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RESEARCH ARTICLE

Comparison of pure-hydrogen production performances of blast furnace slag, and metal powders in sodium borohydride hydrolysis reaction

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Abstract: In this study, hydrolysis reaction performances of raw BFS powder and metal powders (which are ingredients of BFS) that are used as a catalyst are compared. Hydrogen generation by hydrolysis reaction of the Al and Fe_2O_3 Nano&Granule powders with sodium borohydride ($NaBH_4$) addition in water was studied by using different catalysts amounts at reaction vessels. The measured values of reaction temperatures and hydrogen flow rates were measured by using high-precision equipment. As a result of the obtained data, it was determined that Fe_2O_3 and Al catalysts have advantages over hydrogen production rate and fuel conversion, also, these experiments show a very high success in different parameters, and create promising effects in the reactions. Among the Al catalyst samples, the highest efficiency performances are achieved with Al Nanocatalyst samples at 85.31 °C preheat with an instantaneous hydrogen generation rate of approximately 11.226 L / min for 33 minutes. Among the Fe_2O_3 catalyst samples, the highest efficiency performances are achieved with Fe_2O_3 Nanocatalyst samples at 50 °C preheat with an instantaneous hydrogen generation rate of approximately 29.91 L / min for 12 minutes.

Keywords: Metal powders, $NaBH_4$, Hydrogen production, Nano Material, Blast Furnace Slag

1. Introduction

Today most of our energy needs are provided by fossil fuels. Fossil fuel sources are limited as they are non-renewable energy sources, also, are rapidly being depleted with the increase in industrialization. Although fossil fuels seem useful to meet energy needs, serious damage is caused to the environment during the extraction and transportation process, thus, creating very harmful environmental effects of the fossil fuel resources. Since the main component of fossil fuels is carbon, they emit greenhouse gases (CO_2, CH_4, N_2O) that are harmful to the atmosphere when they are combusted. Due to this, air pollution problems arise [1]. Due to the decrease in the reserves of fossil energy resources and the damage they cause to the environment, a search for new energy resources has been started. As a result of this search, renewable energy resources have increased interest in them. These resources are the most ideal energy sources for continuous energy needs and clean energy [2].

Opportunities of rapid technological change such as renewable energy sources, which are more environmentally friendly and more efficient, will come into play for energy production in a sustainable future. One of these sources is hydrogen, which can be used as an energy raw material in today and the future. As one of the renewable energy sources, hydrogen is a great hope to reduce the ever-growing energy need and environmental pollution. Although hydrogen gas has many advantages, its biggest disadvantage is storage difficulty. Due to its low density, it must be stored in high-pressure tanks to provide sufficient fuel to the systems, thus increasing the storage cost [3]. Hydrides have been preferred in recent years due to their high hydrogen volume and reliability. Many types of hydrides such as metal hydrides and chemical hydrides are used in obtaining hydrogen. Examples of the most popular chemical hydrides are $NaBH_4$, NH_3, and MgH_2. Borohydrides such as $NaBH_4$ are preferred due to their environmental friendliness, non-flammability, and stability in alkaline solutions [4]. A popular way to obtain hydrogen is using sodium boron hydride ($NaBH_4$) which is a purposive hydrogen carrier. Therefore, among the chemical hydrides, $NaBH_4$ is the most studied and researched [5].

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Some of the main advantages of $NaBH_4$ can be listed as;

i) High ability to produce hydrogen at room temperature,

ii) It is a hydrogen reservoir and has a high hydrogen density (About 10%),

iii) It has a high reduction,

iv) It has easy storage and transportation features.

As shown in Equation 1, hydrogen gas and Sodium Metaborate ($NaBO_2$) are released end of the reaction of $NaBH_4$ with water, $NaBH_4$ can be synthesized from $NaBO_2$ which is a by-product. As a result of the study done by Schlesinger, it was observed that $NaBH_4$ hydrolysis was slow at room temperature [6]. Adding catalysts to the hydrolysis reaction increases hydrogen production by reducing the activation effect [5,7]. Most of the $NaBH_4$ hydrolysis studies are based on more efficient and faster hydrogen gas generation than the hydrolysis reaction. The various chemicals used during hydrolysis reactions, it is aimed to produce hydrogen gas in a faster and more sustainable manner.

$$NaBH_4 + 2H_2O \rightarrow NaBO_2 + 4H_2 \tag{1}$$

In the literature, the effect of the catalyst used in the hydrolysis of $NaBH_4$ depends on the large area of the surface, high conductivity or high degree of graphitization, a suitable pore structure to ensure mass transport, and the number of metal particles. Metal-containing catalysts used as catalysts in hydrogen production reactions from $NaBH_4$ have high catalytic activity. Noble metal-based catalysts such as Ru, Pd, and Pt are used in the hydrolysis reaction of $NaBH_4$ [7–9]. These catalysts have been observed to greatly increase the catalytic activity of the reaction, but their use is limited due to their rare and costly properties in the industrial field. In studies where transition metals such as Co, Ni, Fe, and Cu are used, it is expected that these metal catalysts will be used as high-efficiency catalysts, considering the high catalytic activity of Ni and Co metal catalysts and because they are less costly than noble metals. Efforts to synthesize cheaper catalysts are quite popular. Therefore, metal powders are produced from industrial wastes such as Al_2O_3 [10], SiO_2 [11], TiO_2 [12], activated carbon [13] or metal-rich clays [14,15], zeolite [16,17]scanning electron microscope (SEM and ceramics [18]. New catalysts can be synthesized for use in hydrogen production reactions [14]. İskenderoğlu et al. examined the catalytic activity of the raw BFS powder in hydrolysis reactions. The hydrogen production of raw BFS catalyst, which is used with 10% $NaBH_4$ by weight in the reaction at 50 °C, is 54.63 L/min$g_{catalst}$ [19].

Also, Al and water react to hydrogen generation, and the hydrogen gas can be released by the reaction shown in equation 2 at a high temperature or high pressure [20].

$$Al + \frac{3}{2}H_2O ® \ Al_2O_3 + \frac{3}{2}H_2 \tag{2}$$

The results of Zhu et al.'s study show that for Al powder with 7% $NaBH_4$ addition by weight, the maximum hydrogen volume concentration obtained by using steam is approximately 21,000 ppm at 650 °C [21].

