



A novel design of solid-state NaBH₄ composite as a hydrogen source for 2 W PEMFC applications

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ABSTRACT

Hydrolysis of sodium borohydride (NaBH₄) is a promising method for on-board hydrogen supply to polymer electrolyte membrane fuel cells (PEMFC). This article presents an attempt to design a novel solid-state NaBH₄ composite, which is made up of NaBH₄ powder, Co²⁺/IR-120 catalyst and silicone rubber, for hydrogen generator. The silicone rubber can act as a stabilizer in the solid-state NaBH₄ composite because of its surface hydrophobicity leading to reduced diffusion rate of water into the composite. The solid-state NaBH₄ composite can produce hydrogen stably near 25 mL min⁻¹ for at least 2 h without employment of any mechanical control system. Using the hydrogen generated from the solid-state NaBH₄ composite, a 2 W PEMFC stack is successfully operated to power a cellular phone.

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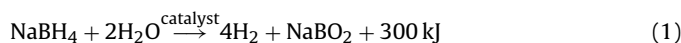
1. Introduction

Many portable electronic devices, such as cell phones, personal digital assistants (PDAs), MP3 players and digital cameras, are powered with rechargeable secondary batteries when they are not connected to grid power. When the secondary battery runs out, the device no longer works. Consequently, the battery must be either recharged or replaced with another fresh one. Unexpected running out of power always makes people frustrated. In response to the ever increasing demand of portable electronic devices that require high power density, fuel cells have attracted much attention as an alternative power source to replace the lithium-ion batteries.

Among various types of fuel cells, the direct methanol fuel cell (DMFC) has been well developed, mainly due to great availability of fuel supply. Even though the polymer electrolyte membrane fuel cell (PEMFC) has a higher efficiency in fuel consumption and a greater power density than DMFC, hydrogen supply has been an obstacle to the applications of PEMFC as portable power sources. In general, there are three applicable ways for hydrogen production on-board. These methods are all based on displacement of hydro-

gen from water by active redoxing chemicals, namely (1) hydrolysis of sodium boron hydrides in presence of suitable catalysts [1–7], (2) interaction of Al-based materials with water [8–10], and (3) interaction of Mg-based materials with water [11,12]. In light of gravimetric H₂ storage efficiency, hydrogen production by the first method, hydrolysis of sodium boranes, e.g. sodium borohydride (NaBH₄), prevails against the others.

In traditional applications, sodium borohydride (NaBH₄), with a solubility of 55 g in 100 g water at 25 °C [13], is usually dissolved in alkaline solutions for long-term preservation. Moreover, hydrogen can be controllably generated by pumping NaBH₄ solution through a catalyst bed, according to the following equation:



However, when NaBH₄ solutions are used for small portable electronic device applications, some problems are often encountered. For instance, liquid NaBH₄ solution is not easy for consumers to carry. In addition, because of the concern in solution leakage, heavy duty cartridges must be used for carrying and storing NaBH₄ solution. Furthermore, because of solubility limitations, quantity of NaBH₄ in aqueous solution that could be carried is further reduced. Accordingly, the amount of produced hydrogen is diminished.

In our previous reports [14,15], we have developed a simple method to fabricate catalysts from metal–polymer template com-

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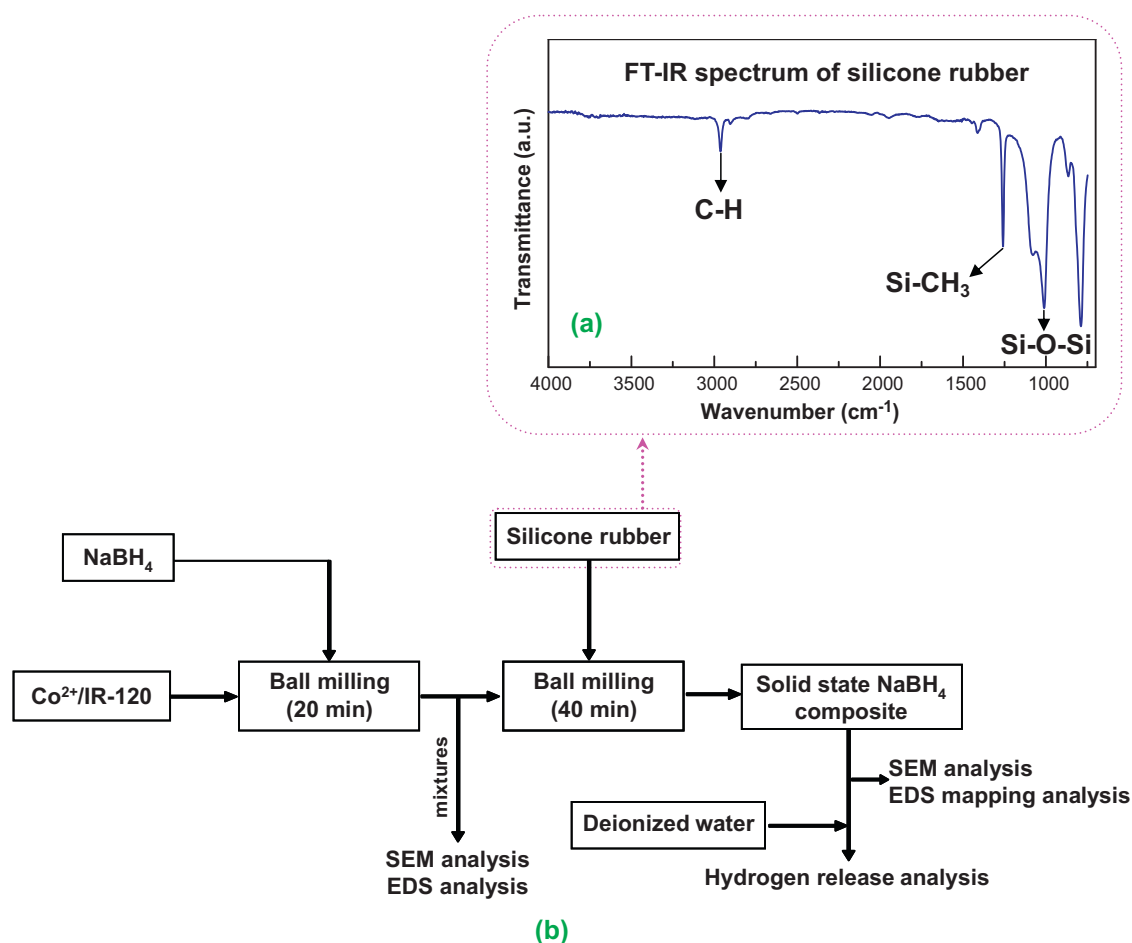


