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Kinetic studies on the $NaBH_4/H_2O$ hydrogen storage system with $CoCl_2$ as a catalyst

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Hydrogen energy is considered at worldwide scale as a substitute to replace fossil fuels and to establish clean and secure energy future. Hydride based hydrogen storage, for instance sodium borohydride ($NaBH_4$), has acknowledged substantial concern as a potential hydrogen source for portable fuel cell applications. The present paper highlights the kinetic studies of the $NaBH_4/CoCl_2/H_2O$ system and the factors affecting the hydrogen generation rate (HGR) - $NaBH_4$ concentration, NaOH concentration and temperature. It also reports the results on addition of alumina nanoparticles to the $NaBH_4/CoCl_2/H_2O$ system, for accelerating the hydrogen generation rate, owing to better reactivity of the nanoparticles. Cobalt chloride is extensively used as a catalyst, being a reactive and cost-effective non-noble metal that expedites $NaBH_4$ hydrolysis reaction. Kinetic studies show that the hydrolysis reaction of $NaBH_4$ is of first order. The rate increases with respect to sodium borohydride and decreases with respect to sodium hydroxide. It is also observed that the rate increases with the increase in temperature. Characterization of residue is performed by SEM, EDS and XRD analysis that confirmed that the particles agglomerate together. Boron, sodium, cobalt, chlorine and oxygen are present with weight percentages of 78.46%, 3.41%, 0.05%, 0.07% and 18.01%, respectively. The diffraction peak at $2\theta = 45^o$ predicted from XRD analysis shows the presence of Co_2B in the residue.

Keywords: Hydrogen energy; Sodium borohydride; Cobalt chloride; Alumina, Nanoparticles

INTRODUCTION

Inquisitiveness in hydrogen as a fuel has grown and numerous advances in hydrogen production and utilization have been made. Technologies for hydrogen storage ought to be significantly advanced if a hydrogen based energy system, particularly in the transportation sector, is to be acknowledged [1]. Storing hydrogen is relatively difficult because of its low density and critical temperature. Presently, there are three ways to store hydrogen: (a) as a compressed gas, (b) as cryogenic liquid hydrogen (LH_2) and (c) solid-state hydrogen storage [2, 3]. The main drawback of compressed gas and cryogenic liquid hydrogen storage is the low volumetric density of the system. Solid-state hydrogen storage involves storage of hydrogen in complex chemical hydrides. These hydrides have high hydrogen content and hydrogen can be released through several chemical pathways. Hydrolysis of chemical hydrides takes place at comparatively low temperatures and gives promising theoretical hydrogen storage efficiencies [4, 5].

Among diverse chemical hydrides, sodium borohydride ($NaBH_4$) has been considered as the most attractive hydrogen storage material. It is a safe practical means of producing hydrogen and has high hydrogen content (10.7 wt %) [6, 7]. Hydrolysis of sodium borohydride is a heat releasing reaction. No side reactions or volatile byproducts are formed and the generated hydrogen is of high purity (no traces of CO and S) and humidified (heat generates some water vapor). Additionally, sodium borohydride is the least expensive metal hydride commercially available. Furthermore, it is safe to use, handle and store. Eqn. 1 gives the hydrolysis reaction of $NaBH_4$.

$$NaBH_4 + 2H_2O \rightarrow NaBO_2 + 4H_2 \qquad (1)$$

Rate kinetics of the above reaction is slow without a catalyst that affects the hydrogen release. Therefore, for the fast kinetics of the hydrolysis reaction of $NaBH_4$, a highly reactive catalyst is obligatory. Many catalysts like $RuCl_3$, $RhCl_3$, $CoCl_2$, H_2PtCl_2, $FeCl_2$, and $PdCl_2$ are used for $NaBH_4$ hydrolysis. In terms of reactivity and cost, $CoCl_2$ is the most favored one [8]. As compared with other non-noble metal catalysts like nickel, maximum hydrogen generation is reported with cobalt based catalysts [9]. Catalytic potential of cobalt (II) and (III) salts has been studied in the literature and $CoCl_2$ showed the preeminent performance as a catalyst for hydrolysis reaction of $NaBH_4$ [10-13]. It is also found that the catalytic active phase of $CoCl_2$ as a catalyst in $NaBH_4$ hydrolysis is cobalt boride that could be formed as Co_xY or Co_xB_y [14]. Heat of hydrolysis of sodium borohydride is lower when compared with other chemical hydrides as shown in Table 1 [15].

