

Short communication

Aluminum and aluminum alloys as sources of hydrogen for fuel cell applications[☆]

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Abstract

Production of hydrogen using aluminum and aluminum alloys with aqueous alkaline solutions is studied. This process is based on aluminum corrosion, consuming only water and aluminum which are cheaper raw materials than other compounds used for in situ hydrogen generation, such as chemical hydrides. In principle, this method does not consume alkali because the aluminate salts produced in the hydrogen generation undergo a decomposition reaction that regenerates the alkali. As a consequence, this process could be a feasible alternative for hydrogen production to supply fuel cells. Preliminary results showed that an increase of base concentration and working solution temperature produced an increase of hydrogen production rate using pure aluminum. Furthermore, an improvement of hydrogen production rates and yields was observed varying aluminum alloys composition and increasing their reactive surface, with interesting results for Al/Si and Al/Co alloys. The development of this idea could improve yields and reduce costs in power units based on fuel cells which use hydrides as raw material for hydrogen production.

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1. Introduction

The development of H₂ fuel cells for vehicles, stationary and mobile applications has been an active area of research during the past 30 years [1]. Nowadays, this research is also important to reduce greenhouse gas emissions from the burning of fossil fuels. Several automotive companies have come up with innovative design features in their prototypes [2]. Although hydrogen is an attractive fuel alternative for the future, attractive methods for hydrogen production and storage must be employed in order to maintain its positive profile. There are different mature methods for hydrogen production [3]. These methods are basically based in fossil fuels, including more than 90% of the industrial hydrogen production [4]. Fifty-five to 60% of the hydrogen being produced in the world today is made by steam reforming of natural gas. Hydrogen is also produced by water electrolysis, its

most important industrial process not based on fossil fuels at the moment, but the costs of CO₂-free hydrogen production are at least €20 GJ⁻¹, which does not compare with the present prices of oil or natural gas, about €5 GJ⁻¹ [5]. Therefore, the development of new technologies for hydrogen production not based on fossil fuels is becoming increasingly important to provide a clean fuel over the 21st century [6,7].

Nowadays, there are no practical ways of storing large amounts of hydrogen once the criteria of capacity, safety, and refuelling are considered [8]. For instance, hydrogen containing vessels, whether they are high pressure gas-containing cylinders or liquid containing vessels, have significant and lingering safety problems. Some new technologies, such as hydrogen storage on metals, graphitic adsorbents or carbon nano-tubes, are being explored but they are far away from practical applications [9,10].

Generation of hydrogen for fuel cell applications by reaction of chemical hydrides with aqueous solutions reduces storage weight and/or volume over high pressure or cryogenic storage [11]. However, hydrogen production from hydrides also has some disadvantages: hydrides are expensive raw materials considering current hydrogen prices and most of them are unstable and sensitive to air moisture. On the other hand,

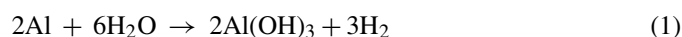
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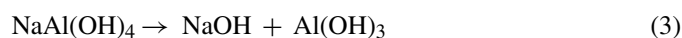
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hydrogen desorption from light metal hydrides, such as alanates, is an endothermic process that requires $T > 373$ K [11,12]. So, advances in new types of safe, cheap and renewable hydrogen sources are very important to establish fuel cells as a real alternative to internal combustion engines powered by fossil fuels.

