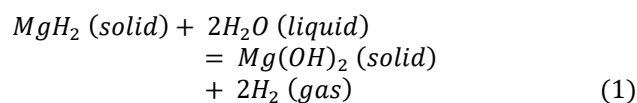
Hydrogen, possessing an energy level between 120 to 142 MJ/kg, has been spotlighted as a secondary energy source with energy content that triples that of petroleum (Shao et al., 2018). This positions it as a potential key player in securing a future supply of eco-friendly energy. However, the journey to making hydrogen a significant global energy source is fraught with challenges, starting from its production process. Despite being the most abundant and inexhaustible element globally, hydrogen is typically found in combination with other elements, forming compounds such as water, hydrocarbon fuels, and various organic materials. This necessitates the need for hydrogen to be extracted or produced before utilization.

Regrettably, the production of hydrogen is still dependent on fossil fuels like coal, natural gas, oil, and naphtha. This mode of production is in contradiction with the Sustainable Development Goals (SDG) and Malaysia national policies like The Renewable Energy Transition Roadmap (RETR) 2035 by SEDA, which are aimed at providing clean and sustainable energy to all as a measure against climate change and its impacts (Sustainable Energy Development Authority (SEDA) Malaysia, 2023).

Given these challenges, the hydrolysis of metal hydrides emerges as a green and sustainable alternative for hydrogen production. Among these hydrides, magnesium hydride (MgH_2) stands out due to its impressive hydrogen capacity (Baykara, 2018). For example, when reacted with water at room temperature, MgH_2 can release up to 1703 mL/g of hydrogen (Nikolaidis & Poullikkas, 2017). Notably, MgH_2 is also a strong contender for solid-state hydrogen storage technology, with pure MgH_2 theoretically able to store up to 7.6 wt% of hydrogen. Numerous studies underscore the potential of MgH_2 in solid-state hydrogen storage, as

evidenced by others works such as from Jalil et al. (2018), (2023), Ismail et al. (2020) and Malahayati et al. (2018).

Furthermore, MgH_2 presents significant advantages for hydrogen generation through hydrolysis. It is non-toxic, cost-effective regarding raw materials (Yahya & Ismail, 2018a) and boasts a high hydrogen storage capacity of up to 15.3 wt.% (Li et al., 2017). In addition, it is thermodynamically stable (Yahya & Ismail, 2018b) in the absence of moisture which means that it has a long shelf life. It is environmentally friendly, and the hydrolysis product can be used and recycled. The reaction process of the MgH_2 hydrolysis can be given as follows:



These advantages are offset by low reaction kinetics due to the interruption of magnesium hydroxide ($Mg(OH)_2$) passivation layers that will form during the hydrolysis process (Zhou et al., 2021). The $Mg(OH)_2$ layer will prevent contact between water and MgH_2 inner particles (Figure 1) which is the key process for hydrolysis (Ruslan et al., 2022). As a result, the rapid hydrolysis reaction at the early stage of the process may slow down or, in the worst-case scenario, come to a complete halt.

Previous attempts had used Bronsted acids like citric or acetic acids to dissolve or destabilize the passivation layers (Uesugi et al., 2011). However, the added acids were detrimental to the equipment and hazardous for the users. As an alternative, by adding specific catalysts such sodium chloride, potassium chloride, or magnesium chloride to the solution or straight to magnesium hydride, the reaction speed can be accelerated (Gan et al., 2018; Ouyang et al., 2015).

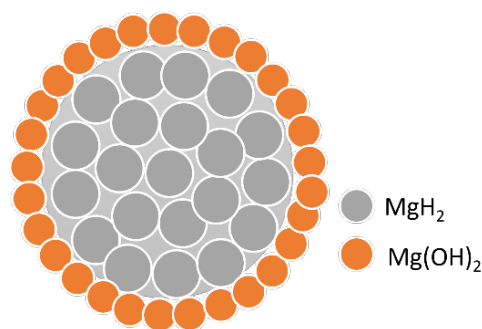


Figure 1. Schematic diagram of passivation layer of $Mg(OH)_2$ on MgH_2 .