In this study, the effects of both BFS powder and Al and Fe_2O_3 metal powders in the content of BFS powder on the hydrolysis reaction of Nano and Granule forms were examined and the results of the experiments were compared. The fact that these metal powders have not been used in studies in the literature before, and the effect of metal powders used in different amounts in the experiments, the effect of BFS powder or HCl acid addition on the test results, makes this study different from its counterparts. As a result, it is aimed to produce efficient hydrogen gas by using a cheaper, more environmentally friendly catalyst material than other catalysts, like many catalyst articles in the literature. In addition to all these, another factor that makes this article special is that the catalyst used is a product that can be obtained from waste product without requiring any processing and by considering the use of zero waste. According to the results obtained as a result of the experiments, it has been proven that the addition of HCl acid to Fe_2O_3 metal powder, which has not been used in hydrolysis experiments before, has a noticeable efficiency effect on hydrogen production.

2. Materials & Experimental Methods

The 98.5% pure $NaBH_4$ that contains 0.05% Si and 0.005% Fe was supplied from a commercial company. Fe_2O_3 was also obtained from a commercial company. Nanosize Fe_2O_3 has 99.55% purity and contains 0.003% Ca, 0.015% Cr, 0.14% Mn, 0.05% Al and 0.091% SiO_2. Al Nano supplied from ZAG Kimya company is 99% pure and 0-150µm in size. Al Granule supplied from Nanokar Company is 98.5% pure and 2 mm in size. The Cobalt (Co) Micron powder was supplied from the Nanografi company. The Cobalt (Co) Micron Powder is 99.99% pure and has a size of 1 µm. BFS was supplied from Iskenderun Iron and Steel factory. the steel BFS, which produced in Turkey, is assumed to consist of SiO_2 (39.66 %), Fe_2O_3 (1.58 %), Al_2O_3 (12.94 %), CaO (34.20 %), MgO (6.94 %), Na_2O (0.20 %), K2O (1.44 %), SO3 (0.72 %) [22].

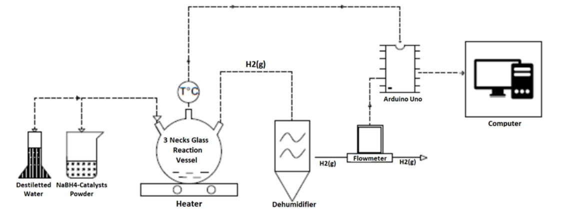

Figure 1. Experimental installation

The schematic view of the experimental setup is shown in Figure 1. It consists of a 4-port reactor, heater, moisture trap, Alicat Scientific brand flow meter, DS18B20 temperature sensor, MTOPS MS300HS brand heater, and Arduino Uno. The amount of $NaBH_4$ was kept constant as 2 g for all experiments. Then, in the hydrolysis reaction of $NaBH_4$, BFS powders, and Fe_2O_3, Al Nano&granule powders were weighed one by one in the analytical balance as 0.2 gr, 0.5 gr, 1 gr, and 2 gr together with distilled water.

The effect of the catalytic activity performances of these powder samples on the hydrolysis reaction of $NaBH_4$ was investigated. These catalytic hydrolysis reactions were carried out in a four-necked round bottom flask reactor. Solid $NaBH_4$ was weighed on an analytical balance and metal powder samples which used as catalysts were transferred to the reactor. Distilled water was added to the solid powder in the reactor by using a measuring cylinder. The reaction temperature was measured by a thermocouple that connected to the Arduino Uno. The hydrogen gas produced as a result of the reaction was passed through a desiccant to separate it from the water vapor contained in it. The volume of hydrogen gas produced was measured by a high precision Alicat Scientific brand flowmeter. The measured data were simultaneously transferred to the microprocessor and saved to the computer.

Hydrogen Production

Hydrogen production took place in a three-necked round bottom flask reactor as shown in Figure 1. The three-necked were used as water and catalyst inlet, thermocouple outlet, and hydrogen outlet connections. All connections of the reactor were wrapped with gas Teflon tape to prevent possible hydrogen leakage, providing a high sealing environment. The catalyst mixture prepared from the water and catalyst inlet and 20 ml of distilled water measured with the aid of a measuring cylinder were transferred into the reactor. Then, with the aid of a heater, the reactor was heated to 50°C. The hydrogen gas produced as a result of the hydrolysis reaction taking place in the reactor was passed through the desiccant and conveyed to the flow meter. The flow rate of hydrogen and the reaction temperature with the thermocouple in the reactor were measured by the flow meter. Using Arduino Uno, data from both the thermocouple and the flow meter were simultaneously recorded in an Excel file.

3. Results and Discussions

3.1. Effect of BFS powder on Hydrolysis Reaction

Experiments were carried out using three different amounts of 2 g, 3 g, and 4 g for the BFS catalyst, 20 ml of distilled water was used for all three experiments and the reaction vessel was heated up to 50 °C. The results obtained from the experiments are shown in Figures 2a and 2b. The experiment using 2 g BFS lasted for 27 minutes and the maximum temperature was up to 59.13 °C. In the experiment, the total flow reached 142.44 L and the maximum flow amount was measured as 0.23 L/min. gcatalyst. The instantaneous flow rate of the reaction was measured as 5.275 L/min. The experiment using 3 g BFS lasted 22 minutes and the maximum temperature was up to 62.88 °C. In the experiment, the total flow reached 146.25 L and the maximum flow amount was measured as 0.28 L/min.gcatalyst. The instantaneous flow rate of the reaction was measured as 6.647 L/min. The experiment using 4 g BFS lasted for 25 minutes and the maximum temperature increased up to 63.81 °C. In the experiment, the total flow reached 216.88 L and the maximum flow amount was measured as 0.28 L/min. gcatalyst. The instantaneous flow rate of the reaction was measured as 8.675 L/min. Among the three reactions, the longest-lasting reaction was the one using 2 g of BFS, and it lags behind the others in terms of total hydrogen produced. The temperatures and maximum flow values reached in the experiments using 3 and 4 gr BFS are approximately the same. In terms of instantaneous flow rate and the total amount of hydrogen produced, the experiment using 4 g of BFS is quite efficient.