In the laboratory, hydrogen can also be generated by the reaction of aluminum with acids or bases. Aluminum has attracted attention as a battery anode because its high theoretical ampere-hour capacity, voltage and specific energy [13]. These kinds of batteries present a parasitic hydrogen generating reaction due to aluminum corrosion in aqueous media:



Although this parasitic hydrogen generation is an undesirable reaction in aluminum/air batteries, aluminum corrosion in aqueous alkaline solutions provides a cheaper source of hydrogen than hydrolysis of hydrides. In addition, hydrogen production from aluminum can be achieved under mild conditions of temperature and pressure. The reactions of aluminum with sodium hydroxide in aqueous solution to produce hydrogen have been already studied [14–17]:



So, sodium hydroxide consumed in the hydrogen-generating reaction (2) can be regenerated in reaction (3) and the overall process is (1). Thus, only aluminum and water are the consumed raw materials to produce hydrogen. Aluminum can be regenerated from aluminum hydroxide, by two processes developed in the late 1800s: the Bayer process [18] which produces pure alumina from bauxite ore, and the Hall–Héroult process [19] which produces aluminum from alumina. The major hurdle to the production of hydrogen via this corrosion reaction is that the aluminum surface is easily passivated when Al is recovered with an $\text{Al}(\text{OH})_3$ layer. This passivation can be reduced by controlling several experimental parameters such as temperature, alkali concentration, aluminum morphology and aluminum raw material used to produce hydrogen [15]. Contrary to aluminum/air batteries, in the present process there is no need to use pure aluminum [13]. In fact, the use of commercially available aluminum or aluminum alloys could reduce hydrogen production costs. Aluminum alloys with particular metals, such as gallium, tin, rhenium, indium, lead, bismuth, magnesium or calcium, have higher reactivity than aluminum metal, but they are not easily available. In this sense, there are some works where aluminum has been tentatively activated by the preparation of new aluminum alloys and composites [20,21], but these alloys are unstable and their storage at ambient temperature leads to their decomposition. Conversely, hydrogen production using commercially available aluminum alloys in mild conditions of temperature and pressure is an issue that has not been deeply studied [22].

Furthermore, the behavior of other hydroxides different than NaOH as corrosion inducers has been scarcely studied so far [23]. The aim of this work is the generation of hydrogen using commercial aluminum alloys, which are stable at normal con-

ditions, trying to enhance aluminum reactivity and prevent the aluminum surface passivation. Therefore, the objective of this study is to demonstrate the feasibility of producing hydrogen for fuel cells applications from commercial aluminum and aluminum alloys (shortened AAA from now on) and aqueous solutions with different bases (e.g. KOH, $\text{Ca}(\text{OH})_2$).

2. Experimental

2.1. Chemicals

Calcium hydroxide powder, sodium hydroxide pellets and potassium hydroxide pellets were supplied by Panreac and were used as received. Deionized water was used to prepare all the aqueous solutions. Al/Si alloy powder (88:12 wt%, –325 mesh, 99% purity) and Al/Co alloy powder (69:31 wt%, –100 mesh, 99% purity) were supplied by Alfa Aesar. Al/Mg alloy powder (92.1:7.9 wt%, 63 μm maximum particle size) was supplied by Goodfellow. Al flakes (1.0 mm particle size, 99.99% purity) were supplied by Aldrich. Al powder (–325 mesh, 99.7% purity) was supplied by Strem Chemicals. Al foil (15 μm thickness) was supplied by Albal. The AAA used in this study are reported in Table 1.

2.2. Apparatus, materials and measurements

Preliminary tests of AAA corrosion have been performed in a Pyrex glass beaker containing 75 cm^3 of KOH or NaOH aqueous solutions at different concentrations and temperatures.