Fig. 1. (a) FTIR-ATR spectrum of silicone rubber and (b) scheme of experimental procedures.

posites, such as ruthenium and cobalt supported on Amberlite IR-120 resin beads (denoted, respectively, as Ru/IR-120 and Co/IR-120), in NaBH₄ hydrolysis for H₂ evolution. Furthermore, it was also demonstrated that sodium metaborate (NaBO₂), namely the hydrolysate of sodium borohydride (NaBH₄) for hydrogen production, could be recycled back to NaBH₄ using Mg-based materials [16,17].

In this paper, we attempt to combine NaBH₄ powder and Co²⁺/IR-120 catalysts to make a solid-state NaBH₄ composite for hydrogen storage and supply. Hydrogen could be immediately produced on demand by reacting it with water. Notably, alkalis or other additives are not required in such a hydrogen storage system. More importantly, this solid-state NaBH₄ composite could be easily made portable. Consequently, it could give consumers more convenience in usage than the conventional liquid NaBH₄ systems.

We also make an effort to design a hydrogen supply system for a 2 W PEMFC stack using these as-fabricated solid-state NaBH₄ composites. By and large, to feed the 2 W PEMFC stack, the hydrogen should be preferentially supplied at a rate higher than 24 mL min⁻¹ with a fuel stoichiometry of 1.5 [6]. Therefore, hydrophobic silicone rubber is introduced as a stabilizer in the aforementioned solid-state NaBH₄ composites to maintain a stable flow rate of hydrogen near 25 mL min⁻¹.

2. Experimental

2.1. Materials

Sodium borohydride (NaBH₄) and cobalt chloride (CoCl₂) were acquired from Riedel-de Haën (Seelze, Germany) and

Sigma-Aldrich (St. Louis, MO, USA), respectively. Both reagents were of analytical reagent grade and were used as received. The hydrophobic silicone rubber used as a stabilizer was purchased from Kwang Way Co. (Taipei, Taiwan). Owing to existence of methyl groups on this silicone rubber, it possesses enough hydrophobicity to resist water wetting onto its surface (Fig. 1a). A strongly acidic cation exchange resin, Amberlite IR-120 in hydrogen form (16–45 mesh size), used as catalyst support, was obtained from Supelco (Bellefonte, PA, USA). The properties of Amberlite IR-120 were also given in details in our previous publication [14].

The synthesis procedure of the Co²⁺/IR-120 catalysts is summarized below. Suitable amounts of cobalt chloride were dissolved in deionized water. Subsequently, a predetermined quantity of Amberlite IR-120 cationic exchange resin beads was added to the CoCl₂ solution that had been stirred for 1 h at the ambient temperature. After chelating with Co²⁺, surface chemistry of Amberlite IR-120 resin beads was transformed from R₂SO₃⁻-H⁺ (R₂ = polymer matrix of the resin) into (R₂SO₃⁻)₂-Co²⁺ (Co²⁺/IR-120). Then, the resin beads were filtrated and washed repeatedly with deionized water. Subsequently, they were dried at 120 °C for 24 h to eliminate any residual water. Finally, the polymer template-Co²⁺ composite (Co²⁺/IR-120) catalyst was obtained.

2.2. Preparation of solid-state NaBH₄ composite

Sequential experiments shown in Fig. 1b were designed to prepare solid-state NaBH₄ composites under ambient conditions.

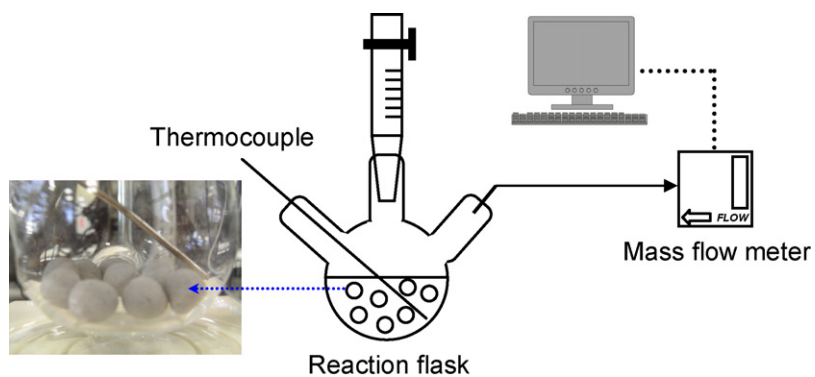


Fig. 2. Schematic of the experimental setup.