In this paper, $PdCl_2$ was studied as a highly potential catalyst to improve the MgH_2 hydrolytic properties. This catalyst was chosen based on its excellent catalytic performance in solid-state hydrogen storage composite materials reported by other researchers (Chawla et al., 2022;

Mustafa et al., 2016). The introduction of this catalyst as well as the ball milling process is hypothesized to significantly improve the hydrolytic process of the MgH_2 system. The ball milling process will reduce the particle size which in return the effective surface area for the reaction to happen will significantly increase. Meanwhile, the catalyst's role is to break the passivation layer so that the hydrolysis process can be accelerated.

2. Materials and methods

MgH_2 with 99% purity was procured from Langfang BLD Trade Co, while PdCl_2 was sourced from Sigma-Aldrich. All powder managing procedures, including weighing and loading, were conducted in an MBraun Unilab glove box under a high-purity Argon gas atmosphere to prevent exposure to moisture and air. The ball-milling technique was employed to prepare pure MgH_2 and $\text{MgH}_2 + \text{PdCl}_2$ mixtures, using a planetary ball mill (NQM-0.4) operating at 400 rpm for an hour, with a weight ratio of 89:1 between the ball and the composite powder.

The hydrolysis process took place in a 250 mL flask with three openings (Figure 2), (i) for distilled water injection, (ii) for a temperature sensor, and (iii) for hydrogen exit, which was connected to a gas counter (Rigamo Milligas Counter) with $\pm 3\%$ uncertainty that linked to a PC. The flask's temperature was maintained using a water bath, and the experiment was conducted at room temperature. In the flask, 0.1 g of the mixture under investigation was combined with 10 mL of distilled water.

After the hydrolysis, the pH of the solution was measured using the CyberScan pH310 pH meter before the drying process. The solution was then filtered, and the solid byproduct was dried at 100°C . X-ray diffraction (using a Rigaku MiniFlex II diffractometer with $\text{Cu K}\alpha$ radiation) was used to study the phase compositions of the dried samples. Each measurement was taken at a scan speed of $2^\circ/\text{min}$ over diffraction angles ranging from 20° to 80° . The microstructure was observed using a scanning electron microscope (TESCAN Vega SEM) and energy dispersive spectroscopy (EDS).

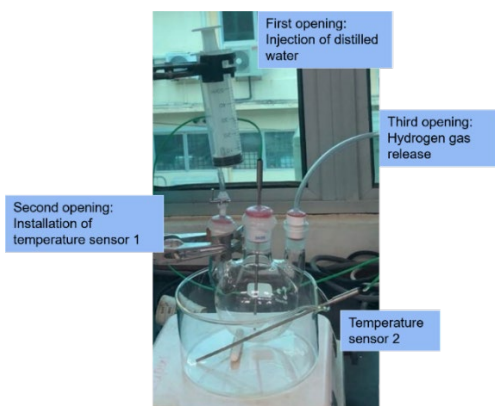


Figure 2. Hydrolysis setup.

3. Results and discussion

Figure 3 presents the hydrolysis kinetics of MgH_2 containing 10 wt.% of PdCl_2 at 30°C . The figure also includes a comparison of the kinetic hydrolysis between the pure MgH_2 and the 1-hour milled MgH_2 . Notably, the characterization indicates that pure MgH_2 exhibits the slowest hydrolysis kinetics. The hydrolysis reaction only occurred for only 3.2 minutes, resulting in a total hydrogen yield of 101 mL/g. As $\text{Mg}(\text{OH})_2$, one of the byproducts of the MgH_2 hydrolysis has low solubility in water (1.22 mg/100 mL at 20°C) (Ropp, 2013), it remains solid and prevents further contact between the water and the hydride. Similar observations on the slow hydrolysis process of pure MgH_2 have been reported by other researchers (Korablov et al., 2021; Tessier et al., 2004).

