

Research Article

Study on Hydrolysis of Magnesium Hydride by Interface Control

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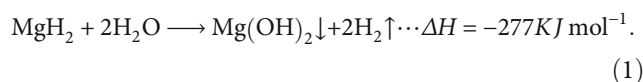
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Magnesium hydride (MgH_2) is one of the competitive hydrogen storage materials on account of abundant reserves and high hydrogen content. The hydrolysis of MgH_2 is an ideal and controllable chemical hydrogen generation process. However, the hydrolyzed product of MgH_2 is a passivation layer on the surface of the magnesium hydride, which will make the reaction continuity worse and reduce the rate of hydrogen release. In this work, hydrogen generation is controllably achieved by regulating the change of the surface tension value in the hydrolysis, a variety of surfactants were systematically investigated for the effect of the hydrolysis of MgH_2 . In the meantime, the passivation layer of MgH_2 was observed by scanning electron microscope (SEM), and the surface tension value of the solution with different surfactants were monitored, investing the mechanism of hydrolysis adding different surfactants. Results show that different surfactants have different effects on hydrogen generation. The hydrogen generation capacity from high to low is as follows: tetrapropylammonium bromide (TPABr), sodium dodecyl benzene sulfonate (SDBS), Ecosol 507, octadecyl trimethyl ammonium chloride (OTAC), sodium alcohol ether sulfate (AES), and fatty methyl ester sulfonate (FMES-70). When the ratio of MgH_2 to TPABr was 5:1, the hydrogen generation was increased by 52% and 28.3%, respectively, at the time of 100 s and 300 s. When hydrolysis time exceeds 80 s, the hydrogen generation with AES and FMES-70 began to decrease; it was reduced by more than 20% at the time of 300 s. SEM reveals that surfactants can affect the crystalline arrangement of $\text{Mg}(\text{OH})_2$ and make the passivation layer three-dimensionally layered providing channels for H_2O molecules to react with MgH_2 .

1. Introduction

With the rapid development of industrial technology, the global demand for energy is growing exponentially. Fossil fuels, as the most widely used energy materials, are not renewable, environmental pollution, and other defects; therefore, it is an urgent problem to find new clean and efficient energy sources at the present stage. Hydrogen has the characteristics of environmental protection, renewable, and high heat energy. Since the 1970s, it has been widely concerned by researchers. Hydrogen storage and release technology limits the development and application of hydrogen energy [1–4]. Among many hydrogen storage materials, the hydrogen content of MgH_2 reaches 7.69% (wt. %), and its theoretical hydrolysis hydrogen yield is 15.3% (wt. %) [5, 6]. Magnesium hydride is considered one of the best choices for portable hydrogen fuel cells due to stable storage and mild

hydrolysis [7–19]. The chemical equation of the reaction between MgH_2 and H_2O is as following:



The magnesium hydroxide is difficult to dissolve in water whose solubility product is $5.6 \times 10^{-12} \text{ mol}^3 \cdot \text{L}^{-3}$. It easily forms the passivation layer during the hydrolysis process, prevents the diffusion of water molecules toward the surface of magnesium hydride, reduces the rate, and shortens the duration of hydrolysis reaction [20–22]. Eliminating the cladding effect of magnesium hydroxide passivation layer on MgH_2 has become an urgent problem to be solved in the hydrogen yield of hydrolysis [23].

