



# Development of Novel Hydrogen-Based Power Systems for ITS Applications: Phase-I

Final Report

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16. Abstract (Limit: 250 words) There are many remote traffic signals on the road that don't have access to a regular power supply, so they use batteries that need to be changed quite often. A hydrogen fuel cell is an electrochemical device that combines hydrogen and oxygen to produce electricity. It offers a clean and high-efficiency energy source to circumvent the problems associated with the conventional batteries. However, one major drawback that limits its utility is the use of compressed metal cylinders as a source of hydrogen. Chemical-based hydrogen production can provide a very compact and low-pressure storage option for the controlled release of hydrogen gas in large amounts. The hydrogen-based fuel cells can also be used as a backup power source at critical traffic signals to prevent accidents during power outages. The other possible applications include alternating-traffic signs, directional signals, speed-limit signs, blinkers in series, and warning blinkers etc. The project deals with the development of novel chemical-based hydrogen storage materials, efficient generation of hydrogen and recycling of spent materials. In this regard, we have explored several boron-based chemical hydrides as hydrogen storage materials. These hydrides offer an attractive solution to our quest in finding out materials that are safe, compact, and readily provide large quantities of hydrogen on demand. In this work, we have carried out generation of hydrogen from several boron hydrides such as sodium borohydride (SBH) and ammonia-borane (AB), etc., utilizing solvents such as water and alcohols under catalytic and non-catalytic conditions.			
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## Executive Summary

There are many remote traffic signals on the road that don't have access to a regular power supply, so they use batteries that need to be changed quite often. A hydrogen fuel cell is an electrochemical device that combines hydrogen and oxygen to produce electricity. It offers a clean and high-efficiency energy source to circumvent the problems associated with the conventional batteries. However, one major drawback that limits its utility is the use of compressed metal cylinders as a source of hydrogen. Chemical-based hydrogen production can provide a very compact and low-pressure storage option for the controlled release of hydrogen gas in large amounts. The hydrogen-based fuel cells can also be used as a backup power source at critical traffic signals to prevent accidents during power outages. The other possible applications include alternating-traffic signs, directional signals, speed-limit signs, blinkers in series, and warning blinkers, etc.

The current project deals with the development of methodologies for chemical hydrogen storage materials from borohydrides and boranes, and efficient generation of hydrogen from these materials. We have carried out a study on the evolution of hydrogen from sodium borohydride and ammonia borane.

Sodium borohydride is air stable, generally non-toxic and a readily available material that is produced in millions of pounds each year for various applications. Hydrogen evolution via the hydrolysis of sodium borohydride is generally a slow process and is not practical for commercial applications. However, the rate of evolution of hydrogen is significantly enhanced by the utilization of reactive alcohols such as methanol, ethanol, etc. Bulky alcohols such as t-butyl alcohol release only trace amounts of hydrogen even upon long standing.

Hydrogen evolution from sodium borohydride could also be significantly enhanced by the utilization of protic or Lewis acid catalysts. We have carried out a study utilizing several protic acids such as vinegar and tartaric acid, and Lewis acids such as iron chloride, ruthenium chloride, cobalt chloride, nickel chloride, etc. The rate of hydrogen evolution in each of these cases was observed to be almost instantaneous. We have also carried out a study on the rate of hydrogen evolution at lower temperatures keeping in mind the colder climates in Minnesota. Our studies revealed that steady pace of hydrogen evolution could be achieved using protic acids in aqueous ethylene glycol even at  $-20^{\circ}\text{C}$  to  $-40^{\circ}\text{C}$ .

Ammonia borane is also a good chemical hydrogen storage material with a high density of hydrogen content (~19.6% by weight). Although this chemical is relatively expensive, it can be very readily synthesized from sodium borohydride using ammonium salts of mineral acids. Ammonia borane releases hydrogen upon thermal dehydrogenation to provide boron nitrides as the final product. However, this process requires significantly high energy input ( $>300^{\circ}\text{C}$ ) and the regeneration of ammonia borane from the spent fuel (boron nitride) is an extremely arduous endeavor. Hence, we adopted hydrolysis and alcoholysis of ammonia borane for the generation of hydrogen. As these reactions are extremely slow in the absence of acid catalysts, we utilized transition metal catalysis such as ruthenium chloride, cobalt chloride, etc. for the facile generation of hydrogen from ammonia borane in water, methanol, and ethanol.

Phase II of this project involves the utilization of these hydrogen storage materials and evolution methodologies for fuel cell-based prototype power systems for intelligent transportation systems (ITS) and other related applications.