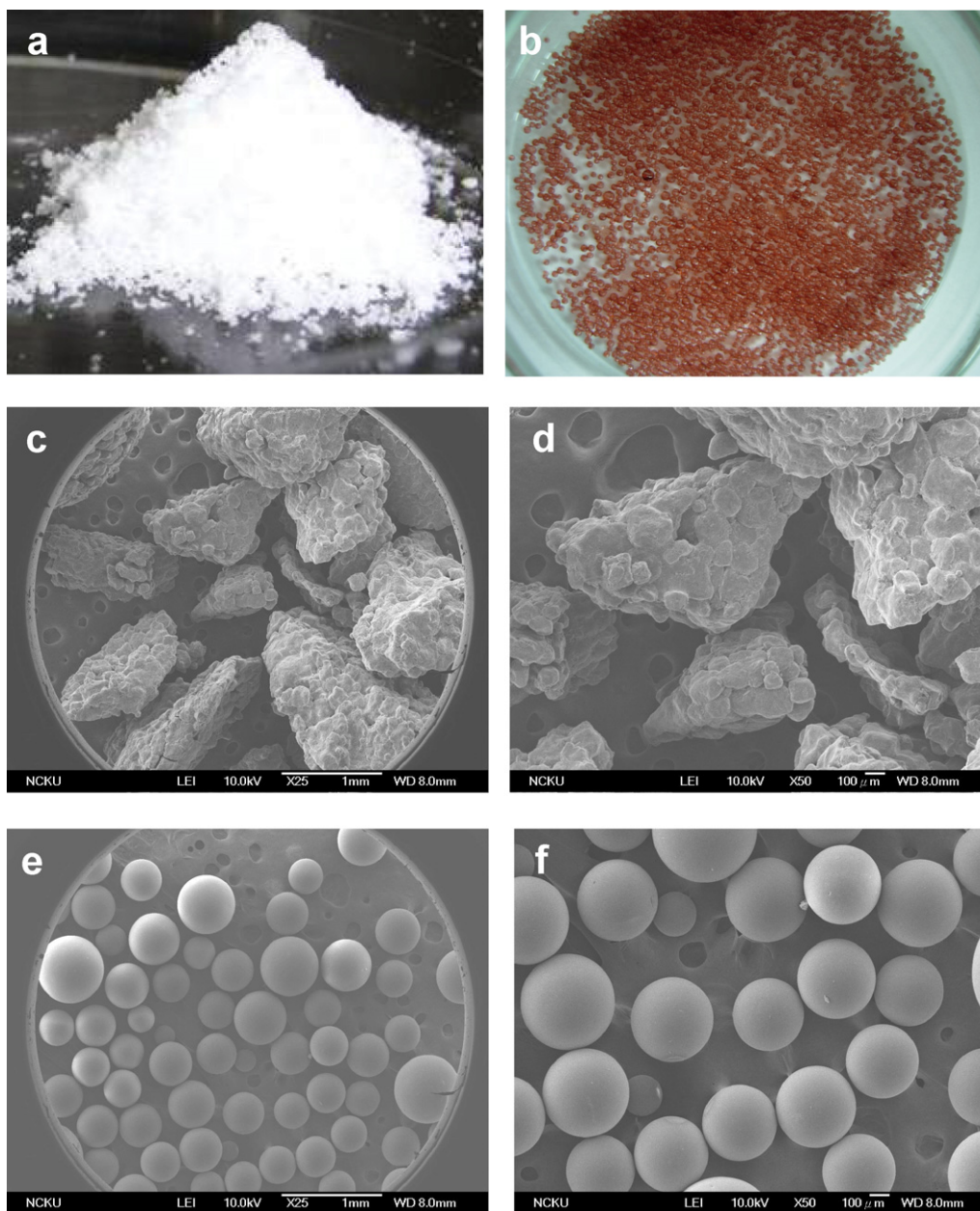


Fig. 3. Photographs of original (a) NaBH_4 powder; (b) $\text{Co}^{2+}/\text{IR-120}$ catalyst; and SEM images of original NaBH_4 powder: (c) 25 \times ; (d) 50 \times and $\text{Co}^{2+}/\text{IR-120}$ catalyst: (e) 25 \times ; (f) 50 \times .