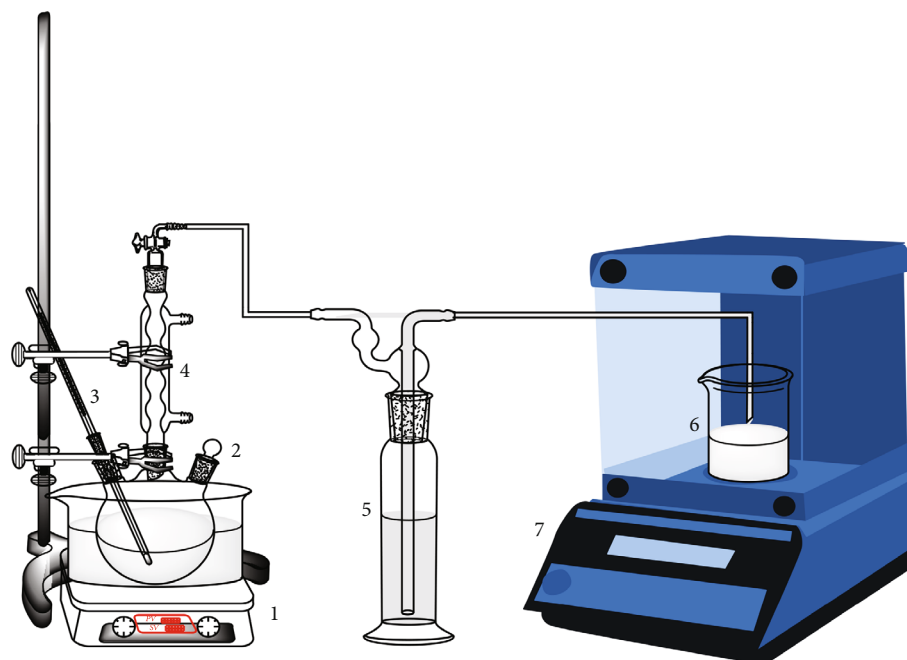


FIGURE 1: Hydrolysis unit. ((1) water bath, (2) three-necked flask, (3) thermometer, (4) condenser, (5) gas washing bottle, (6) beaker, and (7) analytical balance).

As we all know, surfactants can change the surface tension of the solution by means of forming an adsorbed layer with a certain orientation at the solid-liquid interface [24–27]. Based on the above principles, this study selected several typical surfactants to study the influence on the quantity of hydrogen generation and the rate of hydrogen generation. These data were combined with the surface tension value of the aqueous solution and the scanning electron micrograph of the product to analyze the corresponding mechanism. The study of interface control in this work provides the theoretical basis for the future researches of H_2 generation by hydrolysis of MgH_2 .

2. Experimental

2.1. Experimental Materials. H_2 is generated from the reaction of MgH_2 (purity $\geq 99.5\%$, MG Power Technology Co., Ltd) and H_2O (deionized water). To investigate the effects of surfactants on the generation of H_2 , different series of surfactants were added into deionized water: sodium dodecyl benzene sulfonate (SDBS, purity $\geq 90.0\%$, Shandong Yousuo Chemical Technology Co., Ltd), fatty methyl ester sulfonate (FMES-70, purity $\geq 70.0\%$, Shandong Yousuo Chemical Technology Co., Ltd), sodium alcohol ether sulfate (AES, purity $\geq 70\%$, Shandong Yousuo Chemical Technology Co., Ltd), tetrapropylammonium bromide (TPABr, purity $\geq 99.0\%$, Sino-pharm Chemical Reagent Co., Ltd), octadecyl trimethyl ammonium chloride (OTAC, purity $\geq 99.5\%$, Shandong Yousuo Chemical Technology Co., Ltd), and Ecosol 507 (purity $\geq 90.0\%$, Shandong Yousuo Chemical Technology Co., Ltd).

2.2. Experimental Device and Process. The experimental device (Figure 1) is composed of the reaction system and

the metering system. The reaction system is composed of a 250 mL three-necked flask, a condenser, a thermometer, and a water bath; the metering system is composed of a gas washing bottle, a beaker, and an analytical balance. The two systems are connected with silicone tubes.

Firstly, add 200 mL of water to a 250 mL three-necked flask. After the temperature of the water bath reaches $70^\circ C$, surfactant and stir were added. Then, the magnesium hydride was put to the three-necked flask. The hydrogen produced by hydrolysis is condensed by the condenser. An equal volume of water of gas washing bottle was discharge. Finally, the volume of hydrogen generated was calculated as follows:

$$V_{H_2} = \frac{m_{H_2O}}{\rho_{H_2O}} \quad (2)$$

2.3. Analytical Method. The surface morphology and dispersion state of magnesium hydride and hydrolyzed products are tested by SEM (Quanta 250, FEI); the surface tension (ST) of aqueous solutions with different surfactants is tested by surface tension meter K100C-MK2.

3. Results and Discussion

Surfactants with different groups, structures, and dosages have different impacts on the surface energy and the wetting effect between solid and liquid.

3.1. Effect of Anionic Surfactant. According to the structure of hydrophilic groups, anionic surfactants mainly include sulfonate and sulfate ester salts. Typical anionic surfactants were used in this study: SDBS, AES, and FMES-70.