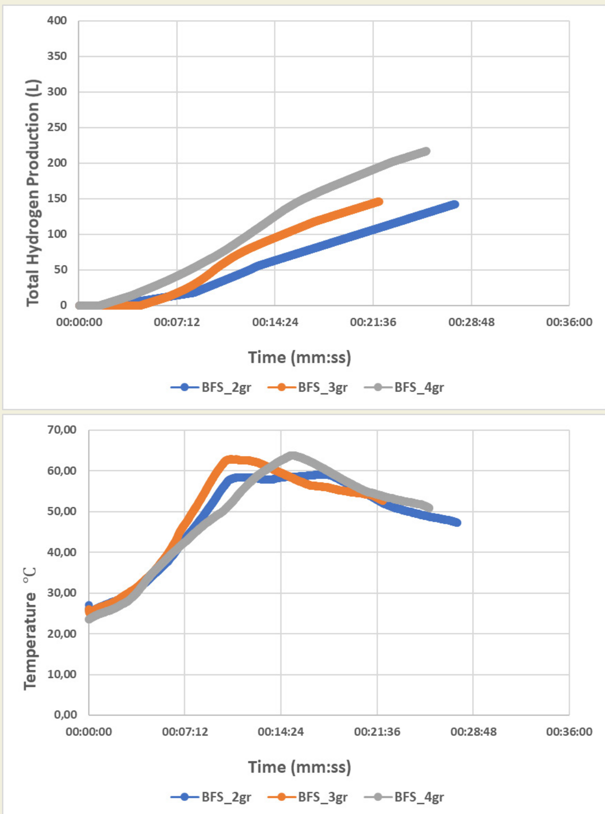

Figure 2. a) The Total Hydrogen Production of Different Amounts of BFS Powders, b) The Temperature Effect of Different Amounts of BFS Powders

3.2. Effect of Granule and Nanoparticles on Hydrolysis Reaction

3.2.1. Effect of Granule particles on Hydrolysis Reaction

Al Granule powders:

When 0.2 gr and 0.5 gr of Al Granule metal powder were used, hydrogen gas output could not be measured because of very low levels. Therefore, experiments were carried out using two different amounts, 1 g, and 2 g, and 20 ml of distilled water and 2 g of NaBH4 were used for both experiments and the reactor was heated up to 50. The results of total hydrogen production and reaction temperatures obtained from the experiments are shown in Figures 3a and 3b. According to these Figures, the experiment using 1 gr Al Granule lasted 26 minutes and the maximum temperature was up to 67.62 °C. The total flow amount measured in the experiment is 166.3 L and the maximum flow rate was measured as 0.18 L/min.gcatalyst. The instantaneous flow rate of the reaction was measured as 6.396 L/min. The experiment using 2 gr Al Granule lasted 26 minutes and the maximum temperature was up to 67.19 °C. The total flow amount measured in the experiment is 172.92 L and the maximum flow rate was measured as 0.28 L/min.gcatalyst. The instantaneous flow rate of the reaction was measured as 6.065 L/min. Test times and maximum temperatures attained for both catalyst quantities are approximately the same. When the amount of hydrogen produced was compared, it was observed that when 2 g of Al granule was used, there was 6 L more production.

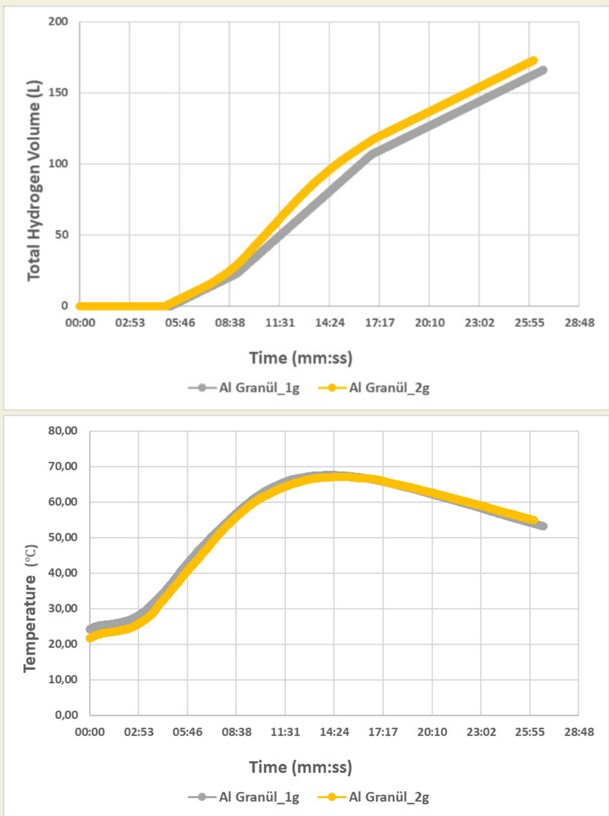

Figure 3. a) The Total Hydrogen Production of Different Amounts of Al Granule Powders, b) The Temperature Effect of Different Amounts of Al Granule Powders

Fe_2O_3 Granule powders:

Fe_2O_3 Granule catalyst was used in 3 different amounts, 0.2 gr- 0.5 gr-1 gr. In all experiments, hydrolysis reactions were carried out using 20 ml of distilled water and 2 g of $NaBH_4$, and the reaction vessel was heated up to 50°C and closed. The results obtained from the experiments are shown in Figures 4a and 4b. In the experiment performed with 0.2 gr Fe_2O_3 catalyst, the reaction time, the maximum temperature reached by the reaction, the maximum flow rate of the reaction, the instantaneous flow rate of the reaction, and the total flow rate were 12 minutes, 88.25 °C, 1.99 L/min.$g_{catalyst}$, 28.47 L/min, and 341.65 L, respectively. was measured. In the experiment where 0.5 g Fe_2O_3 catalyst was used, the reaction time, the maximum temperature reached by the reaction, the maximum flow rate of the reaction, the instantaneous flow rate of the reaction, and the total flow rate were 12 minutes, 87.06°C, 2.52 L/min.$g_{catalyst}$, 28.26 L/min, and 339.21 L, respectively. The last experiment, which used 1 g Fe_2O_3 Nanopowder, ended at 19 minutes, and the highest temperature reached by the reaction was measured as 90.25 °C. The total flow in the experiment is 304.37 L, the maximum flow amount is 4.33 L/min.$g_{catalyst}$ and the instantaneous flow amount in the reaction is 33.81 L/min.