Conversely, the 1-hour milled MgH_2 showed approximately five times higher hydrogen yield compared to pure MgH_2 , reaching 499 mL/g of hydrogen. However, the hydrolysis reaction for this 1-hour milled MgH_2 halted after 81.8 minutes. In the case of MgH_2 containing 10 wt.% of PdCl_2 , it produced an impressive 985 mL/g and 1153 mL/g of hydrogen after 60 min and 250 min, respectively. This hydrogen yield total amount is about 11 times greater than pure MgH_2 and about 131% higher than the 1-hour milled MgH_2 . These results underscore the beneficial impact of the ball milling process on MgH_2 hydrolysis. Furthermore, they corroborate the catalytic influence of PdCl_2 on the hydride hydrolysis of MgH_2 .

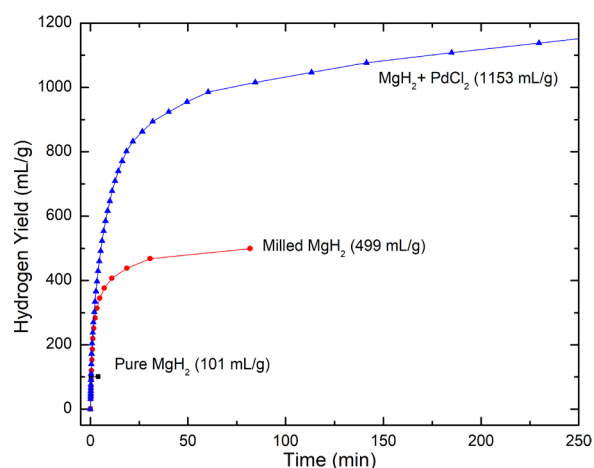


Figure 3. Hydrolysis reaction of the pure, 1-hour milled MgH_2 and MgH_2 with 10 wt.% of PdCl_2 at 30°C .

To better understand the influence of 10 wt.% PdCl_2 on the hydrolysis of the MgH_2 system, experiments were conducted at temperatures of 40°C and 50°C . The results, illustrated in Figure 4, revealed that the hydrolysis at 40°C produced hydrogen for about 1458 mL/g of MgH_2 in 250 min. This represents an improvement of approximately 26.46% compared to the hydrogen yield of 1153 mL/g at 30°C .

Similarly, at 50°C, the hydrogen yield increased to 1745 mL/g, marking a notable 51.34% enhancement relative to the 30°C yield.

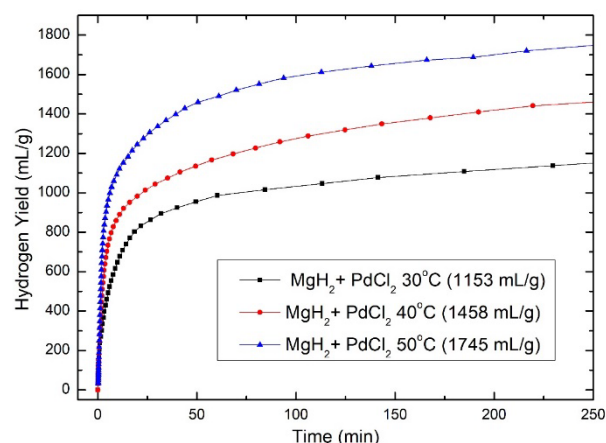


Figure 4. Hydrolysis reaction of MgH_2 with 10 wt% of PdCl_2 at 30, 40 and 50°C.

The impact of ball milling on hydrolysis kinetics was investigated through SEM characterizations performed on the selected samples. SEM image of the pure MgH_2 was captured at a 500x magnification and are displayed in Figure 5 (a). The image revealed that the pure MgH_2 particles are the largest, boasting round and clean surfaces. However, following an hour of milling process, there is a significant decrease in particle size, and numerous surface defects appear, making the particles rough as shown in Figure 5 (b) with 5000x magnification. However, particles agglomeration can be observed for the 1-hour milled MgH_2 . Meanwhile, for Figure 5 (c), where MgH_2 is mixed with 10 wt.% of PdCl_2 , the particles shrink even further, their surfaces becoming rough and marred with defects and less agglomerated. This evidence from the SEM images confirms how ball milling affects the size of MgH_2 particles. It also provides an explanation as to why the catalyst-infused, milled mixture highlights superior hydrolysis kinetics when compared to pure MgH_2 .