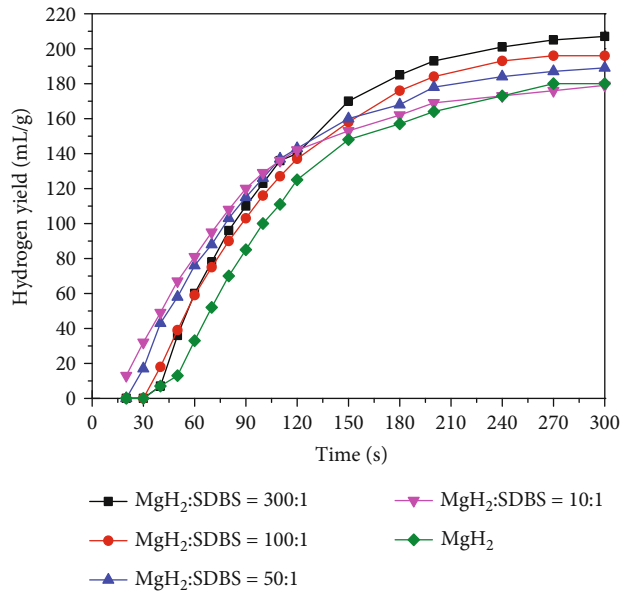


FIGURE 2: The effect of different SDBS dosages on hydrogen generation.

TABLE 1: Surface tension values of aqueous solutions with different SDBS dosages.

Ratio*	ST (mN/m)
Water	71.57
MgH ₂ : SDBS = 300 : 1	70.51
MgH ₂ : SDBS = 100 : 1	59.00
MgH ₂ : SDBS = 50 : 1	47.80
MgH ₂ : SDBS = 10 : 1	38.48

*: the ratio represents dosages of surfactants in experiments without MgH₂.

Under the same condition, the volume of the water, the temperature of the water bath, and the mass of MgH₂, surfactants with different dosages were added, time (seconds) is the x -coordinate, and hydrogen yield (mL/g) is the y -coordinate.

It is shown in Figure 2 that the hydrogen generation rate are approximately the same at 110 s when MgH₂ : SDBS = 300 : 1, MgH₂ : SDBS = 50 : 1, and MgH₂ : SDBS = 10 : 1. While the hydrogen generation rate is slightly lower when MgH₂ : SDBS = 100 : 1 at 110 s. When the hydrolysis time is 120 s, the hydrogen generation rate of MgH₂ : SDBS = 300 : 1 is greater than the other. When the hydrolysis time is 155 s, the hydrogen generation rate of MgH₂ : SDBS = 100 : 1 is higher than MgH₂ : SDBS = 10 : 1 and MgH₂ : SDBS = 50 : 1. All the hydrogen generation rate reduces gradually over 200 s. The final hydrogen yield are ranked as follows: MgH₂ : SDBS = 300 : 1 > MgH₂ : SDBS = 100 : 1 > MgH₂ : SDBS = 50 : 1 > MgH₂ : SDBS = 10 : 1 = MgH₂.

The hydrogen generation rate will improve with the increase of SDBS dosages (time ≤ 100 s). It is possible that the surface tension of water becomes lower as surfactants increase (it is presented in Table 1). However, the lower the surface tension, the easier bubbles will foam. These bubbles

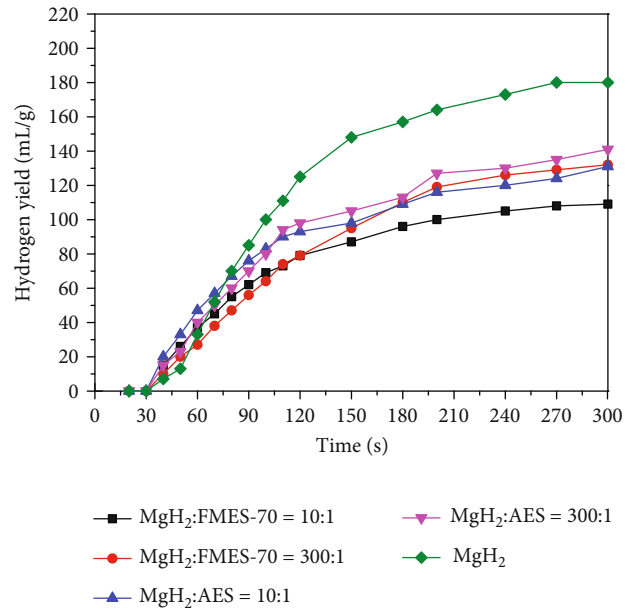


FIGURE 3: The effect of different FMES-70, AES dosages on hydrogen generation.