In the experiments of Fe_2O_3 Granule with 0.2 gr- 0.5 gr

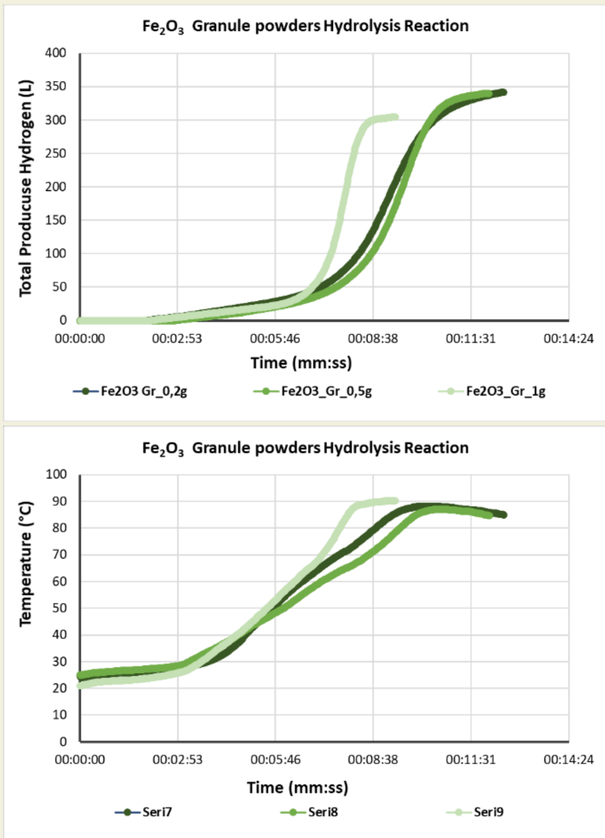

Figure 4. a) The Total Hydrogen Production of Different Amounts of Fe2O3 Granule Powders, b) The Temperature Effect of Different Amounts of Fe2O3 Granule Powders Powders

-1 gr, the reaction temperature, maximum flow amount and instantaneous flow amount are approximately close to each other. However, the experiment in which the total flow was the highest in these experiments according to the nanosize was the experiment using 0.2 gr Fe_2O_3 Granule.

3.2.2. Effect of Nanoparticles on Hydrolysis Reaction

Al Nano Powders:

Experiments were carried out using 4 different amounts of metal powder of Al Nano as 0.2 gr, 0.5 gr, 1 gr, and 2 gr. For all experiments, 20 ml of distilled water, and 2 g of NaBH4 were used and the reaction vessel was heated to 50 °C. The results obtained from the experiments are shown in Figures 5a and 5b. The experiment using 0.2 gr Al Nano lasted 26 minutes and the maximum temperature was up to 58.44 °C. The total flow in the experiment was 184.05 L and the maximum flow amount was measured as 0.28 L/min.gcatalyst. The instantaneous flow rate of the reaction was measured as 7.078 L/min. The experiment using 0.5 g Al Nano lasted 31 minutes and the maximum temperature was up to 73 °C. The total flow in the experiment was 316.11 L and the maximum flow amount was measured as 0.42 L/min.gcatalyst. The instantaneous flow rate of the reaction was measured as 10.197 L/min. The experiment using 1 gr Al Nano lasted 28 minutes and the maximum temperature was up to 84.31 °C. The total flow in the experiment was 366.81 L and the maximum flow amount was measured as 0.62 L/min.gcatalyst. The instantaneous flow rate of the reaction was measured as 13.100 L/min.

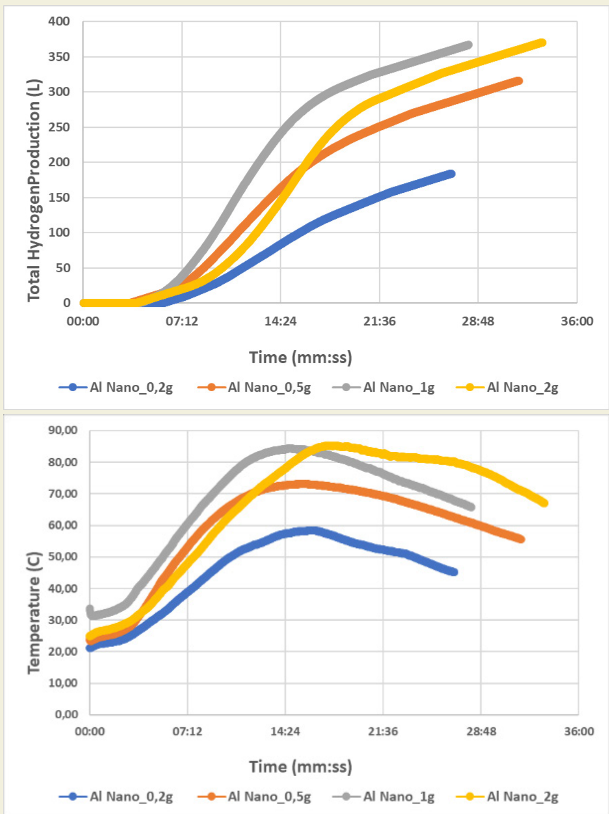

Figure 5. a) The Total Hydrogen Production of Different Amounts of Al Nano Powders, b) The Temperature Effect of Different Amounts of Al Nano Powders

The experiment using 2 gr Al Nano lasted 33 minutes and the maximum temperature was up to 85.31 °C. The total flow in the experiment was 370.46 L and the maximum flow amount was measured as 0.52 L/min.gcatalyst. The instantaneous flow rate of the reaction was measured as 11.226 L/min. For Al Nano, the reaction time and temperature increased as the amount of catalyst increased. In addition, the total amount of hydrogen produced and the instantaneous flow rates have also increased. If we examine Figures 5a and 5b, we can say that the most efficient is 2 gr Al Nano.