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Hydrogen storage as high pressurized gas or liquid cannot fulfill future storage goals. Chemically or physically combined storage of hydrogen in other materials has various advantages over other storage methods. Intensive kinetic studies have been done on complex hydrides for progression of hydrogen generation (HG) properties [4].

Efficiency of hydride based hydrogen storage systems can be improved if the particle size is reduced to the low nanometer range. Hydrogen desorption rate increases due to the high surface-to-volume ratio of the nanoparticles, which further reduces the external energy required for hydrogen desorption [16]. Studies are also carried out on hydrogen generation with a combination of nanoparticles of alumina with $NaBH_4$ due to a mutual promoting mechanism that exists between hydrolysis of $NaBH_4$ and Al_2O_3/H_2O reactions [17, 18]. The constituted dual fuel system exhibited complementary attributes, such as eminent hydrogen density, reduced material cost and adequate controllability of the reactions at moderate temperatures, making it promising for mobile and portable hydrogen source applications [17].

The present kinetic study reports on the reactivity of the $NaBH_4$-$CoCl_2$ system and the effect of Al_2O_3 nanoparticles on the hydrogen generation rate for the $NaBH_4$-$CoCl_2$ system.

MATERIALS AND METHODS

The chemicals used in the investigation were sodium borohydride powder (purity 97%), cobalt chloride salt powder in hexa-hydrate form (purity of 98%) procured from Loba Chemie Ltd., and sodium hydroxide pellets (purity 97%) procured from CDH Laboratory and Al_2O_3 nanoparticles (20 nm, 99% purity) from Sigma Aldrich.

The schematic experimental setup used for the study is shown in Fig.1. It consists mainly of a three-port reactor, the left-hand port of which is equipped with a thermowell for placing a thermometer; the middle port is connected to a dropping funnel and the right-hand port guides the generated hydrogen gas to a water-replacement system. The water-replacement system consists of a measuring cylinder filled with water and a water container to submerge the cylinder. The volume of the hydrogen generated is measured using the measuring cylinder. To adjust the reaction temperature in the reactor, thermostatically controlled water bath is used.

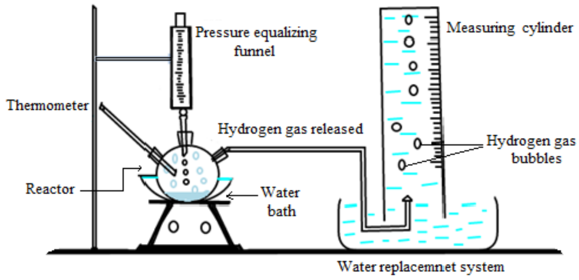

Fig.1. Schematic diagram of the experimental setup.

Dried $NaBH_4$ and $CoCl_2$ are put in the reaction flask. Then 10 mL solution of NaOH and water is added with a pressure-equalizing funnel and guided to the reactor. Hydrogen gas is collected in the measuring cylinder by downward displacement of water and the volume of hydrogen produced is monitored with time.

The residue left after hydrolysis reaction of $NaBH_4$ is kept in an incubator at 60-70°C overnight for drying. The black solid obtained after drying is characterized by SEM (scanning electron microscopy), EDS (energy dispersive X-ray spectroscopy) and XRD (X-ray diffraction) analysis.

Table 1. Comparison of chemical hydrides with heat of hydrolysis reaction [5]

Sample No.	Hydride Name	% H_2 by weight	% H_2 in stoichiometric mixture	Heat of hydrolysis reaction (kJ/mole H_2)
1.	$LiBH_4$	18	8.5	-90
2.	LiH	13	8	-145
3.	$NaBH_4$	10.5	7.5	-80
4.	$LiAlH_4$	10.5	7.5	-150
5.	AlH_3	10	7	-158
6.	MgH_2	7.5	6.4	-161
7.	$NaAlH_2$	7.2	6	-140
8.	CaH_2	4.9	5.1	-140
9.	NaH	4.9	4	-150

The morphology of the residue was observed on a scanning electron microscope (JEOL JSM-6510 LV) equipped with an integrated silicon drift detector (SDD) technology to carry EDS analysis. The specimens were coated with 50 μm thick gold film in an automatic sputter coater (Polaron) to avoid charging under an electron beam prior to SEM.