TABLE 2: Surface tension values of aqueous solutions with different FMES-70, AES dosages.

Ratio	ST (mN/m)
Water	71.57
MgH ₂ : FMES-70 = 300 : 1	45.25
MgH ₂ : FMES-70 = 10 : 1	39.74
MgH ₂ : AES = 300 : 1	30.05
MgH ₂ : AES = 10 : 1	37.86

gather into a foam layer, which binds H₂. It is difficult for H₂ escape from the reaction system (e.g., the curve of MgH₂ : SDBS = 10 : 1 in the Figure 2). Besides, the SDBS concentration is bigger than the critical micelle concentration (CMC = 1.47×10^{-3} – 1.60×10^{-3} mol/L) when MgH₂ : SDBS = 10 : 1. Self-polymerization process of SDBS molecules possibly employed, which inhibits the detachment of Mg(OH)₂ from the surface of MgH₂ and the dispersion of Mg(OH)₂ in the water.

From Figure 3, the hydrogen generation of two experiments (MgH₂ : FMES = 10 : 1, MgH₂ : AES = 10 : 1) are 100% and 153.8% at the hydrolysis time of 50 s, which is higher than that without surfactant. When the hydrolysis time is over 75 s, the generation rate of four experiments (MgH₂ : FMES-70 = 300 : 1, MgH₂ : FMES-70 = 10 : 1, MgH₂ : AES = 300 : 1, and MgH₂ : AES = 10 : 1) are lower than that without surfactants.

It is observed from Table 2 that surface tension of solutions becomes lower as the ratio of MgH₂ to surfactants increases. On one hand, the system foams more easily [28, 29], and the foam layer weakens the diffusion of H₂. On the other hand, the hydrolysis reaction is essentially a

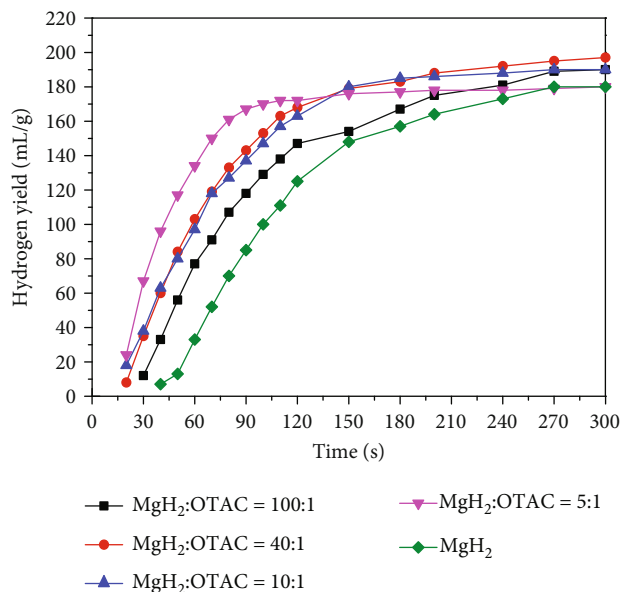


FIGURE 4: The effect of different OTAC dosages on hydrogen generation.

TABLE 3: Surface tension values of aqueous solutions with different OTAC dosages.

Ratio	ST (mN/m)
Water	71.57
MgH ₂ :OTAC = 100:1	36.87
MgH ₂ :OTAC = 40:1	35.79
MgH ₂ :OTAC = 10:1	34.24
MgH ₂ :OTAC = 5:1	32.99

reaction with H⁺, while the negatively charged groups of FMES-70 and AES may combine with H⁺ decreasing the hydrogen generation.

3.2. Effect of Cationic Surfactant. Cationic surfactants are mainly nitrogen-containing organic amine derivatives, composed of a long-chain hydrophobic group and a positive charged hydrophilic group. With good emulsification, wetting effect, and other properties, it is easier to absorb on the solid surface and improves the solid-liquid interface effect.