Fe_2O_3 Nano Powders

Fe2O3 Nanocatalyst was used in 3 different amounts, 0.2 gr- 0.5 gr -1 gr. In all experiments, hydrolysis reactions were carried out using 20 ml of distilled water and 2 g of NaBH4, and the reaction vessel was heated up to 50°C and closed. The results obtained from the experiments are shown in Figures 6a and 6b.In the experiment performed with 0.2 gr Fe2O3 catalyst, the reaction time, the maximum temperature reached by the reaction, the maximum flow rate of the reaction, the instantaneous flow rate of the reaction, and the total flow rate were measured at 26 minutes, 79.19 °C, 0.57 L/min.gcatalyst, 12.55 L/min, and 339.07 L, respectively. In the experiment where 0.5 g Fe2O3 catalyst was used, the reaction time, the maximum temperature reached by the reaction, the maximum flow rate of the reaction, the instantaneous

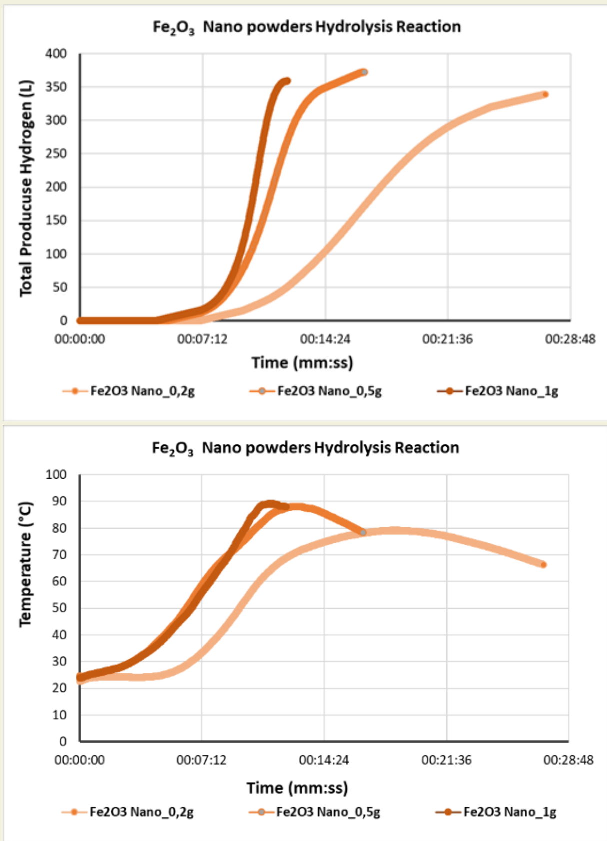

Figure 6. a) The Total Hydrogen Production of Different Amounts of Fe_2O_3 Nano Powders, b) The Temperature Effect of Different Amounts of Fe_2O_3 Nano Powders

flow rate of the reaction, and the total flow rate were 16 minutes, 88.12°C, 1.5 L/min.gcatalyst, 23.28 L/min, and 372.5 L, respectively. The last experiment, which used 1 g Fe2O3 Nano, ended in 12 minutes and the highest temperature reached by the reaction was measured as 89.25 °C. The total flow in the experiment is 358.97 L, the maximum flow amount is 2.53 L/min.gcatalyst and the instantaneous flow amount in the reaction is 29.91 L/min.

0.2 gr- 0.5 gr-1 gr Fe_2O_3 In nano experiments, the highest temperature, maximum flow amount, and total flow values were seen in the experiment performed with 1 gr Fe_2O_3. The experiment with the highest total flow amount is the value made with 0.5 gr Fe_2O_3 Nano. The longest experiment was the experiment using 0.2 gr Fe_2O_3 Nano. As a result, with the increase in the amount of catalyst used in hydrolysis reactions, the total amount of hydrogen gas produced increases in direct proportion.

3.2.3. Comparison of Nano and Granule particules

As seen in Figure 7, it is more efficient than Al Granule in experiments where Al Nano is used as a catalyst. The amount of hydrogen produced is approximately 2 times higher. This is because the surface area width of Al Nano is more than Al Granule. This is because the solution formed in the hydrolysis reaction comes into contact with more catalyst surface area. In this case, it causes an increase in the reaction time. If two different amounts of Al Granule are compared, it can be observed that 1 gr Al Nano gives better results in experiments using Al Nano, although they are approximately the same efficiency.

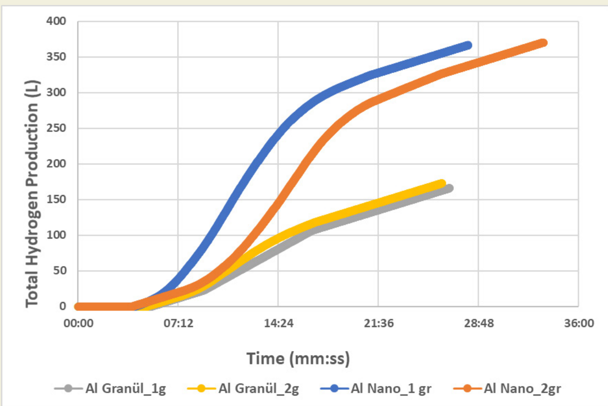

Figure 7. Time-Dependent Total Hydrogen Produced in Al Nano and Al Granule

In 0.2 gr experiments, Fe_2O_3 Granule produced more hydrogen than Fe_2O_3 Nano and reached higher temperatures. As seen in Figure 8, Fe_2O_3 Granule gave better results at maximum flow rate and instantaneous flow rate. In the 0.5 g experiment, Fe_2O_3 Nano produced more hydrogen and increased to a higher temperature. Fe_2O_3 Granule reached a higher value at maximum flow amount and instant flow rate. In 1 gr experiments, Fe_2O_3 Granule has the highest temperature value and Fe_2O_3 Nano has the highest amount of hydrogen produced. Fe_2O_3 Granule has higher values at maximum flow amount and instant flow rate.