Structural characterization of the residue was carried out by recording its X-ray diffraction patterns using PANalytical X'Pert Pro Diffractometer operated at 45 kV and 40 mA with monochromatic Cu–Kα radiation (λ=1.5406 Å), in the 2θ range of 20 - 80°.

ANALYSIS

Qualitative analysis of hydrogen gas is done by pop test and the presence of hydrogen gas is confirmed by a squeaky pop sound. Quantitative analysis test is conducted by gas chromatography on AIMIL-NUCON gas chromatograph. The analysis confirmed the presence of hydrogen gas with a purity of 85%, the rest being nitrogen from air as per recovery basis from the sample. The rate eqns. describing the relation between the rate of hydrogen generation and the concentration of sodium borohydride are given below.

$$r_{H_2} \approx C^{\alpha}{}_{NaBH_4} \qquad (2)$$

Here r_{H_2} is the rate of hydrogen generation in moles/L.sec, C_{NaBH_4} is the concentration of $NaBH_4$ in moles/L, and α is the reaction order. Hydrogen generation rate (HGR) linearly decreases with the increase in NaOH concentration at a fixed $NaBH_4$ concentration and temperature. As a result,

$$r_{H_2} = \frac{1}{(1 + k_1\, C_{NaOH})} \qquad (3)$$

where, C_{NaOH} is the concentration of NaOH in moles/L and k_1 is a proportionality constant. Combining eqns. (2) and (3), the rate of hydrogen generation is obtained assuming k to be another proportionality constant [19].

$$r_{H_2} = \frac{kC^{\alpha}{}_{NaBH_4}}{1 + k_1 C_{NaOH}} \qquad (4)$$

RESULTS AND DISCUSSION

In the present study, the factors affecting the rate of sodium borohydride hydrolysis reaction: sodium borohydride concentration, sodium hydroxide concentration and temperature were observed. The hydrolysis reaction is given below [20].

$$\begin{aligned} &CoCl_2 + 2NaBH_4 + 3H_2O \rightarrow \\ &0.5CO_2B\downarrow + 2NaCl + 1.5HBO_2 + 6.25H_2\uparrow \end{aligned} \qquad (5)$$

Effect of $NaBH_4$ concentration on the rate

In a series of experiments by varying the initial concentration of $NaBH_4$ and keeping the NaOH (0.25 moles/L) and $CoCl_2$ (0.02 moles/L) concentrations constant, the effect of $NaBH_4$ on HGR was determined. On increasing the concentration of sodium borohydride, the number of collisions between the molecules increases, consequently increasing the rate of the reaction. According to Fig. 2, the HGR increases with increase in $NaBH_4$ concentration at constant NaOH and $CoCl_2$ concentrations. The solution becomes more viscous at higher concentrations of $NaBH_4$ due to the conversion of $NaBH_4$ to sodium metaborate [21].

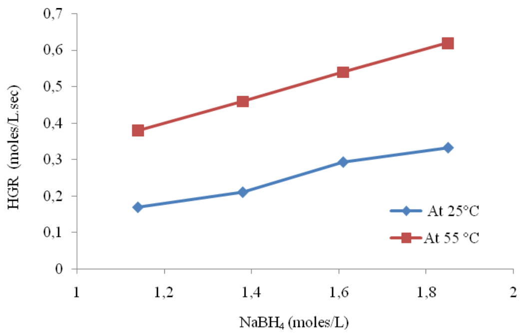

Fig. 2. Effect of $NaBH_4$ concentration on HGR.

Effect of temperature on the rate

The temperature is a measure of the kinetic energy of a system. Higher temperature implies higher average kinetic energy of molecules and more collisions occurring per unit time. Acceleration of H^+, OH^-, BH^{4-} and Cl^{2+} ions is directly related to the temperature of hydrolysis reaction [22]. The reaction rate increases with the increase in temperature as indicated by the slope of the line shown in Fig. 3.