It is shown in Figure 4 that the hydrogen generation of MgH₂:OTAC = 5:1 and MgH₂:OTAC = 100:1 are 70% and 29% higher than that without surfactants at time of 100 s. When the hydrolysis time is over 100 s, the hydrogen generation curves gradually become smooth. At the time of 300 s, the hydrogen generation of MgH₂:OTAC = 40:1 reaches the maximum which is 11.0% higher than that without surfactants.

Within 120 s of the hydrolysis, the hydrogen generation and the hydrogen generation rate improve as the ratio of MgH₂ to OTAC. After 120 s, the foam layer hinders the continuation of the hydrolysis. It can be seen from Table 3 that the surface tension of MgH₂:OTAC = 100:1 decrease to 36.87 mN/m. When MgH₂:OTAC = 5:1, the surface tension

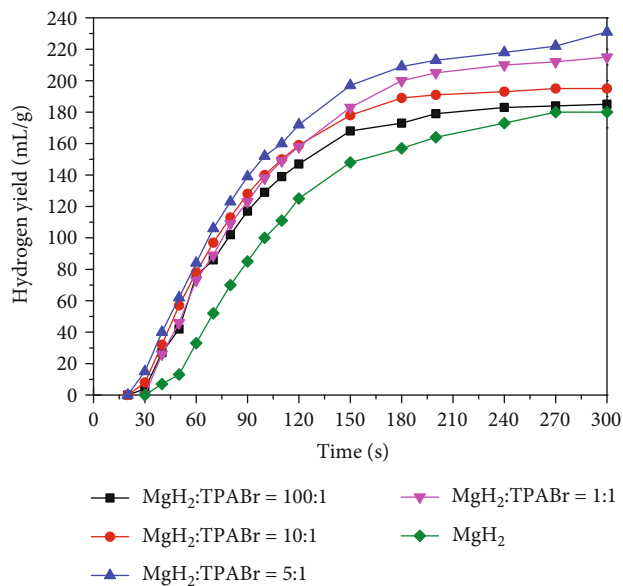


FIGURE 5: The effect of different TPABr dosages on hydrogen generation.

TABLE 4: Surface tension values of aqueous solutions with different TPABr dosages.

Ratio	ST (mN/m)
Water	71.57
MgH ₂ :TPABr = 100:1	61.54
MgH ₂ :TPABr = 10:1	65.39
MgH ₂ :TPABr = 5:1	68.67
MgH ₂ :TPABr = 1:1	69.23

decreases to 32.99 mN/m slightly. Therefore, OTAC reduces the surface energy of solution preventing the agglomeration of Mg(OH)₂ between crystals.

It is also possible that the positively charged groups of the cationic surfactants may combine with OH⁻ inhibiting the combination of OH⁻ and Mg²⁺ and delay the formation of the passivation layer in the surface of MgH₂. Zheng et al. [30] studied the hydrolysis of Mg in different solutions (MgCl₂, MnCl₂, NiCl₂, AlCl₃, NH₄Cl, and HCl). They found that there was a significant improvement in NH₄Cl and HCl solution, the conversion efficiency increased by more than 60%. Researchers believe that the higher the affinity between the cations and OH⁻ in the solution, the more effective it is to inhibit the formation of the passivation layer.

In addition, the groups of the cationic surfactants can absorb on Mg(OH)₂ along the direction of growth and prevent its growth [31].

Figure 5 reveals the effect of TPABr on hydrogen generation. When the time is 100 s, the hydrogen generation of MgH₂:TPABr = 5:1 and MgH₂:TPABr = 100:1 increases by 52% and 29% compared with that without surfactants, respectively. At the time of 300 s, the hydrogen generation of MgH₂:TPABr = 5:1 and MgH₂:TPABr = 100:1 increases by 28.3% and 3%.