Table 1
Aluminum and aluminum alloys composition and morphology

AAA number	Composition (wt%)	Morphology	Supplier
#1	Al 69/Co 31	Powder	Alfa Aesar
#2	Al 95.6/Li 1.9/Cu 1.5 Mg 1	Rod	Goodfellow
#3	Al 88/Si 12	Powder	Alfa Aesar
#4	Al 92.1/Mg 7.9	Powder	Goodfellow
#5	Al 94.3/Si 0.190/Fe 0.190/Cu 0.068/Mn 0.484/Mg 4.530/Zn 0.020/Ti 0.021/Cr 0.235	Bar	Alu-Stock
#6	Al 100	Foil	Albal
#7	Al 100	Powder	Strem chemicals
#8	Al 96.45/Mg 2.6/Mn 0.8/Cr 0.15	Tube	Goodfellow
#9	Al 97.93/Mg 1.0/Si 0.6/Cu 0.27/Cr 0.2	Plate	Alfa Aesar
#10	Al 100	Flakes	Aldrich
#11	Al 98.6/Mn 1.2/Cu 0.12	Tube	Alfa Aesar
#12	Al 88.94/Si 0.110/Fe 0.240/Cu 1.770/Mn 0.246/Mg 2.490/Zn 5.960/Ti 0.021/Pb 0.027/Cr 0.191	Bar	Alu-Stock
#13	Al 97.5/Si 1.0/Mg 0.8/Mn 0.7	Rod	Goodfellow
#14	Al 98/Mg 1/Si 1	Tube	Goodfellow
#15	Al 50/Ni 50	Powder	Goodfellow
#16	Mg 96/Al 3/Zn 1	Foil	Goodfellow

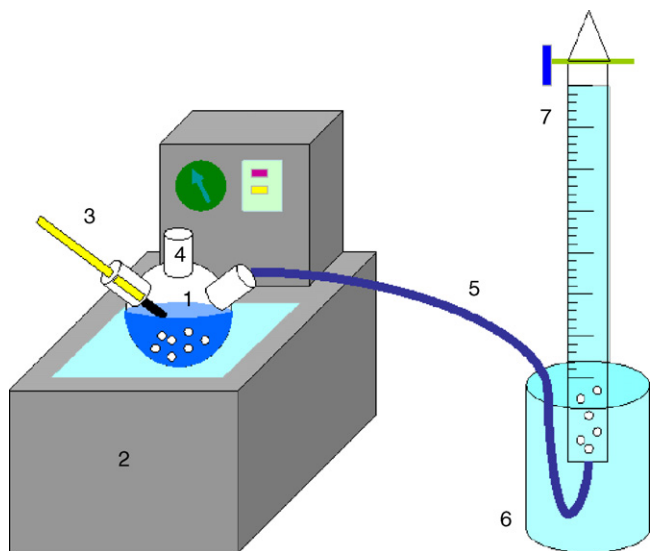


Fig. 1. Scheme of the experimental set up. (1) Pyrex glass reactor; (2) thermostatic water bath; (3) thermometer; (4) reagents input; (5) Tygon tube; (6) beaker filled with water at room temperature; (7) water filled burette.

A piece of 0.1 g of aluminum foil was crumpled until forming a small ball. In these preliminary experiments, 0.1 g Al was added into the alkaline solution and aluminum consumption time was measured. Solids produced from aluminum corrosion were filtered using a vacuum pump and a funnel provided with a filter plate. Precipitate was dried in an oven at 348 K during 1 day, and it was analyzed using X-ray diffraction and attenuated total reflectance-Fourier transform infra red spectroscopy (ATR-FTIR) in order to determine its composition.

The equipment used to quantify hydrogen production rates and yields is illustrated in Fig. 1. Reagents were added into a 100 cm³ Pyrex glass reactor containing 75 cm³ of working solution. All the experiments carried out with the aim of comparing AAA initial hydrogen production rates were performed at room temperature (298 K), with no external heating. Contrary, the reactor was heated with a water bath to maintain a constant temperature of 348 K in all the experiments carried out to study the effect of base concentration for Al/Si alloy corrosion. Hydrogen production reaction (2) started when aluminum or aluminum alloy came into contact with the aqueous solution. Hydrogen produced by aluminum corrosion emerged from the reactor through a Tygon tube of 40 cm length and 3 mm internal diameter, it was passed through a water bath at ambient temperature in order to condense the water vapor, and hydrogen was collected in an inverted burette to measure the quantity of hydrogen produced.

3. Results and discussion

According to reaction (1), AAA in basic aqueous solutions produce heat as well as hydrogen, consuming only water. In the following sections, the results obtained on the effects of several parameters are reported.

