



Effect of addition of water-soluble salts on the hydrogen generation of aluminum in reaction with hot water



S.S. Razavi-Tousi^{*}, J.A. Szpunar

Department of Mechanical Engineering, University of Saskatchewan, S7N 5A9 Saskatoon, Saskatchewan, Canada

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ABSTRACT

Aluminum powder was ball milled for different durations of time with different weight percentages of water-soluble salts (NaCl and KCl). The hydrogen generation of each mixture in reaction with hot water was measured. A scanning electron microscope (SEM) as well as energy-dispersive spectroscopy (EDS) were used to investigate the morphology, surfaces and cross sections of the produced particles. The results show that the presence of salts in the microstructure of the aluminum considerably increases the hydrogen generation rate. At shorter milling times, the salt covers the aluminum particles and becomes embedded in layers within the aluminum matrix. At higher milling durations, salt and aluminum phases form composite particles. A higher percentage of the second phase significantly decreases the milling time needed for activation of the aluminum particles. Based on the EDS results from cross sections of the milled particles, a mechanism for improvement of the hydrogen generation rate in the presence of salts is suggested.

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1. Introduction

Researchers have adopted different approaches to improving the kinetics of the reaction of aluminum with water for the purpose of hydrogen generation. Adding low melting point metals to aluminum, using basic solutions, applying supercritical water conditions, and ball milling in the presence of oxides or water-soluble salts are among the techniques used to improve the hydrogen generation rate [1–18]. Although most of these methods have been effective in improving the reaction, some reservations remain about their potential for large-scale application. For example, low melting point metals are expensive and most of them are toxic, the supercritical condition is not hardly applicable for onboard hydrogen generation, and the use of basic solutions raises safety and environmental concerns. Among different methods, ball milling of aluminum powder in the presence of water-soluble salts is considered effective and safe [9,19–25]. Water-soluble salts (NaCl, KCl, etc.) are neither expensive nor toxic and their presence significantly shortens the completion time of the reaction.

The effects of water-soluble salts during milling on the reaction

kinetics and consequently on hydrogen generation have been explained in two ways: Firstly, some researchers point out that the reaction is promoted because of the Cl^- ions released by dissolving salts in water [3]. These Cl^- ions can deteriorate the protectiveness of the hydroxide layer on the surface of the particles [26–28], and thus the aluminum substrate is corroded more effectively. Secondly, the presence of salt particles can modify the structure of aluminum particles. Alinejad et al. consider that salt particles modify the structure of aluminum particles by providing more surfaces for the reaction. They suggest a schematic model to illustrate how the presence of salt particles promotes the reaction [23]. In this model, they assume that salt particles cover the aluminum particles during ball milling. As the mixture is exposed to water, salt particles are dissolved and a fresh surface of aluminum is exposed to water.

In spite of the number of works that have used water-soluble salts to activate aluminum, no study has attempted to distinguish whether their addition affects the chemical reaction or the structure of particles. The structural and chemical effects can be separated by introducing a particular amount of a salt during ball milling or adding it to the hot water during the reaction and then measuring the hydrogen production rate for either case. In this study, the hydrogen generation of pure aluminum was firstly compared with that of an aluminum-salt mixture. Then, the microstructure of the aluminum-salt particles was studied after

^{*} Corresponding author.

E-mail addresses: ser105@mail.usask.ca, s.razavitousi@gmail.com (S.S. Razavi-Tousi).

different milling times and the results correlated to hydrogen generation. Finally, the effects of the different salt types (NaCl or KCl) were examined.

2. Experimental procedure

2.1. Ball milling

Alfa Aesar Company provided aluminum powder of 99.8% purity (MFCD00134029, average particle size of 227 μm). After mixing 50 wt% of sodium chloride (purity: +99%, average particle size of 281 μm) with the aluminum powder, the mixture was ball milled. High-energy ball milling was done in a planetary ball mill (Torrey Hills- ND2L) with stainless steel cups (285 ml capacity) and balls (28 of 16 mm and 6 of 18 mm diameter) in an argon atmosphere. The ball to powder ratio was 30:1 and the mill speed was maintained at 200 RPM for durations of 0.25, 0.5, 1, 2, 4, 7, 11 and 19 h. In order to evaluate the effect of the weight percentage and type of salts, mixtures of 25 wt% NaCl- 75 wt% Al, 75 wt% NaCl- 25 wt% Al and 50 wt% KCl- 50 wt% Al were also ball milled for durations of 2, 7 and 19 h. The KCl powder, with a commercial purity, was refined from Potash Corporation of Saskatchewan products.