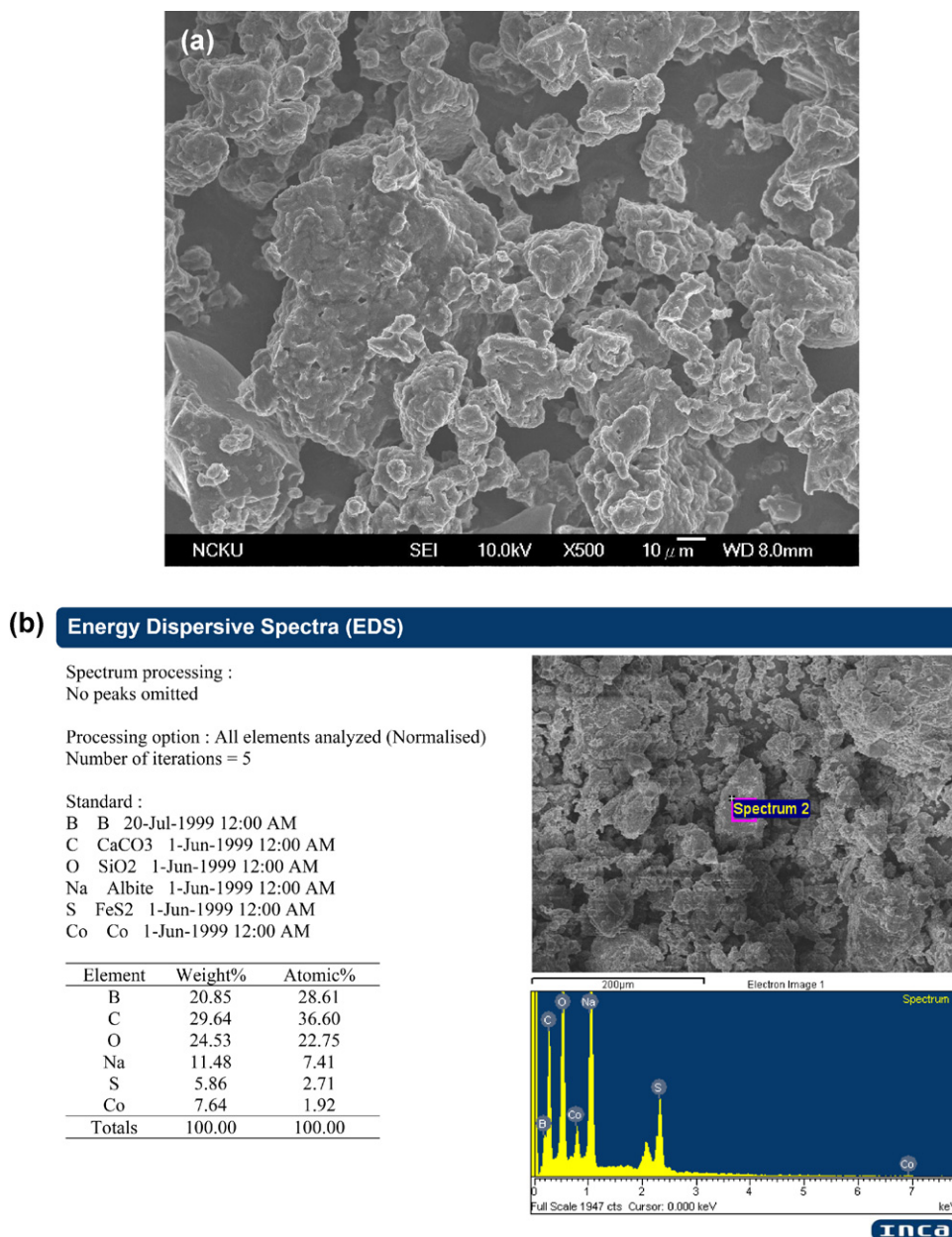


Fig. 4. (a) SEM images and (b) EDS spectrum of powder produced after ball-milling the $\text{NaBH}_4\text{-Co}^{2+}$ /IR-120 mixture.

Blend of raw materials, including NaBH_4 powder, Co^{2+} /IR-120 catalyst and silicone rubber, was pulverized in the high-energy ball mill (SPEX CertiPrep 8000-series Mixer/Mills) at room temperature. This ball mill shook containers back and forth approximately 1080 cycles per minute.

Initially, predetermined amounts of NaBH_4 powder and Co^{2+} /IR-120 catalyst were placed into a 65-mL grinding bowl with two grinding balls having diameters of 13 mm. After 20 min of ball milling, a preset quantity of silicone rubber was added into the grinding bowl and, then, ball-milled again for another 40 min. Finally, the solid-state NaBH_4 composite was obtained. The solid-state NaBH_4 composite prepared by the above-mentioned method was of plastic nature and, hence, could be easily formed into various geometric shapes.

Surface morphology and chemistry of these NaBH_4 composites were observed using a scanning electron microscope (SEM; JEOL JSM-6700F) and the energy dispersive spectrometer (EDS; Oxford INCA-400) installed on this JEOL JSM-6700F, respectively.

2.3. Generation of hydrogen

Fig. 2 depicts the experimental apparatus devised to simultaneously measure the instantaneous H_2 generation rate from hydrolysis of solid-state NaBH_4 composite with 20 g of deionized water, and to record the temperature of this hydrolysis reaction. Notably, no temperature control device was employed on the reacting system. That is, the reacting system was exposed to ambient conditions. The hydrogen generation rate and reaction temperature were measured using a mass flow meter (MFM) and recorded with a personal computer.

3. Results and discussion

3.1. Characterization of solid-state NaBH_4 composite

In a typical hydrogen generator of liquid NaBH_4 system, crystals of hydrated sodium metaborate ($\text{NaBO}_2 \cdot x\text{H}_2\text{O}$), i.e. the main

hydrolysate of NaBH_4 for H_2 production, always appears when the concentration of sodium boride initially present in the reacting system is more than 10 wt%. The presence of hydrated metaborate crystals inevitably affects the subsequent H_2 production, especially when crystals emerge on the surface of the catalyst and clog active sites of catalyst [18]. That is, the hydrolysis reaction of NaBH_4 for hydrogen evolution is deteriorated as active reaction sites on catalyst are blocked by these as-produced metaborate precipitates [18]. Thus, to make the hydrolysis reaction proceed as completely as possible even after metaborate crystals appearing on catalyst surface, it is of utmost importance for these catalysts able to be evenly and finely distributed within the reacting system [18].

Among all possible methods to make well dispersion of reactants and catalyst, the high-energy ball milling process has been proven to be able to serve the purpose [15,16]. In this work, $\text{Co}^{2+}/\text{IR}-120$ catalyst granules were pulverized and uniformly blended with NaBH_4 powder to make the solid-state NaBH_4 composite. Consequently, hydrogen could be produced immediately after introduction of water into the solid composite. Explicitly, the hydrolysis reaction of NaBH_4 could proceed as quickly as possible in response to demand of hydrogen, and could also lead to an almost complete conversion of NaBH_4 to hydrogen.

Fig. 3 displays the optical photographs and SEM images of pristine NaBH_4 powder and $\text{Co}^{2+}/\text{IR}-120$ catalysts. The appearance of NaBH_4 powder and $\text{Co}^{2+}/\text{IR}-120$ is white and pink, respectively, as shown in Fig. 3(a) and (b). Moreover, fresh NaBH_4 powder is chunky with rock-like morphology and has a size varying from 0.5 to 2.5 mm, as shown in Fig. 3(c). In contrast, $\text{Co}^{2+}/\text{IR}-120$ catalyst is granular with ball-like morphology and had a size varying from 0.3 to 0.7 mm, as shown in Fig. 3(d).