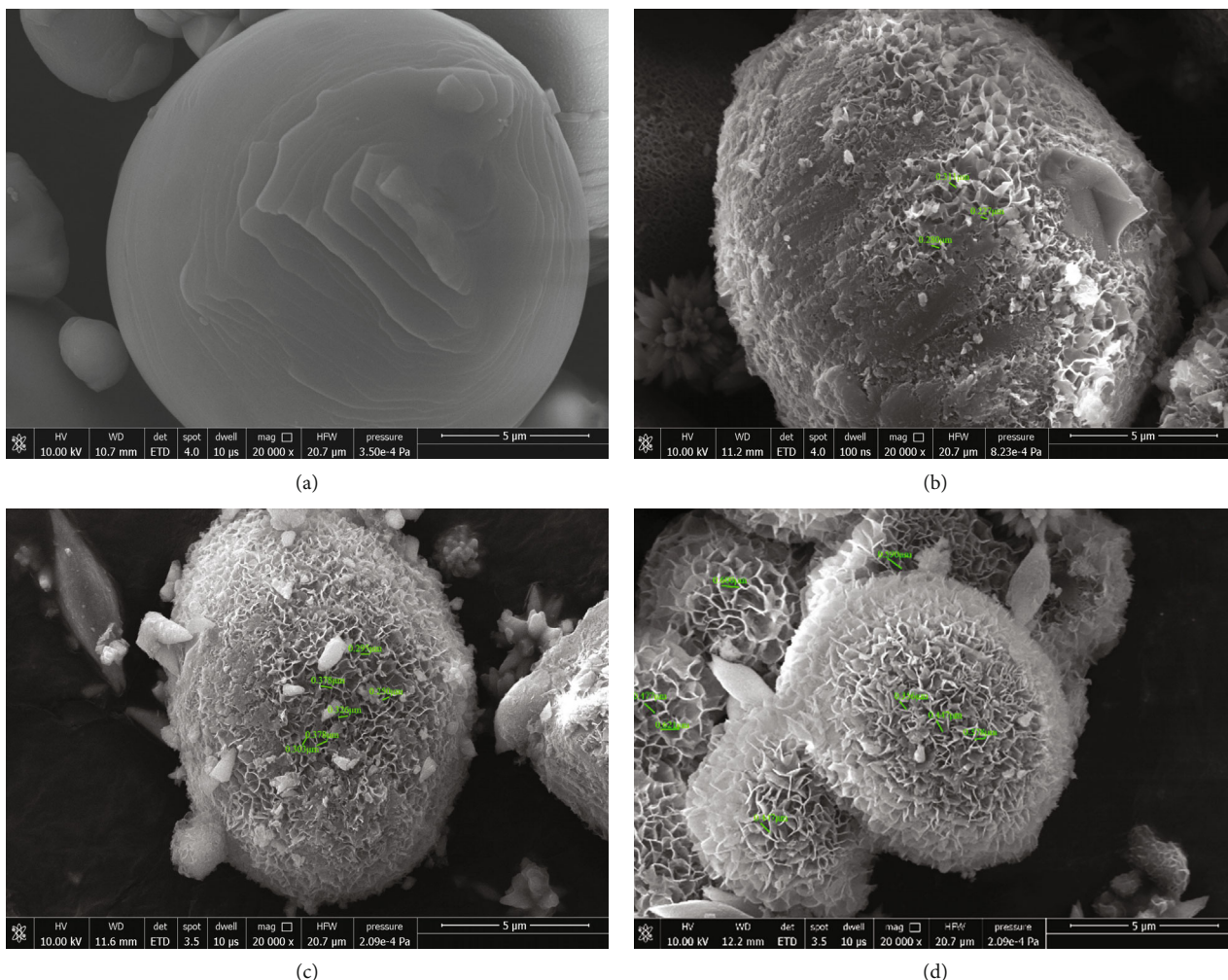


FIGURE 6: (a) Magnesium hydride. (b) Hydrolyzed product without surfactants. (c) MgH₂:SDBS = 300:1, hydrolyzed product. (d) MgH₂:TPABr = 1:1, hydrolyzed product (×20000).

According to the previous results in this work, the surface tension of solution decreases with the increase of the ratio of MgH₂ to surfactants. At the initial stage of the reaction, the lower the surface tension, the more hydrogen generation. And at the middle and late stages, the foam layer caused by the low surface tension impedes the hydrogen generation. However, there are some differences when TPABr added to the solution. As shown in Table 4, the surface tension decreases from 71.57 mN/m to 61.54 mN/m and then increases to 69.23 mN/m with increase of the ratio of MgH₂ to TPABr. And the hydrogen generation has the same trend, the final hydrogen yield are ranked as follows: MgH₂:TPABr = 5:1 > MgH₂:TPABr = 1:1 > MgH₂:TPABr = 10:1 > MgH₂:TPABr = 100:1 > MgH₂.