2.2. Hydrogen measurement

The details of the hydrogen measurement procedure are similar to those described in Ref. [18]. We added powder mixtures with the equivalent of one g of aluminum in to an Erlenmeyer flask containing 200 ml of distilled water at 80 ± 3 °C with a constant stirring rate of 120 rpm. The produced hydrogen gas passed through a desiccant (CoCl_2) to absorb moisture, and then an ADM2000 flowmeter to measure its flow with an accuracy of 0.1 ml/min. The flowmeter was connected to a computer running ADM Trend software to acquire the data.

A baseline curve was obtained by measuring the flow from 200 ml of distilled water at 80 °C with a constant stirring rate of 120 rpm with no powder added. This was then subtracted from the data obtained from the reactions to ensure that the measurements did not include any contribution from water moisture or expansion of the air in the flask during heating.

2.3. Scanning electron microscopy

A Hitachi SU6600 field emission scanning electron microscope (SEM) was employed to examine the surfaces and cross sections of the powders. A small amount of each powder was added to a conductive carbon resin powder, mixed and mounted. The mounted samples were polished using abrasive grinding papers from 600 to 2000 grit followed by additional grinding with diamond pastes of 3 and 0.04 μm . Particle sizes of the powders were calculated using software written in Visual Basic using several SEM images. The weighted average (Eq. (1)) was calculated and reported as the average particle size:

$$D = \frac{\sum_i n_i D_i}{\sum_i n_i} \quad (1)$$

where D_i is the diameter of the particle i , n_i is the number of particles with size D_i , and D is the weighted average.

3. Results and discussion

3.1. Effect of milling time and presence of salt

3.1.1. Hydrogen generation

Fig. 1(a) shows the effect of milling time on the hydrogen generation of aluminum-salt mixtures through their reaction with hot water. Hydrogen generation was not observed for the mixture of aluminum-salt that was not ball milled, thus it is not reported in Fig. 1(a). Milling the mixture for durations as short as 15 or 30 min resulted in measurable hydrogen generation. Increasing milling time from 1 h up to 7 h significantly enhanced the hydrogen generation rate. However, further milling from 7 h up to 19 h did not appreciably increase the amount of generated hydrogen as the amount for the 7 h sample was already close to the theoretical limit.

Fig. 1(b) presents the hydrogen generation results from the pure aluminum powder milled for the same durations, which are published in Ref. [18]. Since the starting material, milling, and the hydrogen measurement conditions were the same for both experiments, the comparison of Fig. 1(a) and (b) illustrates the effect of the presence of sodium chloride on the hydrogen generation rate. Clearly, the aluminum milled in the presence of salt reacted significantly faster than the pure aluminum; the maximum amount of produced hydrogen in one hour increased from 230 ml to 1200 ml.

Nevertheless, it is significant that the presence of salt without milling was ineffective, as neither pure aluminum nor an aluminum-salt mixture could generate hydrogen in the as-received

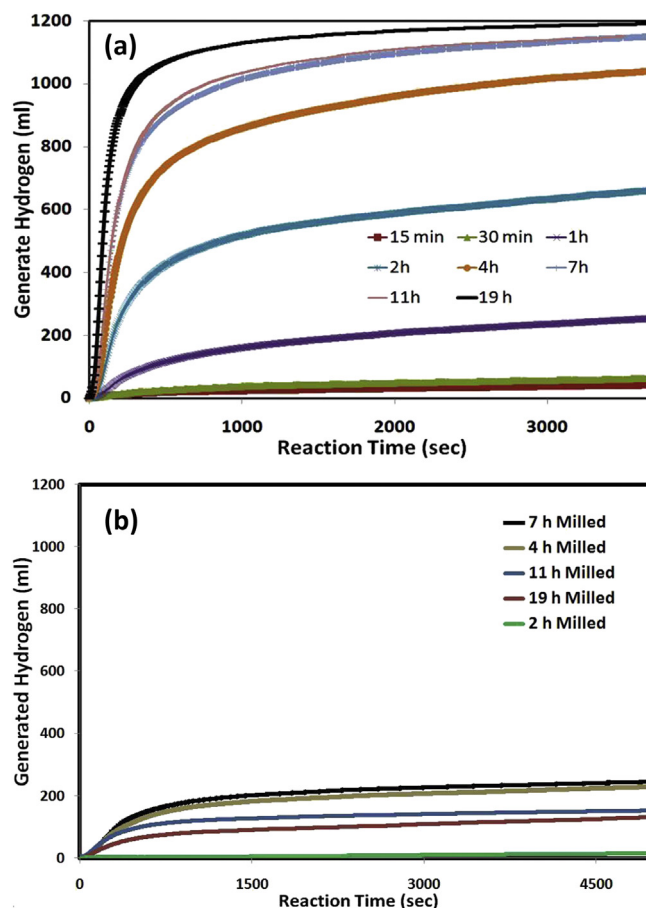


Fig. 1. Effect of milling time on hydrogen generation of (a) the aluminum-salt mixture, and (b) pure aluminum during the reaction with hot water.

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