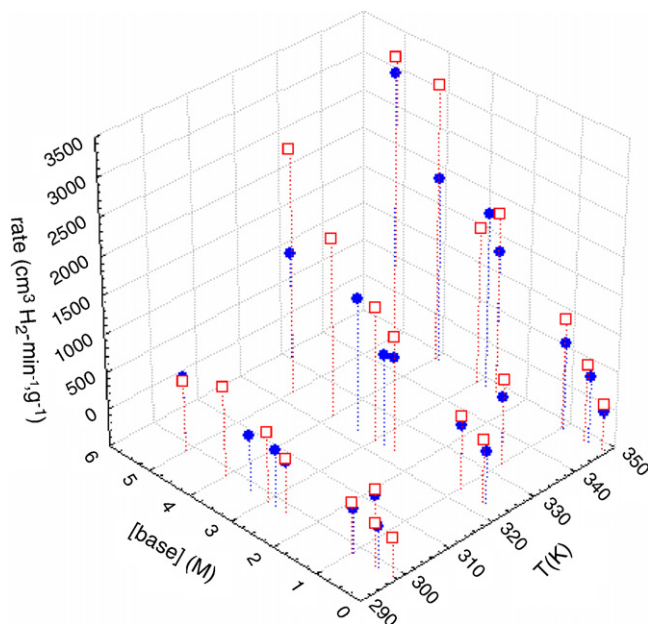


Fig. 2. Comparison of hydrogen production rates obtained consuming 0.1 g Al in NaOH (□) and KOH (●) at different concentrations (M) and temperatures (K).

3.1. Preliminary experiments with KOH and NaOH

The obtained results from preliminary experiments carried out using 0.1 g of Al foil in KOH solutions are shown in Fig. 2. As expected, an increase of KOH concentration causes an increase of Al corrosion rate and, consequently, produces an increase of hydrogen production rate. This rate is also increased when temperature is higher. The white solid produced was filtered, dried and analyzed by X-ray diffraction (Fig. 3). These results confirm that solid formed is basically composed by Al(OH)₃ (Gibbsite) and KHCO₃ (Kalicinite) both identified by X-ray diffraction data base. The ATR-FTIR technique was used to confirm the characteristic OH stretching vibrations in Gibbsite compounds (Fig. 4). The performed spectra present absorption bands at 3400–3700 cm⁻¹ attributed to ν(OH) stretch and bands

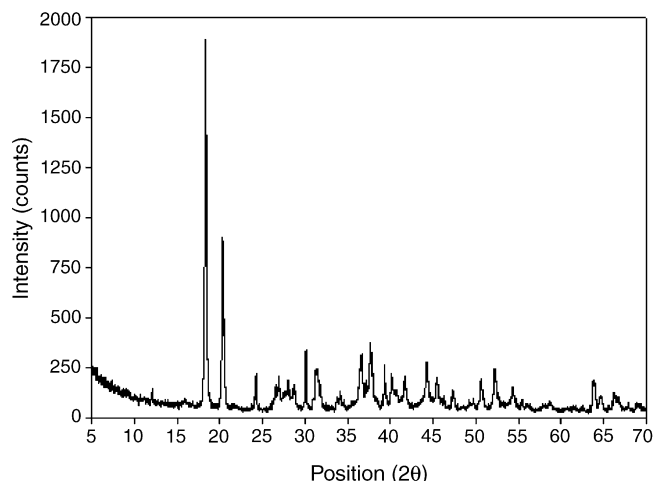


Fig. 3. X-ray diffraction corresponding to solid produced with aluminum corrosion in KOH 5 M.