Fig. 4 displays the SEM image and EDS spectrum of the $\text{NaBH}_4\text{-Co}^{2+}/\text{IR}-120$ mixture after being shaker-milled. With 20 min of ball milling, NaBH_4 powder and $\text{Co}^{2+}/\text{IR}-120$ catalyst particles were pulverized and disintegrated into fragmentation due to the significant impact forces caused with the very high shear stress generated from impactful collision of grinding balls and ground particles. The NaBH_4 and $\text{Co}^{2+}/\text{IR}-120$ mixtures became much finer and layer-like agglomeration appeared (Fig. 4(a)). These layer-like agglomerations were subject to instrumental analyses to examine whether NaBH_4 and $\text{Co}^{2+}/\text{IR}-120$ could be ground and blended well through the high-energy ball milling process. Consequently, selective area EDS analysis was performed. Fig. 4(b) presents the EDS spectrum of the ball-milled mixture of NaBH_4 and $\text{Co}^{2+}/\text{IR}-120$ catalyst, which confirmed existence of cobalt (Co) and sodium (Na) on these layer-like agglomerations. This result certainly reveals that both NaBH_4 reagent and $\text{Co}^{2+}/\text{IR}-120$ catalysts could be uniformly mixed through the high-energy ball milling process.

In order to realize the distribution of NaBH_4 and $\text{Co}^{2+}/\text{IR}-120$ in the solid-state NaBH_4 composite, EDS mapping was also performed. Fig. 5 depicts the low-magnified EDS mapping image of the cross-section of solid-state NaBH_4 composite. The dotted line on the SEM image indicates the selected area where the EDS mappings of boron (B) and cobalt (Co) were probed. With the information given by EDS mappings it was found that NaBH_4 (B) and $\text{Co}^{2+}/\text{IR}-120$ (Co) were uniformly dispersed in the solid-state NaBH_4 composite.

3.2. Hydrogen generation from solid-state NaBH_4 composite

For a traditional hydrogen generator using aqueous NaBH_4 solution, NaBH_4 solution was pumped to flow upward into the bottom of the reactor containing the honeycomb monolith [12]. The hydrogen generation rate was controlled by the contact time of the catalyst with the NaBH_4 solution. However, the hydrogen production from a liquid solution of NaBH_4 was not only limited by its solubility, but also gave inconvenience for consumers

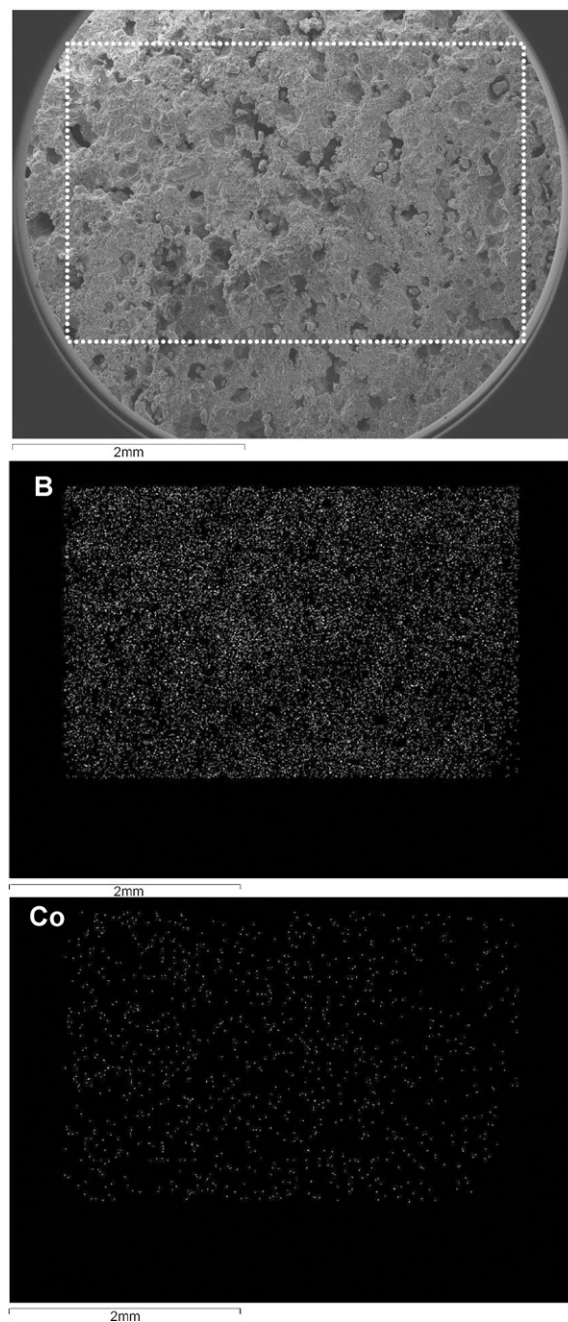


Fig. 5. SEM image and EDS mapping of solid-state NaBH_4 composite and the elemental distributions of B and Co. Scale bar = 2 mm.

to carry the bulky hydrogen generator for applications in small portable electronic devices. To resolve the aforementioned problems of liquid- NaBH_4 hydrogen generator, it is essential to study the characteristics and designs of hydrogen generation from a more compact system, especially a solid reacting system of NaBH_4 . Consequently, the answers and designs to how it could be made with a constant and controllable generation rate of hydrogen from solid-state NaBH_4 system free of any temperature-control systems were attempted in this work.