Figure 6(d) illustrates that Mg(OH)₂ grows evenly around the surface of MgH₂, like a blooming flower. The “petals”-Mg(OH)₂ form uniform channels instead of coating MgH₂ in the form of flakes. Because of the better passability, H₂O molecules can contact MgH₂ through these channels. Contrarily, the high concentration of TPABr may cause Mg(OH)₂ to grow as many points as possible on the surface of MgH₂ forming narrow and long channels. As a result, it

is difficult for H₂O molecules to reach the surface of MgH₂ through the channels.

When the surface tension decreases to 61.54 mN/m, there is no obvious foam layer. Thus, H₂ can easily escape from the solution. In addition, TPABr inhibits the formation of passivation layer and prolongs the time for Mg(OH)₂ to reach the critical volume. It may also because of the adsorptive effect of TPABr. When TPABr absorbs on the surface of Mg(OH)₂ newly formed and the adsorption layer cannot only inhibit the growth of the Mg(OH)₂ [31, 32], it also reduces the surface energy. Therefore, the interaction among the particles is weakened, and it prevents the agglomeration among the particles [33–35].

3.3. Effect of Nonionic Surfactant. Nonionic surfactants are different in structure from other ionic surfactants. The main hydrophilic group is an ether group that does not dissociate in aqueous solution. It has an excellent wetting effect and superior antideposition ability for particles.

As shown in Figure 7, when the time is 100 s, the hydrogen generation of MgH₂:Ecosol 507 = 100:1 is 50% higher

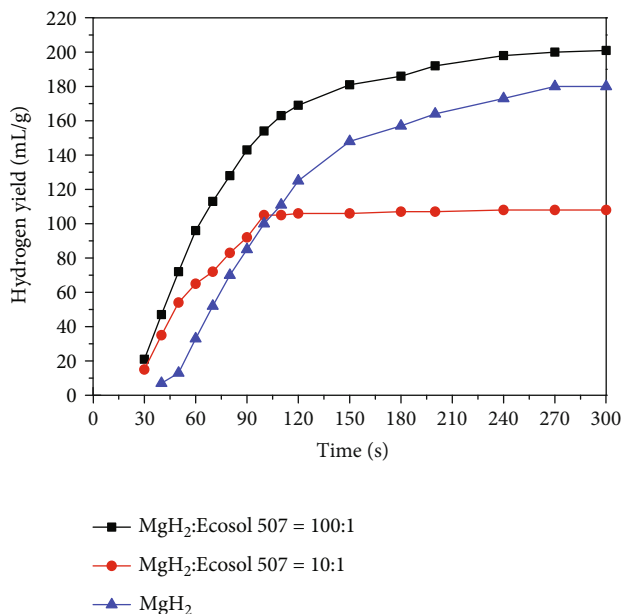


FIGURE 7: The effect of different Ecosol 507 dosages on hydrogen generation.

TABLE 5: Surface tension values of aqueous solutions with different Ecosol 507 dosages.

Ratio	ST (mN/m)
Water	71.57
MgH ₂ : Ecosol 507 = 100 : 1	28.38
MgH ₂ : Ecosol 507 = 10 : 1	20.49

than that without surfactants. When the time is 300 s, the hydrogen generation of MgH₂:Ecosol 507 = 10:1 is 40% lower than that without surfactants, but the hydrogen generation of MgH₂:Ecosol 507 = 100:1 is 11.7% higher than it.

It can be seen from Table 5 that Ecosol 507 significantly reduces the surface tension. In this experiment, we observed the volume of the foam was far much larger than the other surfactants. When the reaction time is over 100 s, the foam of MgH₂:Ecosol 507 = 10:1 fills the whole three-necked flask and seriously weakens diffusion of H₂.

3.4. Comparison of Maximum Hydrogen Generation. Sort by maximum hydrogen generation (Figure 8): TPABr > SDBS > Ecosol 507 > OTAC > no surfactants > AES ≈ FMES – 70.

3.5. Analysis of SEM. In order to further explore the influence of surfactants on the hydrogen generation and the hydrogen generation rate, samples are observed and analyzed by SEM.