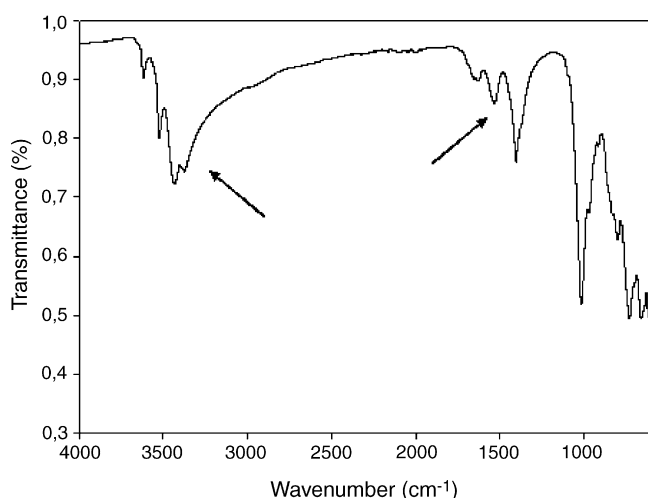


Fig. 4. ATR spectra corresponding to solid produced with aluminum corrosion in KOH 5 M.

at $1600\text{--}1400\text{ cm}^{-1}$ attributed to $\nu(\text{CO})$ stretching in KHCO_3 . So potassium hydrogen carbonate was formed due to CO_2 reaction with alkaline solutions in contact with air and caused a diminution of free hydroxyl concentration. That fact proves that although aluminum corrosion does not consume the alkali, its concentration is reduced along time. So, in a practical situation contact with air should be avoided.

3.1.1. Experimental variables evaluation

In order to compare aluminum corrosion produced either by KOH or NaOH, the same experiments were carried out using both bases. All the obtained results are plotted in Fig. 2. Good hydrogen production rates were obtained using both bases, but aluminum corrosion was always faster using NaOH instead of KOH at the same concentration and temperature. Maximum rates were reached using NaOH 5 M at 348 K, producing hydrogen at rate of $3.23\text{ dm}^3\text{ H}_2\text{ min}^{-1}\text{ g}^{-1}\text{ Al}$. Taking into account that 1 dm^3 of hydrogen at standard conditions is equivalent to an energy content of approximately 3 Wh (although due to the limited efficiency of actual fuel cells the practical value is assumed to be about 1 Wh [24]), the hydrogen production rates obtained by aluminum corrosion with KOH and NaOH seem to be high enough to power H_2 -based fuel cells.

The experimental aluminum corrosion data were analyzed using multivariable data analysis and modeling software tools (Umetrics Modde 5.0) with the aim of set a model for aluminum corrosion and quantify the influence of each parameter in the reduction of aluminum corrosion time. The coefficients obtained for each experimental variable and its interactions are shown in Table 2. Temperature and base concentration are critical parameters in order to increase hydrogen production rate. Additionally, a synergistic effect was found when increasing temperature and base concentration at the same time. The obtained NaOH response surface confirms previous results reported from reaction of aluminum with sodium hydroxide in aqueous solution to produce hydrogen [14–17]. Comparing our NaOH results with those of aluminum from soft drink cans [16], treated in very similar conditions, the cans corrosion in NaOH 1.12 M at

Table 2

Influence of experimental parameters on Al corrosion time, calculated with Umetrics Modde 5.0

Parameter	NaOH coefficient	KOH coefficient
Concentration	959 ± 99	800 ± 83
Temperature	789 ± 80	664 ± 70
Concentration \times temperature	673 ± 109	614 ± 99
Temperature \times temperature	18 ± 142	101 ± 119
Concentration \times concentration	151 ± 185	2 ± 138
R2 adjusted	0.980	0.977

298 K had an initial rate of $12.5\text{ cm}^3\text{ H}_2\text{ min}^{-1}\text{ g}^{-1}$, and using aluminum foil in NaOH 1 M at 298 K the initial rate was about $150\text{ cm}^3\text{ H}_2\text{ min}^{-1}\text{ g}^{-1}$, so by using commercial aluminum foil we have increased this initial rate an order of magnitude. This rate increase can be attributed to the different thickness and quality of raw material between aluminum cans and aluminum foil.