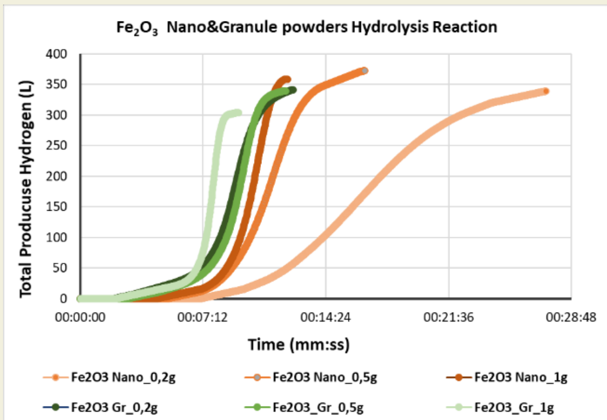

Figure 8. Time-Dependent Total Hydrogen Produced in Fe_2O_3 Nano and Fe_2O_3 Granule

3.3. Adding BFS To The Hydrolysis Experiment

3.3.1. Effect of Al Nano and BFS on Hydrolysis Reaction

1 g and 2 g of Al nano metal powder, 1 g of BFS, and 2 g of $NaBH_4$ were reacted. For both experiments, 20 ml of distilled water was used and the reaction vessel was heated to 50 °C. The results obtained from the experiments are shown in Figures 9a and 9b. The experiment using 1 gr Al Nano lasted 28 minutes and the maximum temperature was up to 71°C. The total flow in the experiment was 189.18 L and the maximum flow amount was measured as 0.28 L/min.gcatalyst. The instantaneous flow rate of the reaction was measured as 6.756 L/min. The experiment using 2 gr Al Nano lasted 25 minutes and the maximum temperature was up to 71°C. The results obtained from the experiments are shown in Figures 9a and 9b. The total flow in the experiment was 216.45 L and the maximum flow amount was measured as 0.28 L/min.gcatalyst. The instantaneous flow rate of the reaction was measured as 8.538 L/min.

The addition of BFS to the Al Nano metal powder kept the reaction time of 1 g Al Nano the same as in the BFS-free experiment. However, it reduced the maximum temperature reached by the reaction by 13 °C. It reduced the instantaneous flow rate of the reaction by 7 L/min and the total hydrogen flow amount by 177 L. For 2 gr Al Nano, it extended the reaction time by 8 minutes. With this; It decreased the maximum temperature value by 14 °C, the total hydrogen flow amount by 154 L, and the instantaneous flow rate by 3 L/min, respectively.

3.3.2. Effect of Fe_2O_3 Nano&Granule and BFS on Hydrolysis Reaction

In this experiment, in addition to 20 ml distilled water, 2 gr NaBH4 and 0.5 gr Fe2O3 Granule powder, 2 gr BFS was used. The reaction was completed in 18 minutes and

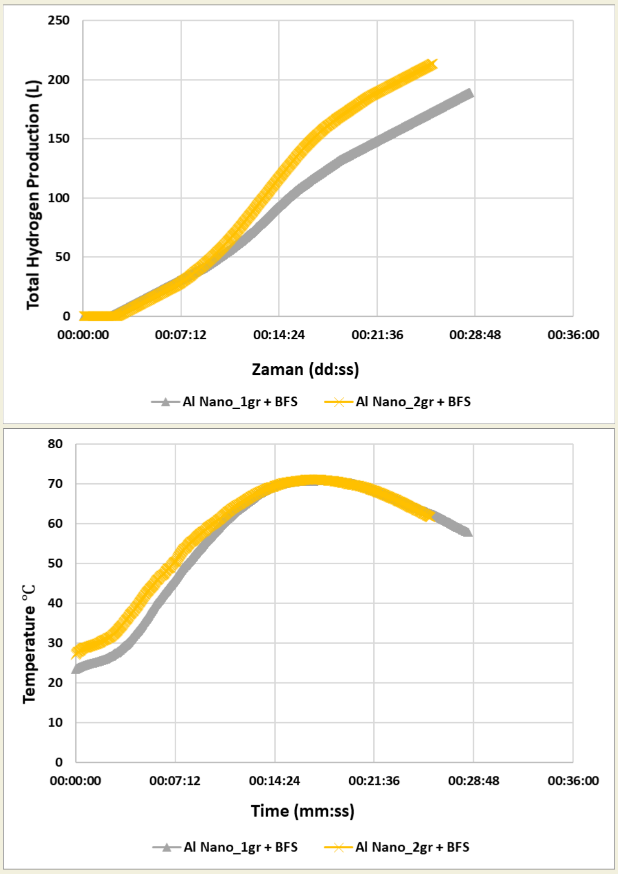

Figure 9. a) The Total Hydrogen Production of Different Amounts of Al Nano&BFS Powders, b) The Temperature Effect of Different Amounts of Al Nano&BFS Powders

the maximum temperature was measured as 84.62°C. The results obtained from the experiments are shown in Figures 10a and 10b.In this experiment, a total of 290.08 L of hydrogen is produced, the maximum flow is 1.01 L/min. gcatalyst, and the instantaneous speed of the experiment is 16.11 L/min. These reaction results gave lower values than the 0.5 g Fe_2O_3 Granule test without BFS.

In this experiment, in addition to 20 ml distilled water, 2 gr $NaBH_4$, and 0.5 gr Fe_2O_3 Nanopowder, 2 gr BFS was used. The experiment lasted 19 minutes and the maximum temperature was 82.06°C. In this experiment, a total of 262.9 L hydrogen was produced, and the maximum flow was measured as 0.67 L/min.gcatalyst. The instantaneous velocity of the experiment is 13.83 L/min. These values gave lower results than the 0.5 gr Fe_2O_3 Nano experiment without BFS.

3.3.3. Comparison of Fe_2O_3 Granule, Fe_2O_3 Nano, and Al Nano Experiments Using BFS

The BFS, Al Nano, Al Nano + BFS powders used in the same amounts were compared according to Figure 11. The catalyst that produces the least hydrogen compared to other amounts is 2 g of BFS. The experiment in which Al Nano and BFS powder are used together is more efficient than the experiment in which only BFS powder is used. For this reason, although the BFS powder harmed the Al Nano experiment, it was observed that the Al Nanopowder had a positive effect on the hydrolysis experiments in which the BFS was used.