Fig. 6 shows the effect of the presence of silicone rubber in the solid-state- NaBH_4 composite on H_2 production rate and reaction temperature. The solid-state NaBH_4 composite was composed of 0.1 g $\text{Co}^{2+}/\text{IR}-120$ and 1 g sodium borohydride. Without silicone rubber present in the formula, in general, three-stage behavior in hydrogen release could be observed. During the first stage, the

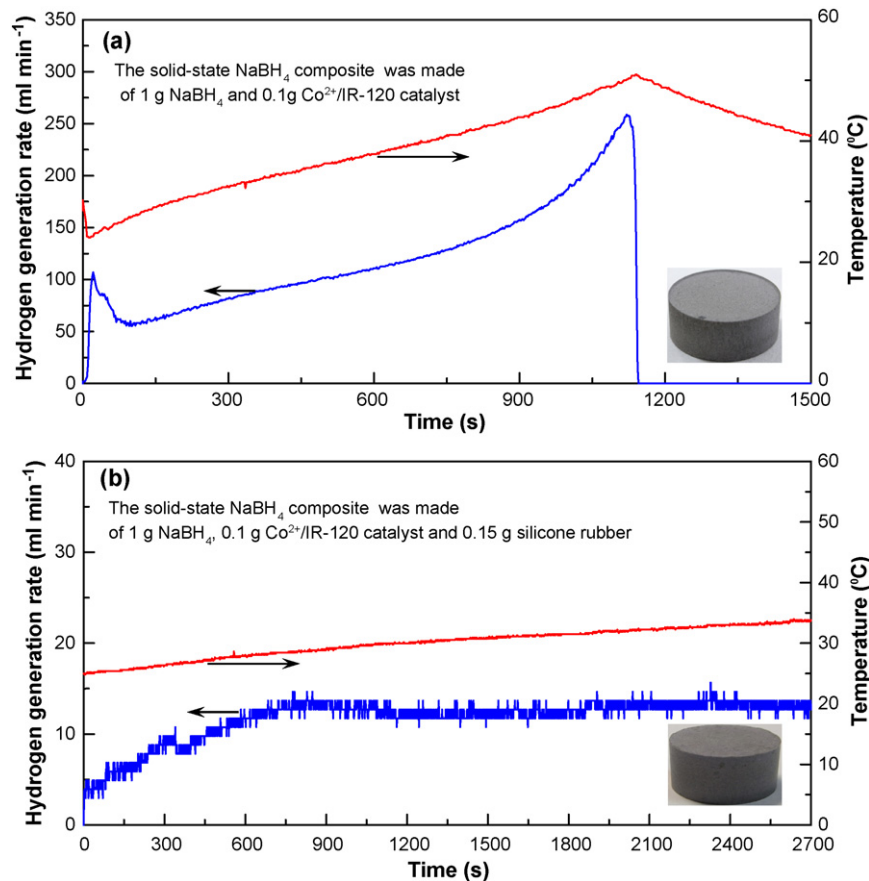


Fig. 6. Effect of the (a) nonexistence and (b) existence of silicone rubber in solid-state NaBH_4 composite on H_2 production rate and reaction temperature.

reduction reaction on $\text{Co}^{2+}/\text{IR-120}$ by NaBH_4 took place very quickly and, accordingly, the temperature of the reacting system increased sharply. In this stage, the NaBH_4 -catalyst composite turned black from original pink color immediately after 20 g of deionized water was injected into the flask containing NaBH_4 -catalyst composite. After the black $\text{Co}/\text{IR-120}$ was formed, it acted as an active catalyst for the subsequent hydrolysis reaction of NaBH_4 to produce hydrogen [15,16]. Notably, both $\text{Co}^{2+}/\text{IR-120}$ and $\text{Co}/\text{IR-120}$ can catalyze the NaBH_4 hydrolysis reaction [15,16]. Therefore, hydrogen production rate up to about $110 \text{ mL}\cdot\text{min}^{-1}$ could be reached, along with a rise in temperature to 30°C (Fig. 6(a)).

During the second stage, hydrogen generation rate was slowed down to about $55 \text{ mL}\cdot\text{min}^{-1}$ at about 120 s from the onset of hydroly-

sis reaction and, then, gradually increased to about $250 \text{ mL}\cdot\text{min}^{-1}$. In this stage, the temperature of the reacting system roughly followed the trend in hydrogen production rate and increased gradually to ca. 50°C , owing to heat ejected from the exothermic hydrolysis reaction of NaBH_4 , shown as Eq. (1). At this stage, the catalytic hydrolysis reaction of NaBH_4 was mainly assisted with in situ as-reduced $\text{Co}/\text{IR-120}$ catalyst from the first stage. In the final stage, the reacting system ceased in hydrogen production. The hydrogen generation rate plunged quickly to zero in a few seconds. In general, a surge of hydrogen generation took place near the end of the second stage, when the reaction temperature reached above 40°C . As can be seen in Fig. 6(a), the hydrogen generation rate was elevated to a value as high as $250 \text{ mL}\cdot\text{min}^{-1}$. Hence, it was difficult to

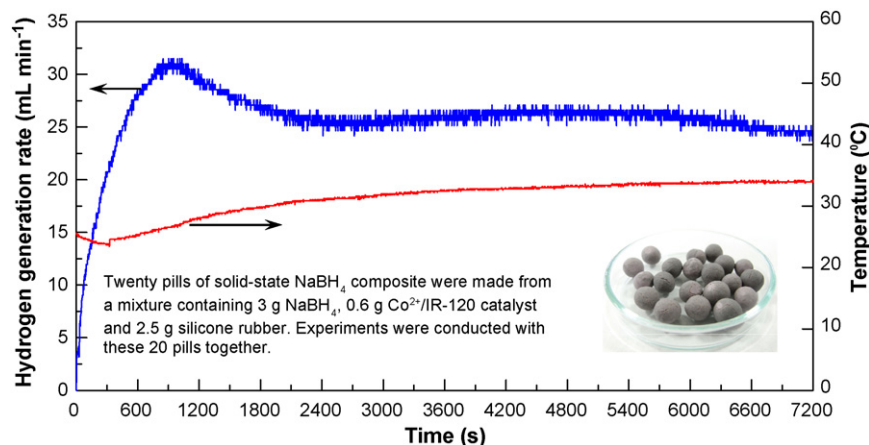


Fig. 7. Hydrogen generation rate for applying to a 2 W PEMFC stack.