Readings are taken at $NaBH_4$ equal to 1.38 moles/L and $CoCl_2$ 0.02 moles/L. Activation energy calculated for $NaBH_4/CoCl_2/H_2O$ system from the Arrhenius equation is E = 37.931 kJ/mol and A = 12.54 × 10^8 sec^{-1}.

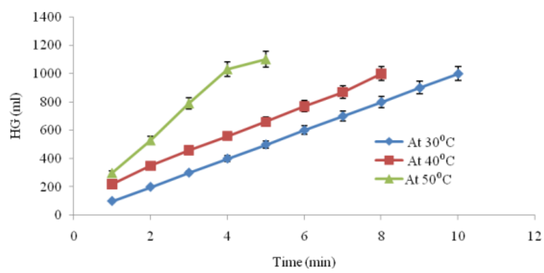

Fig. 3. Hydrogen generation with time at different temperatures

Effect of sodium hydroxide (NaOH) concentration on the rate

To increase the shelf life of $NaBH_4$ solutions and to suppress its self-hydrolysis, $NaBH_4$ solutions were maintained at alkaline conditions by adding NaOH. According to Fig. 4 (a, b), HGR initially increases with the increase in NaOH but decreases beyond 0.25 moles/L. These experiments were conducted at 1.14 and 1.38 moles/L $NaBH_4$ and constant $CoCl_2$ (0.02 moles/L) concentration at 45°C.

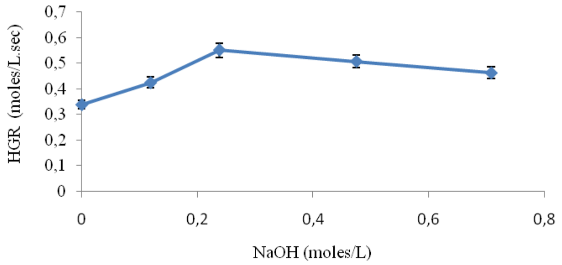

Fig. 4(a). Variation of HGR with NaOH concentration at 1.14 moles/L $NaBH_4$.

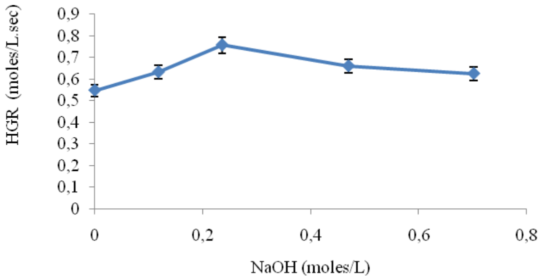

Fig. 4(b). Variation of HGR with NaOH concentration at 1.38 moles/L $NaBH_4$.

These observations were made at relatively low concentrations of NaOH and it was observed that at higher concentrations of NaOH, HGR decreases due to high alkalinity of the solution.

REGRESSION ANALYSIS

It is assumed that the maximum hydrogen generation rate is the initial value of hydrogen generation. The parameters ($k/(1 + k_1C_{NaOH})$) and α in Eqn. (4) can then be determined by regression analysis and are given in Table 2. To determine the rate constants k and k_1, Eqn. (4) was modified to Eqn. (6).

$$\frac{1}{r_{H_2}} = \frac{1}{k} C_{NaBH_4} + \frac{k_1}{k} \frac{C_{NaOH}}{C_{NaBH_4}} \quad , (6)$$

Table 2. Kinetic parameters at different temperatures.

Sample No.	Temperature (°C)	NaOH (moles/L)	$k/(1 + k_1C_{NaOH})$ (mL^2/mol.sec)	α
1.	25	0	0.165	1.28
2.	35	0	0.270	1
3.	45	0	0.324	1.3
4.	25	0.25	0.246	1.03
5.	35	0.25	0.343	1.17
6.	45	0.25	0.527	1.04
7.	25	0.70	0.29	1.3
8.	35	0.70	0.3	1.27
9.	45	0.70	0.479	1.2

The order of the reaction with respect to $NaBH_4$ concentration α equals to 1 with an experimental error of ± 0.2 (Table 2). Plot of inverse of hydrogen generation rate ($1/r_{H_2}$) *versus* the ratio of NaOH and sodium borohydride concentration (C_{NaOH}/C_{NaBH_4}) gives a straight line, as shown in Fig. 5. The intercept on the *y* axis gives ($1/kC_{NaBH_4}$) and the slope gives (k_1/k), from which both k and k_1 may be determined as shown in Table 3.