3.2. Influence of composition and morphology of aluminum alloys

A systematic study was performed in order to determine the effect of composition and morphology of AAA on hydrogen production rate. All the experiments were carried out in KOH 1 M at 298 K, since aluminum corrosion with NaOH had been already studied [14–17]. Results obtained are reported in Table 3. Initial rates were calculated and normalized per weight or specific surface of the studied alloy. A typical hydrogen production curve (H_2 produced versus time) is shown in Fig. 5 for an Al/Co alloy (#1). As can be observed, hydrogen production rate is higher at the beginning of the experiment but it decelerates as aluminum is being consumed.

From results reported in Table 3, it can be concluded that the composition of the five first alloys in the table present initial

Table 3

Hydrogen production initial rates for all the studied AAA, normalized per surface ($v_{0,s}$) or per weight ($v_{0,m}$)

AAA number	Specific surface ($\text{cm}^2\text{ g}^{-1}$ alloy)	$v_{0,s}$ ($\text{cm}^3\text{ H}_2\text{ min}^{-1}\text{ cm}^{-2}$)	$v_{0,m}$ ($\text{cm}^3\text{ H}_2\text{ min}^{-1}\text{ g}^{-1}$)
#1	149	0.49	73
#2	9.0	0.48	4.3
#3	505	0.43	216
#4	353	0.33	117
#5	3.5	0.30	1.0
#6	494	0.28	139
#7	505	0.27	138
#8	31.2	0.26	8.2
#9	3.0	0.24	0.7
#10	112	0.23	26
#11	23.2	0.23	5.3
#12	2.8	0.21	0.6
#13	8.6	0.19	1.6
#14	13.9	0.18	2.5
#15	148	0.09	14
#16	Not determined	0.00	0.0

Rates are reported in diminishing $v_{0,s}$ order.

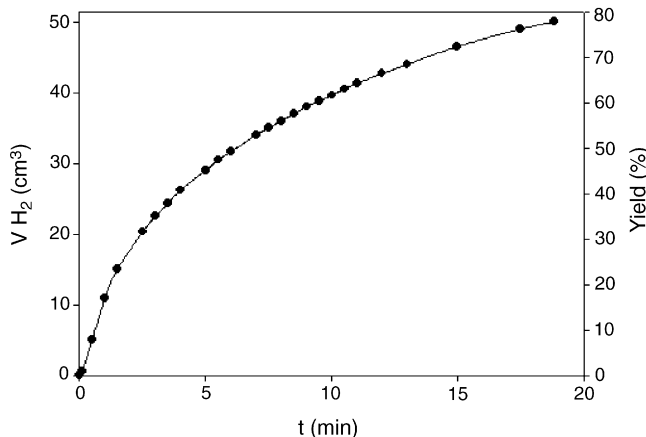


Fig. 5. Hydrogen production curve obtained for 0.07 g Al/Co in KOH 1 M at 298 K.

hydrogen production rates (normalized per surface) higher than pure aluminum (#6, #7 and #10). Al/Si alloy (#3) also shows the highest initial rate normalized per alloy weight. All the commercial AAA tested are stable under standard conditions of temperature and pressure, and, in addition to pure Al, the composition of first five alloys are suitable to produce hydrogen with good yields and rates.

It is important to note that morphology of AAA is a key parameter which has an effect on Al corrosion rate. For that reason, it is necessary to normalize all the hydrogen production rates per specific surface, in order to study and compare rate variations due to AAA different composition, independently of its morphology. An example of this can be observed comparing rate values for aluminum foil (#6), powder (#7) and flakes (#10) (see Table 3): the difference observed between hydrogen production rates normalized per weight of aluminum flakes respect to other aluminum raw materials, can be attributed to the lower specific surface of aluminum flakes, which suffers more passivation than powder or foil. Those differences are visibly smaller in rate values normalized per surface.