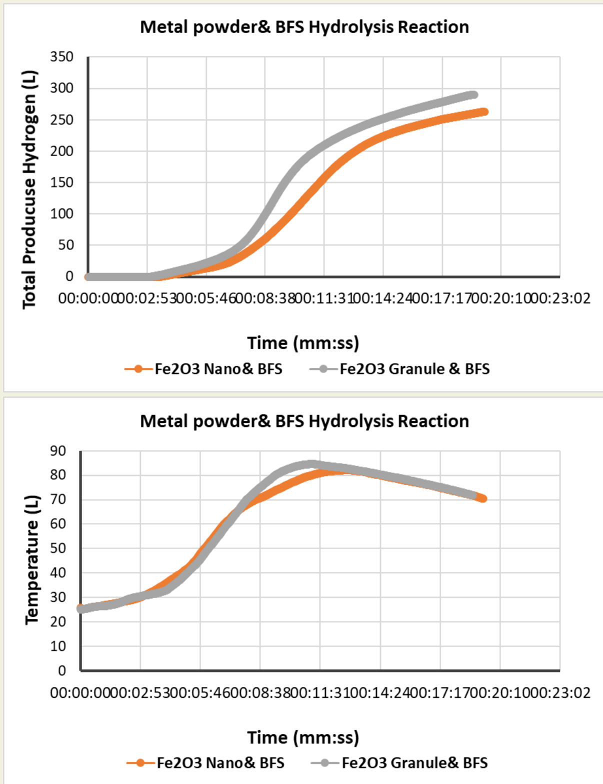

Figure 10. a) The Total Hydrogen Production of Different Amounts of Fe_2O_3 Nano&BFS Powders, b) The Temperature Effect of Different Amounts of Fe_2O_3 Nano&BFS Powders

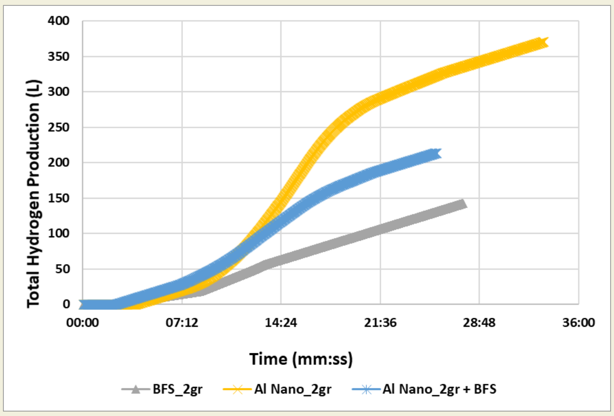

Figure 11. Effect of Al Nano + BFS Powder on Hydrolysis Reaction

The BFS, Fe_2O_3 Granule, Fe_2O_3 Nano, Fe_2O_3 Nano + BFS, and Fe_2O_3 Granule + BFS powders used in the same amounts were compared according to Figure 12. The 0.5 gram Fe_2O_3 Granule test with BFS gave better results than the BFS Fe_2O_3 Nano test. The BFS Fe_2O_3 Nano experiment lasted longer than the experiment using granules.

3.4. Adding HCl Acid to the Hydrolysis Reaction

Al Metal Powders

In addition to the experiments above, the effect of HCl

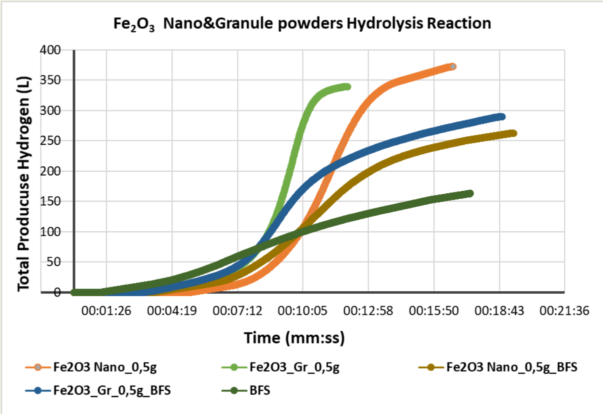

Figure 12. Effect of 0.5 gr Fe_2O_3 Nano, Fe_2O_3 Nano+BFS, Fe_2O_3 Granule, Fe_2O_3 Granule+BFS Powder on Hydrogen Production

acid in experiments using Al Metal powders is examined in this section. In addition to 2 gr Al Granule or 2 gr Al Nanopowders, 1 ml HCl acid was added to the reaction using 2 gr $NaBH_4$. For both experiments, 20 ml of distilled water was used and the reaction vessel was heated to 50 °C. The results obtained from the experiments are shown in Figure 12. The experiment using 2 gr Al Granule and 1 ml HCl acid took 4 minutes and the maximum temperature was up to 80.87°C. In the experiment, the total flow was 332.25 L and the maximum flow amount was measured as 11.18 L/min.gcatalyst. The instantaneous flow rate of the reaction was measured as 80.562 L/min. The experiment using 2 gr Al Nano and 1 ml HCl acid took 11 minutes and the maximum temperature was 70.44°C. The total flow in the experiment was 234.52 L and the maximum flow amount was measured as 5.75 L/min.gcatalyst. The instantaneous flow rate of the reaction was measured as 23.452 L/min. Adding HCl acid to Al Granule metal powders decreased the reaction time by 22 minutes compared to the experiment without adding HCl acid. The maximum temperature reached by the reaction increased by 13 °C, the instantaneous flow rate of the reaction increased by 74 L/min, and the total hydrogen flow rate by 150 L.

The addition of HCl acid to Al Nano metal powders decreased the reaction time by 22 minutes compared to the experiment without adding HCl acid. However, compared to the Al Granule experiments, it decreased the maximum temperature reached by the reaction by 15 °C and the total hydrogen flow amount by 140 L, respectively. However, it increased the instantaneous flow rate of the reaction by 12 L/min. This is due to the hydrogen gas released as a result of the reaction of HCl acid with water.

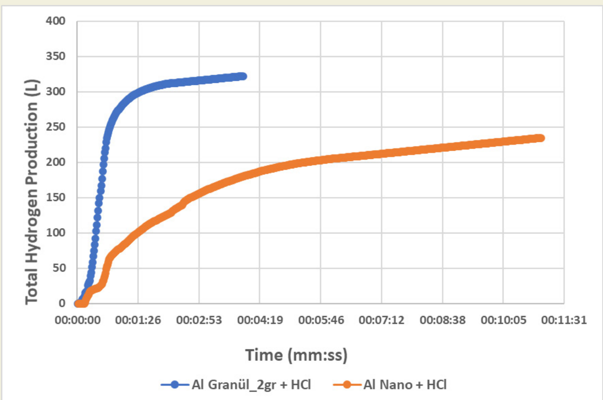

Figure 13. The effect of adding HCl Acid to the Hydrolysis Reaction with Al Nano and Granule catalysts