Figure 6 shows the scanning electron microscope pictures of the products of four illustrative experiments. The surface of MgH₂ particles used in the experiments can be observed in Figure 6(a) with a smooth surface and localized layered deposition. Figure 6(b) shows the morphology of MgH₂ after the hydrolysis reaction without surfactants. The smooth surface is pitted by H₂O molecules. There are two forms of Mg(OH)₂ on the surface of MgH₂. One forms a dense passiv-

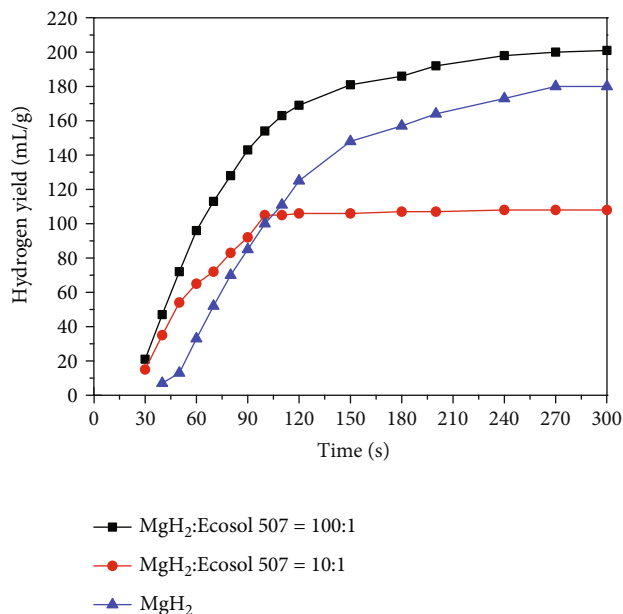


FIGURE 8: Comparison of hydrogen generation curves of MgH₂.

ation layer that prevents H₂O molecules from contacting with MgH₂, and the other forms a discontinuous layered structure in the form of three-dimensional stacking that provides channels for H₂O molecules.

Surfactants can improve the morphology of the hydrolysate through interface control. After adding SDBS (MgH₂:SDBS = 300:1), it is observed that the area of the coating layer in Figure 6(c) is significantly smaller than that in Figure 6(b), the relative stratigraphic structure becomes more and more evenly distributed on the hydrogenated, and the number and diameter of the channels have also increased. Therefore, the addition of SDBS increases the hydrogen generation and the hydrogen generation rate of the reaction system compared without surfactants. On one hand, adding TPABr increases the growth sites of Mg(OH)₂ to make the distribution of Mg(OH)₂ more uniform; on the other hand, it makes the directional growth of Mg(OH)₂, which further changes the morphology. Compared with Figure 6(b), the hydrolyzed product in Figure 6(d), there is no cladding layer; instead, a layered structure is formed by three-dimensional accumulation. Compared with Figure 6(c), the layered structure in Figure 6(d) is more uniform and stable (i.e., the distribution and the diameter of channels is more uniform and larger), and it allows more H₂O molecules to reach the surface of MgH₂ improving the hydrogen generation rate. At the same time, Mg(OH)₂ crystals formed by the reaction grow in the form of three-dimensional accumulation instead of encapsulating MgH₂, so that H₂O can react with MgH₂ constantly.

4. Conclusion

- (1) The addition of surfactants reduces the surface tension of the liquid, improves the wetting effect of the liquid on the MgH₂, and increases the hydrogen

generation and the hydrogen generation rate at the initial stage of the hydrolysis

- (2) With the addition of surfactants, the reaction system is more prone to foam. When these foams gather to form a foam layer, it hinders the escape of hydrogen and reduces the hydrogen generation and the hydrogen generation rate
- (3) Surfactants can change the morphology of $\text{Mg}(\text{OH})_2$. $\text{Mg}(\text{OH})_2$ stake in three-dimensions to form a discontinuous layered structure improving the continuity of the hydrolysis reaction
- (4) When the surfactant concentration reaches CMC, the self-polymerization of surfactants inhibits the separation of $\text{Mg}(\text{OH})_2$ and affects the dispersion effect
- (5) On one hand, the negatively charged groups of surfactants may combine with H^+ in the water, which reduces the concentration of H^+ affecting the hydrogen generation; on the other hand, the positively charged groups may combine with OH^- inhibiting the nucleated growth of $\text{Mg}(\text{OH})_2$

Data Availability

The experimental data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare no conflict of interest.

Acknowledgments

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