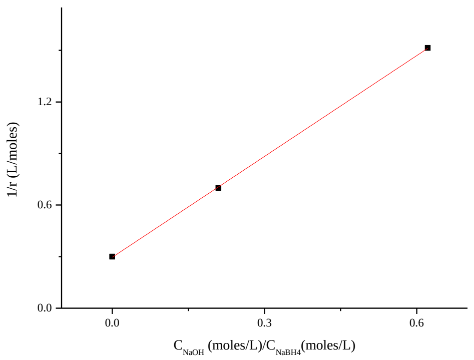

Fig. 5. Effect of NaOH/ $NaBH_4$ ratio on the rate.

Table 3. Rate constants k and k_1

Sample No.	$NaBH_4$ (moles/L)	Temperature (°C)	k	k_1
1.	1.14	25	0.604	3.03
2.	1.14	45	2.009	5.843
3.	1.14	65	3.02	5.88
4.	1.38	25	0.875	2.85
5.	1.38	45	2.09	3.76
6.	1.38	65	3.81	8.11
7.	1.61	25	0.76	1.13
8.	1.61	45	1.94	2.96
9.	1.61	65	4.31	6.20

CHARACTERIZATION OF THE RESIDUAL SUBSTANCE

The morphology of the black colored solid residue formed after hydrolysis reaction of $NaBH_4$ in presence of $CoCl_2$ catalyst was analyzed by SEM and the images are shown in Fig. 6 (a, b, c and d). The particles are observed to agglomerate together and the rough surface indicates the presence of cobalt [10].

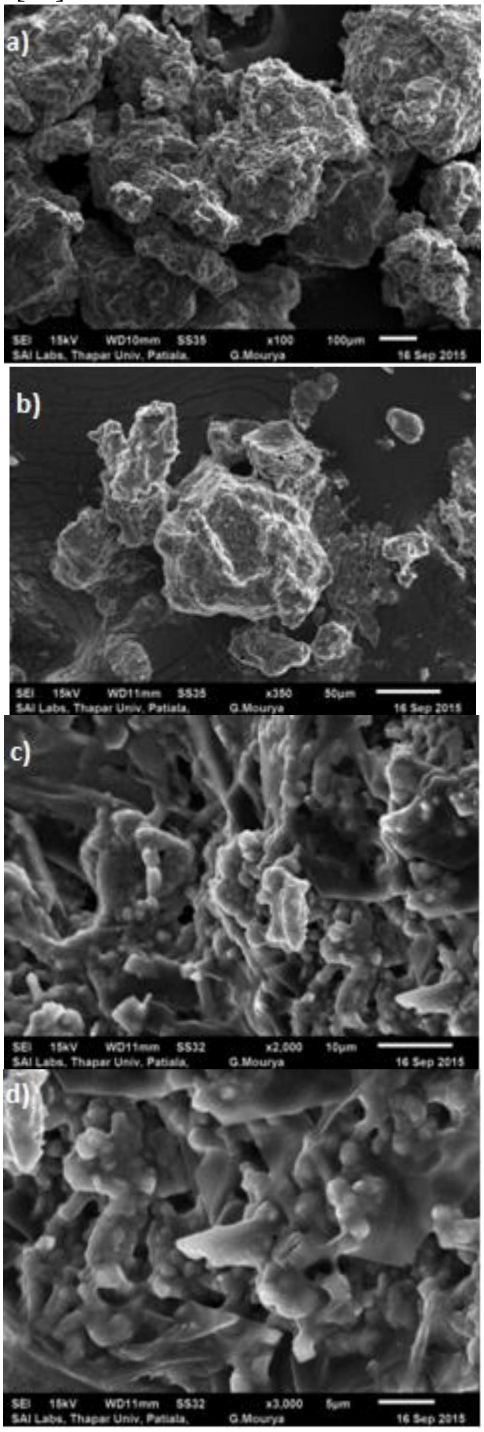

Fig. 6. (a, b, c and d) SEM images of the residual material.