The obtained results can be compared with those found in the literature for similar systems. Both aluminum–carbon synthetic composites [20] and rapidly quenched Ni–Al alloys [22] give hydrogen production rates normalized per weight that are comparable to the obtained results found for Al/Si alloy (#3) and only slightly higher than that obtained for pure aluminum (#6 and #7). Alloys containing aluminum, gallium, indium, tin, and zinc among other elements do give higher rates but these synthetic materials are unstable when stored in air at room temperature [21]. Therefore, some of the studied AAA can be considered as an interesting alternative option for hydrogen production.

Finally, several experiments were carried out with the aim of study the effect of base concentration in Al/Si alloy corrosion. This alloy was chosen due to its high hydrogen production rates (see Table 3). In addition, part of silicon could react in basic media generating an extra amount of hydrogen [25]. Fig. 6 shows hydrogen production curves with 0.210 g Al/Si at different calcium hydroxide concentrations. Calcium hydroxide was chosen because it is an economical base with a safer pH even at saturation level. In said figure, it can be observed that initial

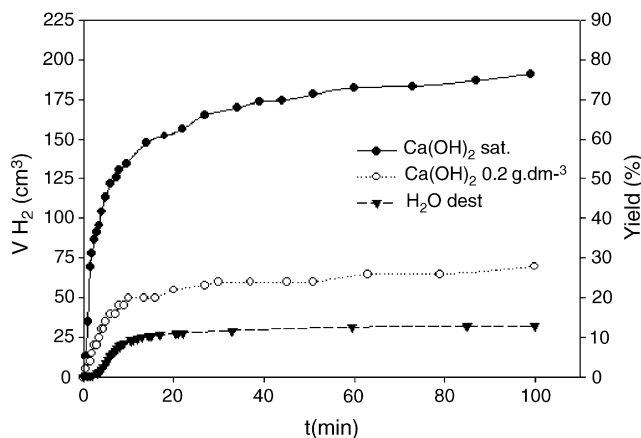


Fig. 6. Hydrogen production curves with 0.210 g Al/Si alloy at 348 K in H₂O (▼), Ca(OH)₂ 0.2 g dm⁻³ (○) and saturated Ca(OH)₂ (●).

hydrogen production rate is higher when the base concentration is increased in working solution. Concretely, for a saturated Ca(OH)₂ with an initial pH of 12.6 a hydrogen production rate of 146 cm³ H₂ min⁻¹ g was obtained. At the end of the experiment, pH was 11.2 and hydrogen production yield reached a 76% (after 100 min). By using Ca(OH)₂ 0.2 g dm⁻³, hydrogen production rate obtained was 33 cm³ H₂ min⁻¹ g⁻¹, and yield reached a value of 28% (100 min). Finally, another experiment was performed using pure water, in order to study the hydrogen production efficiency of reaction (1) for Al/Si alloy in absence of basic compounds in the working solution. The obtained results showed that Al/Si alloy can even react with pure water to produce hydrogen, although the reaction efficiency observed had a low value, reaching a hydrogen production yield of 12.5% (100 min) and an initial rate of 19 cm³ H₂ min⁻¹ g⁻¹. Similar results of efficiency were obtained for Al/Co alloy using pure water as working solution.

4. Conclusions

Considering the experiments reported here, the technical feasibility of the described process has been demonstrated. Corrosion of commercial AAA in aqueous solutions could reduce hydrogen production costs for several applications which use chemical hydrides as raw materials to generate hydrogen in situ. Preliminary experiments with KOH and NaOH showed a synergistic effect of base concentration and temperature to increase hydrogen production rate. Besides, it has been observed that carbon dioxide from air diminishes free hydroxide concentration in aqueous solutions, so that the working solution should not be in contact with air in order to maintain a good efficiency of hydrogen production process.

Concretely, the results obtained for Al/Si and Al/Co alloys are potentially applicable in devices that can provide a source of CO₂ free hydrogen at an adaptable rate to fuel cell requirements. Also, it has been observed experimentally that hydrogen production rate can be regulated varying parameters such as the base concentration used, the temperature or the morphology and composition of the AAA.

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