The XRD characterizations were conducted to study the byproducts of the hydrolysis process. The hydrolysis residues of pure MgH_2 , 1-hour milled MgH_2 , and MgH_2 containing 10 wt.% of PdCl_2 are contrasted in Figure 6 (a) to (c), respectively. From Figure 6(a), it is evident that the hydrolysis of pure MgH_2 leaves behind observable peaks of MgH_2 and just one peak for $\text{Mg}(\text{OH})_2$. This outcome aligns with the hydrolysis kinetics of pure MgH_2 , from which only a minimal quantity of hydrogen (101 mL/g) was released. Consequently, multiple MgH_2 peaks remain noticeable after the hydrolysis process showing that most of the MgH_2 remained, due to the passivation layer of $\text{Mg}(\text{OH})_2$ which prevents the diffusion of the water to the inner parts of the particles (Ouyang et al., 2015; Verbovitsky et al., 2020).

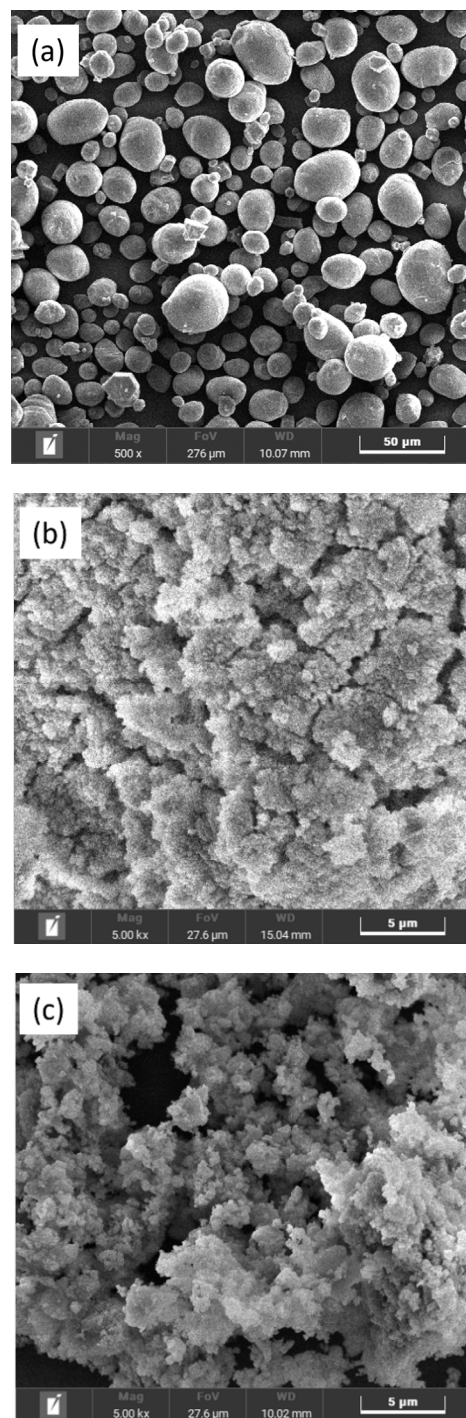


Figure 5. SEM images of the (a) pure MgH_2 , (b) milled MgH_2 and (c) MgH_2 with 10 wt.% of PdCl_2 prior the hydrolysis.

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In terms of the milled MgH_2 , several MgH_2 peaks are still visible after the hydrolysis, albeit less intense compared to those of pure MgH_2 . In addition, a greater number of Mg(OH)_2 peaks become apparent. This is in accordance with the hydrolysis kinetics, indicating that the milled MgH_2 could release a larger volume of hydrogen during the hydrolysis as compared to pure MgH_2 due to larger surface area facilitating the hydrolysis process (Verbovitsky et al., 2020).