EDS analysis of residual material's surface (Fig.7) provides quantitative information about its elemental composition. Elemental percentages of byproducts of $NaBH_4$ hydrolysis are given in Table 4.

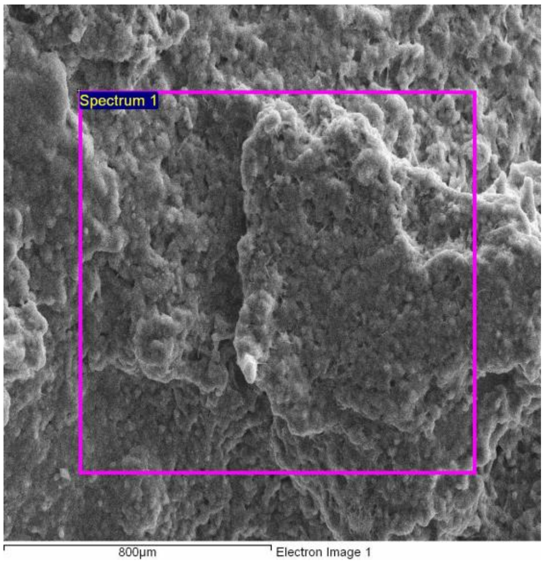

Fig.7. Sectioned area of residual material for EDS analysis

It is observed that boron (78.46 wt%) is present in maximum percentage in the residue followed by oxygen (18.01 wt%), sodium (3.41 wt%), chlorine (0.07 wt%) and cobalt (0.05 wt%)

Table 4. Elemental percentages of $NaBH_4$ hydrolysis byproducts.

Element	Weight (%)	Atomic (%)
B	78.46	85.04
O	18.01	13.19
Na	3.41	1.74
Cl	0.07	0.02
Co	0.05	0.01

According to literature, cobalt boride (Co_2B, Co_3B) and other Co-B compounds with different degrees of oxidation are expected to be present in the residue obtained after hydrolysis of $NaBH_4$ in presence of $CoCl_2$ catalyst. These compounds are reported to be an amorphous mixture of Co-B based catalysts having varying cobalt, boron and oxygen contents [23]. The diffraction peak around 2θ range between 40° and 50° is assigned to cobalt borides (Co_xB) or metallic Co° phases [8, 24]. Similar results are observed by XRD analysis (Fig. 8) of the residual material. Diffraction peaks around 2θ = 45° confirm the presence of Co_2B (Ref. Code 01-089-1994) [14]. Another Co-B based compound like cobalt borate $Co_2B_2O_5$ (Ref. Code 01-073-1772 and Na based byproducts like sodium borate hydrate oxide ($Na_2B_4O_7$. $10H_2O$, Ref Code 01-088-1411 and sodium boride (Na_3B_2O, Ref. Code 00-012-0) are also present in the residue.

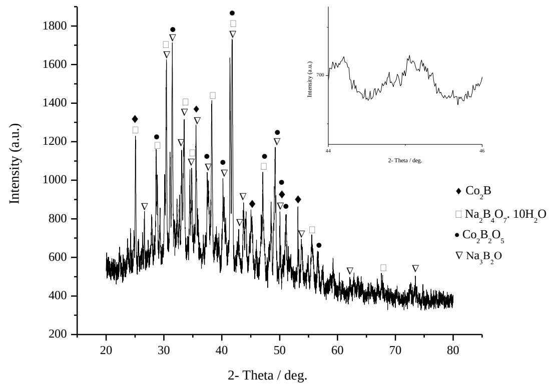

Fig.8. XRD analysis of the residual material

Hydrogen generation rate (HGR) studies using Al_2O_3 (nanoparticles) in the $NaBH_4/CoCl_2/H_2O$ system

Experiments were performed to study the effect on HGR of the addition of Al_2O_3 (nanoparticles) to the $NaBH_4/CoCl_2$ system. As shown in Fig. 9, HGR increases with $NaBH_4$ concentration at all concentrations of Al_2O_3. Readings are taken for 1.008, 1.26, 1.49 and 1.73 moles/L $NaBH_4$ at 0.046, 0.065 and 0.083 moles/L Al_2O_3 for constant $CoCl_2$ (0.02 moles/L) concentration. The addition of Al_2O_3 nanoparticles is found to improve the kinetics of $NaBH_4$ hydrolysis [22].