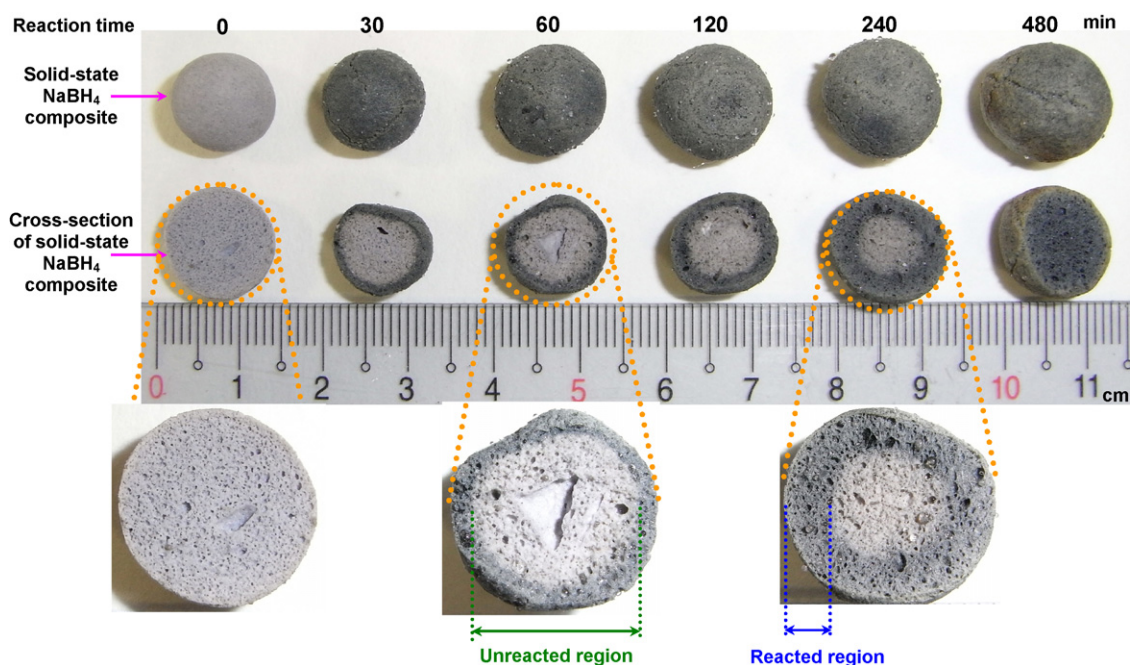


Fig. 8. Photographs of the cross-section of solid-state NaBH_4 composite for different reaction time.

achieve a constant and controllable supply of hydrogen gas due to the rise of temperature.

An attempt to resolve the issues on the surge of hydrogen generation was carried out by adding hydrophobic silicone rubber into the solid-state NaBH_4 composite to regulate the hydrogen production. The principle underlying it is that hydrophobic silicone could resist water wetting and imbibing into the pores inside the NaBH_4 -catalyst composite, and, thus, could deter the water diffusion rate and the hydrolysis reaction of NaBH_4 . Hence, a better controllability in, but also in the expense of, hydrogen evolution rate could be achieved. As a result, a better and satisfactory self-sustained hydrogen flow pattern was obtained (Fig. 6(b)). A smoother hydrogen production profile and less variation in reaction temperature were observed when the silicone was introduced to the solid-state NaBH_4 composite.

Furthermore, to feed the 2 W PEMFC stack, the rate of hydrogen supply should be higher than $24 \text{ mL} \cdot \text{min}^{-1}$ with a fuel stoichiometry of 1.5 [6]. The above hydrogen generation rate was obtained (Fig. 7) by adjusting the quantity of composition and shape of

solid-state NaBH_4 composite. As a result, the hydrogen generation rate and reaction temperature could be maintained at about $25 \text{ mL} \cdot \text{min}^{-1}$ and 33°C for at least 2 h. To investigate the mechanism leading to the smoother hydrogen production and reaction temperature, the cross-sectional morphology of the solid-state NaBH_4 composites harvested during different duration of NaBH_4 hydrolysis reaction was examined and compared. Fig. 8 displays the change in appearance of the cross-section of solid-state NaBH_4 composite garnered during different intervals of hydrolysis reaction. Before the onset of NaBH_4 hydrolysis reaction, the color of the solid-state NaBH_4 composite was pink owing to the presence of $\text{Co}^{2+}/\text{IR-120}$ catalyst. Obviously, after 30 min of reaction, the outer portion of the solid-state NaBH_4 composite, namely the depleting zone of NaBH_4 reactant, became black arising from the black $\text{Co}/\text{IR-120}$ catalyst formed in situ in the reacting system. In the meantime, water was continuously imbibed into the solid-state NaBH_4 composite for hydrogen evolution. After 480 min of reaction, almost all NaBH_4 in the solid composite was reacted out as the interior of the solid-state NaBH_4 composite turned black completely.