Fe_2O_3 *Metal Powders*

In this experiment, in addition to 20 ml distilled water, 2 gr $NaBH_4$, 0.5 gr Fe_2O_3 Granule or 0.5 gr Fe_2O_3 Nanopowder, 1 ml of HCl acid was added to the reaction. The reaction vessel was heated to 50 °C. In the experiment using 0.5 gr Fe_2O_3 Granule and 1 ml HCl acid, the reaction time, the maximum temperature reached by the reaction, the maximum flow rate of the reaction, the instantaneous flow rate of the reaction, and the total flow rate were 16 minutes, 77.62 °C, 3.36 L/min.gcatalyst, 18.11 L, respectively. It is measured as /min and 289.76 L. In the experiment using 0.5 gr Fe_2O_3 Nano and 1 ml HCl acid, the reaction time, the maximum temperature reached by the reaction, the maximum flow rate of the reaction, the instantaneous flow rate of the reaction, and the total flow rate were measured 7 minutes, 87.62 °C, 4.53 L/min.gcatalyst, 69.49 L/min, and 416.95 L respectively. The addition of HCl acid to Fe_2O_3 Granule metal powders increased the reaction time by 4 minutes compared to the experiment without adding HCl acid. The maximum temperature reached by the reaction increased by 9.4 °C, also increased the instantaneous flow rate of the reaction by 10.16 L/min, and decreased the total amount of hydrogen produced by 49.45 L. Addition of HCl acid to Fe_2O_3 Nano metal powders increased the reaction time by 10 minutes compared to the experiment without adding HCl acid. However, according to the Fe_2O_3 Granule ex-

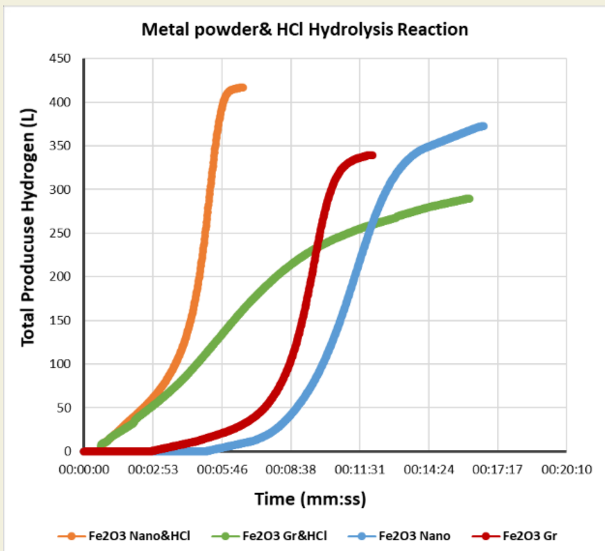

Figure 14. Effect of 0.5 gr Fe_2O_3 Nano, Fe_2O_3 Nano+HCl Fe_2O_3 Granule, Fe_2O_3 Granule+HCl Powder on Hydrogen Production

periments, the maximum temperature reached by the reaction is approximately the same, respectively, it increased the total hydrogen flow amount by 44.45 L and decreased the instantaneous flow rate of the reaction by 8.6 L/min.

3.5. Discussion

In this study, it has been proved by experiments that Al and Fe_2O_3 catalysts can be used as efficient catalysts in $NaBH_4$ hydrolysis reaction. In these experiments, the catalyst samples were made suitable for the $NaBH_4$ hydrolysis reaction by various experiments in different amounts, BFS by-product, and HCl acid effect. In this study, the analysis of the hydrogen production reaction carried out under 5 different conditions with the Al and Fe_2O_3 catalysts was made. It has been shown that both types of catalysts can be used efficiently in hydrolysis reactions when the experimental results of these metal powder catalysts are compared. According to the results of these optimization experiments, more effective test results are obtained by using different parameters and combinations.

In all experiments, hydrolysis reactions were carried out by using 20 ml of distilled water and 2 g of $NaBH_{4,}$ and the reaction vessel was preheated to 50 ° C. Al and Fe_2O_3 Nano metal powders catalyst were used in 3 different amounts as 0.2 g - 0.5 g - 1 g. During the experiments with 0.2 g, 0.5 g, and 1g of Fe_2O_3 catalyst, the total flow rates were 339.07 L, 372.5 L, and 358.97 L, respectively. On the other hand, the total flow rate results of Al catalyst for 0.2 g, 0.5 g, and 1g usages were 184.05 L, 316.11 L, and 366.81 L, respectively. In the hydrolysis experiment in which Fe_2O_3 metal powder was used together with BFS, approximately 45% more hydrogen production was observed compared to the experiment using only BFS. In the Al Nano experiment with the addition of BFS, approximately 30% more hydrogen production was observed compared to the experiment using only BFS. However, the use of BFS does not increase the yield in hydrolysis reactions where only Al and Fe_2O_3 metal powders are used as catalysts. In hydrolysis reactions using HCl acid, total hydrogen production increased by 13% in the experiment where Fe_2O_3 Nano and HCl acid were used together, while the total amount of hydrogen produced increased by 20% in the experiment where Al Nano and HCl acid were used together. It has been observed that adding HCl acid gives efficient results in the reaction. Therefore, it can be recommended to use HCl acid solutions with different molarities in reactions.

4. Conclusions

In this study, the yields of Fe_2O_3 and Al catalysts were compared with the experiments conducted for hydrogen production from the catalytic hydrolysis of $NaBH_4$ supported with BFS by-product and HCl acid. As a result of the obtained data, it was determined that Fe_2O_3 and Al catalysts have advantages over hydrogen production rate and fuel conversion. In line with the results obtained from the experiments, the Fe_2O_3 catalyst and the Fe_2O_3 catalyst & HCl acid experiments show a very high success in different parameters, and also Fe_2O_3 catalysts create promising effects in the hydrolysis reactions of $NaBH_4$. Among the catalyst samples, the highest efficiency performances are achieved with Fe_2O_3 Nanocatalyst samples at 50 °C preheat with an instantaneous hydrogen generation rate of approximately 29.91 L / min for 12 minutes. There is no study in the literature with Fe_2O_3 catalysts in $NaBH_4$ hydrolysis reactions. In addition, other metal catalysts used in these reactions are quite expensive and inefficient compared to Fe_2O_3 catalysts. The use of Fe_2O_3 catalysts as cheap and efficient catalysts in hydrolysis reactions has made a great contribution to literature studies.

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