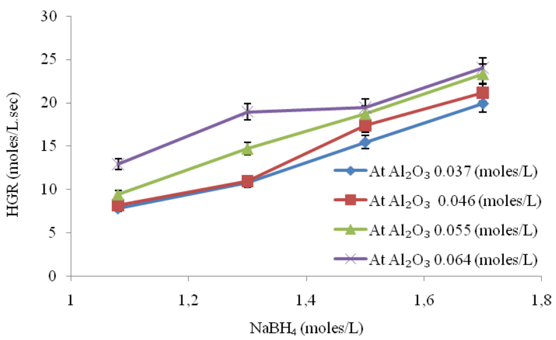

Fig. 9. Effect of $NaBH_4$ concentration on HGR.

The addition of alumina nanoparticles to the $NaBH_4/CoCl_2/H_2O$ system results in high HGR thus improving the system storage density. Due to the supporting mechanism existing between $NaBH_4$ hydrolysis reaction and Al_2O_3/H_2O reaction, hydrogen generation performance is increased [17]. Studies are still in progress to understand the actual role of alumina nanoparticles for the increased performance characteristics of the examined system.

CONCLUSIONS

Hydrolysis reaction of sodium borohydride with cobalt chloride as a catalyst is a first-order reaction. Hydrogen generation rate increases with the increase in temperature, sodium borohydride ($NaBH_4$) concentration and decreases with sodium hydroxide (NaOH) concentration.

The rate constant 'k' with respect to sodium borohydride and the rate constant 'k_1' with respect to sodium hydroxide increase significantly when the temperature is increased from 45 to 65°C.

The gas chromatography analysis indicates that the hydrogen gas has a purity of 85%, the rest being nitrogen.

The residue of the $NaBH_4$ hydrolysis reaction is characterized by SEM, EDS and XRD.

According to SEM and EDS analysis, the particles agglomerate together with maximum percentage of boron in the residue. XRD analysis shows the presence of Co-B and Na based compounds in the residue.

The hydrogen generation rates are found to be higher for the hydrolysis in the $Al_2O_3/NaBH_4/CoCl_2/H_2O$ system compared to the $NaBH_4/CoCl_2/H_2O$ system.

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КИНЕТИЧНИ ИЗСЛЕДВАНИЯ ВЪРХУ СИСТЕМАТА ЗА СЪХРАНЕНИЕ НА ВОДОРОД $NaBH_4/H_2O$ С $CoCl_2$ КАТО КАТАЛИЗАТОР

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(Резюме)

Водородът се приема в световен мащаб като заместител на изкопаемите горива и да установи чисто и сигурно енергийно бъдеще. Съхранението на водорода като хидриди (например натриев борохидрид ($NaBH_4$) е срещнало сериозна загриженост като потенциален източник на водород за портативни устройства. Настоящата работа засяга кинетични изследвания върху системата $NaBH_4/CoCl_2/H_2O$ и факторите, влияещи на скоростта на генериране на водород (HGR): концентрацията на $NaBH_4$, на NaOH и температурата. В нея също се съобщава за резултатите за добавянето нано-частици от диалуминиев триоксид към системата $NaBH_4/CoCl_2/H_2O$, за ускоряването на скоростта на генериране на водород, дължащи се на по-добрата реактивоспособност на нано-частиците. Кобалтовият хлорид е широко използван като изгоден катализатор с неблагороден метал, който ускорява хидролизата на $NaBH_4$. Кинетичните изследвания показват, че хидролизата на $NaBH_4$ се описва от уравнение от първи порядък. Скоростта нараства с натриевия борохидрид и намалява с натриевата основа. Освен това е наблюдавано че скоростта на реакцията нараства с температурата. Охарактеризирането на остатъка е извършено чрез SEM, EDS и XRD, при което се установява, че частиците се агломерират. В остатъка се съдържат бор, натрий, кобалт, хлор и кислород в тегловни проценти съответно 78.46%, 3.41%, 0.05%, 0.07% и 18.01%. Дифракционният пик при $2\theta = 45^{o}$, установен от рентгеноструктурния анализ показва наличието на Co_2B в остатъка.