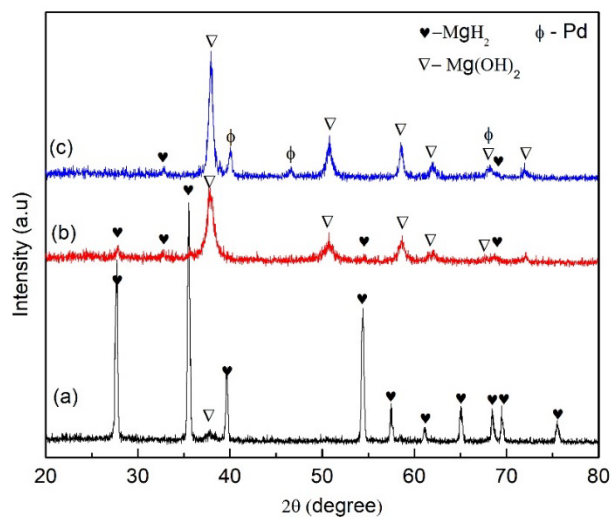


Figure 6. XRD patterns of the (a) pure MgH_2 , (b) milled MgH_2 and (c) MgH_2 with 10 wt% of PdCl_2 after the hydrolysis.

In the catalyzed mixture, peaks corresponding to MgH_2 and Mg(OH)_2 are evident. Additionally, byproduct peaks, attributed to the presence of PdCl_2 before the hydrolysis process are discernible. The emergence of peaks associated with Pd suggests that PdCl_2 reacted with H_2O to produce Pd^{2+} ions and Cl^- anion. In this context, the hydrogen gas yielded from the hydrolysis reaction can function as the reducing agent, potentially reducing Pd^{2+} ion to elemental Pd (Koo et al., 2006; Ranjbar et al., 2011). Meanwhile anions from strong Brønsted acids like Cl^- influence the reaction mixture's pH positively, aligning with the observed pH measurements. Specifically, the pH value of the hydrolyzed solution from the milled MgH_2 stood at 12.27. In contrast, for the hydrolyzed solution of MgH_2 containing 10 wt.% of PdCl_2 , the pH value dropped to 10.23. This indicates that a lower pH significantly impacts the hydrolysis of MgH_2 . However, as shown in Figure 6 (c), MgH_2

peaks remain evident, signifying that the hydrolysis process has not been fully completed. The reaction yielded about 1153 mL/g of hydrogen, which is less than the theoretical hydrogen content in MgH_2 .

To support the XRD findings, EDS analysis was conducted on the samples after they were subjected to hydrolysis and subsequently dried. The EDS images (Figure 7) verified the presence of three elements: Mg, Pd, and O. In addition, the elemental mapping shows that the distribution of Mg, Pd and O is uniform. It is worth noting that the H element, due to its low atomic number, cannot be detected by EDS. This is because it lacks sufficient electrons to generate a characteristic X-ray as mentioned by Wang and Chu (2013).

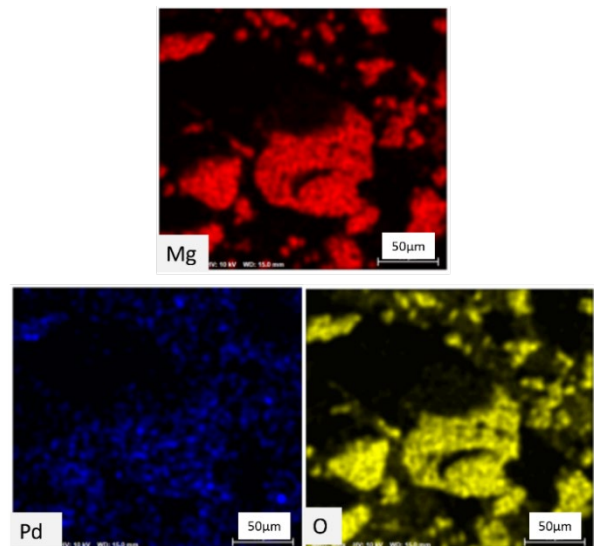


Figure 7. EDS images for the MgH_2 with 10 wt% of PdCl_2 compound after the hydrolysis.