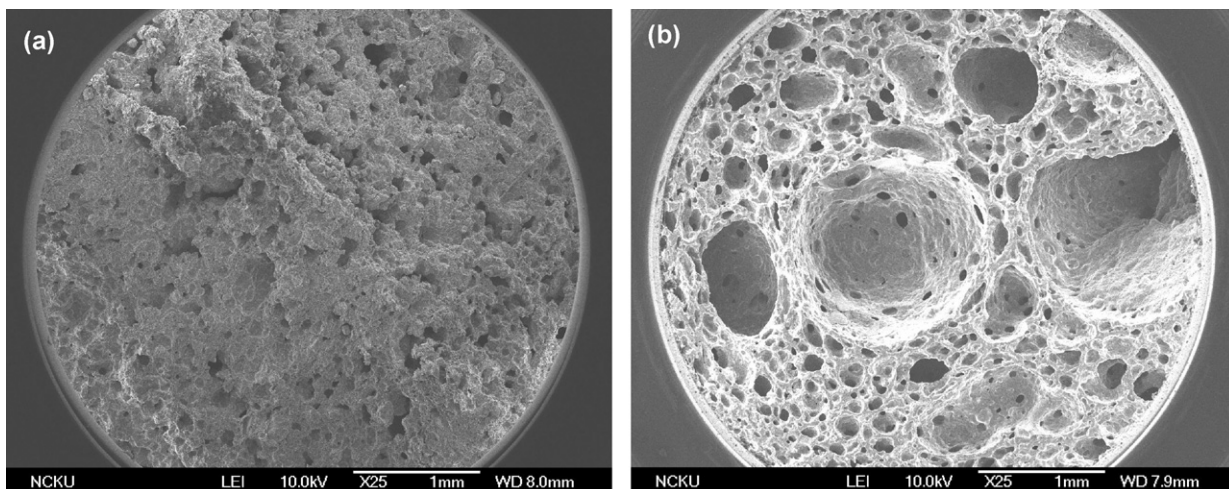


Fig. 9. SEM image of the cross-section of solid-state NaBH_4 composite for (a) before and (b) after reaction.

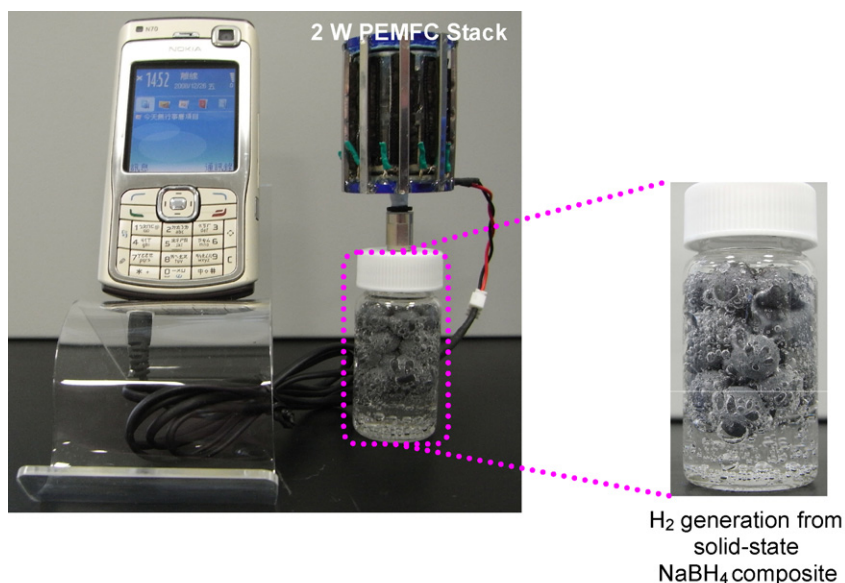


Fig. 10. A cellular phone powered by a miniaturized passive air-breathing PEMFC stack operating on hydrogen generated from the solid-state NaBH_4 composite.

This result certainly indicates that the silicone rubber used in this work can function as a stabilizer in the solid-state NaBH_4 composite because of its surface hydrophobicity to capably reduce the diffusion rate of water imbedded into NaBH_4 composite. Notably, water is also the required reactant in the hydrolysis reaction of NaBH_4 for hydrogen production. Presence of silicone rubber could restrain the water uptake by the solid NaBH_4 composite, so that the hydrogen generation rate could be easily regulated. Comparison of interiors and internal structures of solid-state NaBH_4 before and after reaction was made by SEM observation on their cross-sectional morphology (Fig. 9). After 480 min of hydrolysis reaction, many interconnected pores and voids appear inside the solid-state NaBH_4 composite, compared to the very dense interiors of NaBH_4 composite before reaction. This result further confirms that water can be really diffused into the solid-state NaBH_4 composite and react with NaBH_4 over active sites on $\text{Co}^{2+}/\text{IR-120}$ surface. Still, there are many factors influencing the hydrogen evolution and content of hydrogen storage yet to be investigated. For example, effects of catalyst and NaBH_4 loadings as well as amount of silicone rubber used in the solid-state NaBH_4 composite on the hydrogen generation rate will be studied in details in our future work.

3.3. Hydrogen supply to a 2 W PEMFC stack

Based on the results presented in Fig. 7, a hydrogen supply system was designed and practically applied to a 2 W PEMFC stack. The nine-cell PEMFC stack in a miniaturized passive air-breathing type was fabricated as a power source for a cellular phone. No temperature control was adopted on this hydrogen generator. Instead, the hydrogen generator was exposed to ambient conditions. The hydrogen generation rate was kept nearly constant at about $25 \text{ mL} \cdot \text{min}^{-1}$ for at least 2 h as shown in Fig. 7. As presented in Fig. 10, the PEMFC stack was successfully operated on hydrogen supplied from the solid-state NaBH_4 composite. This PEMFC also powered a cellular phone with power consumption of 2 W. The 2 W prototype for 2 h of operation (with 6.1 g of the solid-state NaBH_4 composite fuel) delivers at least 4 Wh of energy. The 4 Wh of energy can, in turn, fulfill recharging a 3.7 V/1100 mAh Li-ion battery in 2 h.

4. Conclusion

We have successfully developed a 2 W-scale hydrogen generator that could produce ultrapure H_2 gas from a novel solid-state NaBH_4 composite. This novel solid-state NaBH_4 composite provides con-

sumers not only with better portability but also more convenience in use, compared to the conventionally aqueous NaBH_4 reacting system. The hydrogen generation rate from this solid-state reacting system of NaBH_4 could be kept constant at about 25 mL min^{-1} for at least 2 h without any temperature control system applied to the system. Furthermore, this produced hydrogen does not need any further purification and could be directly fed to a passive air-breathing 2 W PEMFC stack. As estimated, at least 4 Wh of energy is produced from this PEMFC stack fed directly with the as-produced hydrogen.

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