Table 1 summarizes the hydrolysis results of various mixtures reported by previous researchers. The hydrogen yields presented in the table pertain to a 60-minute hydrolysis process, except for the MgH_2 - 5 wt% AlCl_3 mixture, which completed its hydrolysis in just 10 minutes. Our current study utilizing the PdCl_2 catalyst has displayed promising results. This mixture could produce significant amounts of hydrogen at both low (30°C) and elevated temperatures (50°C), standing out when compared to other mixtures. However, a direct comparison of hydrogen yields across these mixtures is challenging. Variations in factors such as ball milling duration during sample preparation, the acidity of the catalyst solution, and the hydrolysis methods used by different researchers make exact comparisons difficult. There has not been a standardized method or technique established for this process. Despite these challenges, these results can serve as a benchmark for evaluating the performance of the catalyst in our study relative to previous research.

Table 1. Comparison of hydrogen yield from several mixtures by previous researchers.

No	Mixture	Temperature	Hydrogen yield (mL/g) in 60 min	Citation
1	MgH ₂ -0.5 M AlCl ₃ (solution)	30 °C	1683	(Gan et al., 2018)
2	MgH ₂ -4.5 wt% NH ₄ Cl(solution)	60 °C	1658	(Huang et al., 2015)
3	Current work	50 °C	1488	
4	MgH ₂ - 10 wt%CoCl ₂ (solid)	30 °C	1475	(Zhou et al., 2021)
5	MgH ₂ -10%NH ₄ Cl (solid)	Room temperature	1311	(Li et al., 2017)
6	MgH ₂ -0.5 wt% NH ₄ Cl(solution)	60 °C	1144	(Huang et al., 2015)
7	MgH ₂ -10%MgCl ₂ (solid)	Room temperature	1055	(Li et al., 2017)
8	MgH ₂ -10%NaCl (solid)	Room temperature	989	(Li et al., 2017) (Zhou et al., 2021)
9	Current work	30 °C	985	
10	MgH ₂ - 10 wt%CaCl ₂ (solid)	30 °C	661	(Zhou et al., 2021)
11	MgH ₂ - 10 wt%NaCl ₂ (solid)	30 °C	600	(Zhou et al., 2021)
12	MgH ₂ - 10 wt%KCl (solid)	30 °C	600	(Zhou et al., 2021)
13	MgH ₂ - 5 wt%AlCl ₃ (solid)	-	557	(Korablov et al., 2021)

4. Conclusions

Though MgH₂ offers potential as a hydrogen storage material, its real-world application is constrained by the creation of a passivating layer of Mg(OH)₂ layer during hydrolysis. This research demonstrated that ball milling can boost the hydrogen yield up to 5 times higher than pure MgH₂. The incorporation of 10 wt% of PdCl₂ further enhanced the hydrogen release, showing a 131% increase relative to the milled MgH₂. The enhancement in hydrogen release is attributed to the ball milling process in conjunction with PdCl₂, which significantly increases the total surface area available for the hydrolysis reaction. This increase in surface area, coupled with reduced particle agglomeration, ensures a more efficient interaction between water and MgH₂ particles. Additionally, the presence of PdCl₂ plays a crucial role in modifying the chemical environment of the reaction. It aids in reducing the pH of the system, a factor that not only mitigates the formation of the passivating Mg(OH)₂ layer but also promotes faster hydrolysis. This dual function of PdCl₂ enhancing the physical properties of MgH₂ through surface area expansion and chemically tuning the hydrolysis environment leads to a substantial improvement in hydrogen production. Thus, the study concluded that PdCl₂ effectively expedites MgH₂ hydrolysis through particle surface modification and pH adjustment, thereby facilitating greater hydrogen production during hydride hydrolysis.

Conflict of interest

The authors have no conflict of interest to declare.

Acknowledgements

The authors would like to acknowledge the Ministry of Higher Education Malaysia for the financial support provided under the Fundamental Research Grant Scheme FRGS/1/2020/STG05/UM T/02/3.

The authors would like to express gratitude to Universiti Malaysia Terengganu and Universiti Sultan Zainal Abidin for providing the equipments and facilities to conduct this project. Additionally, we extend our profound appreciation to our co-authors for their significant contributions, unwavering support, and collaborative efforts throughout this research and the co-authors for this research.

Funding

This work was financially supported by the Ministry of Higher Education Malaysia under the Fundamental Research Grant Scheme FRGS/1/2020/STG05/UMT/02/3.

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