## Chapter 1 Introduction

In the current hydrocarbon economy, all transportation needs are fueled primarily by petroleum-based products such as gasoline, diesel, and natural gas. As we all know, the burning of these fuels causes the emission of greenhouse gases and several other pollutants. Other problems include the limited supply of hydrocarbon resources, and the ever increasing demand for fuels all over the world. Hydrogen-based energy appears as a clear and clean alternative to the problems associated with hydrocarbon-based fossil fuels in transportation and other energy related applications.

However, the main drawback with this system involves the greenhouse gas  $\text{CO}_2$  as a by-product and the non-recyclability of the spent materials. In this regard, hydrogen fuel cells offer distinct advantages in terms of water as the by-product. Although fuel cell technology has been well developed and has been in use for the past fifty years, the technologies involving hydrogen storage are very scarce and economically non-viable for routine and commercial applications. The conventional hydrogen fuel cells employ hydrogen gas stored in metal cylinders. Such systems suffer from the disadvantages namely: limited volume that requires frequent change of cylinders, and production of limited electricity. For example, a 26 x 140 cm cylinder with a tare weight of 63 kgs holds about 49L of hydrogen, which would produce ~45 W-h of electricity. Based on these calculations, a 15 W-h traffic light would run for ~3 hours with one cylinder of hydrogen.

Hydrogen storage in liquid state has high storage capacity, but it requires a lot of energy in liquation and low temperature keeping, therefore, the energy utilization efficiency is low. This protocol is highly expensive and is used in space related energy applications.

The US Department of Energy's minimum target by 2010 for hydrogen storage materials include: gravimetric density of 6 kg  $\text{H}_2$  in a 100 kg tank and volumetric density of 45 g  $\text{H}_2/\text{L}$ . By 2015 these targets will rise to 9 wt% for gravimetric density and 81 g  $\text{H}_2/\text{L}$  for volumetric density. Thus there is an urgent need for lightweight hydrogen storage materials (>10 wt%  $\text{H}_2$ ).

United States has the world's largest reserves of borax, and boron-based hydrides can be prepared from this ore. Chemical hydrides especially boron-based hydrides offer an attractive solution to our quest in finding out materials that are safe, compact, and readily provide large quantities of hydrogen on demand and spent materials that could be readily recycled [1]. The most notable boron hydrides that are actively being pursued are sodium borohydride (SBH) and ammonia-borane (AB). However, studies have shown several limitations in terms of efficiency in hydrogen generation and recycling of the spent materials. Hence there is a need to develop new materials that are easy to prepare, readily generate hydrogen in a controlled way and efficiently recycled back to complete the cycle for fuel cell.



## Chapter 2 Sodium Borohydride for Hydrogen Generation

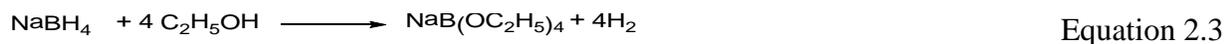
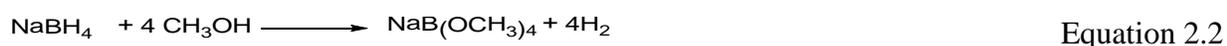
Sodium borohydride is nontoxic and nonflammable and is available in large supplies; in fact, the United States has the world's largest reserves of borax, an ore from which sodium borohydride could be readily prepared. The main benefits of using this compound as hydrogen storage material include the fact that it does not contribute to the greenhouse gas buildup; and/or smog-forming compounds [2,3].

### Generation of Hydrogen from Sodium Borohydride (NaBH<sub>4</sub>)

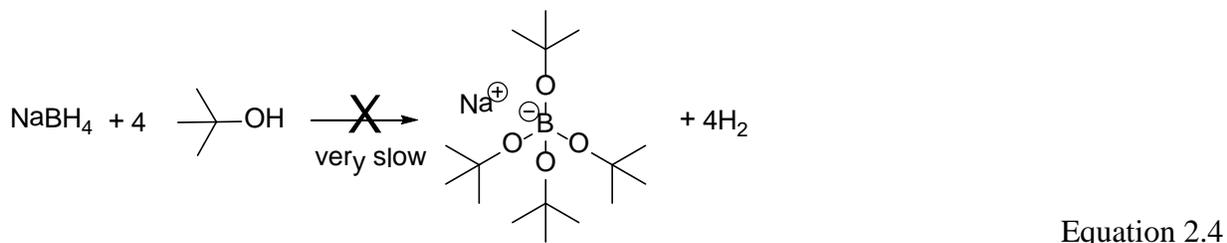
The reaction of sodium borohydride with water [4,5] led to the slow release of hydrogen gas along with sodium borate as shown in equation 2.1.



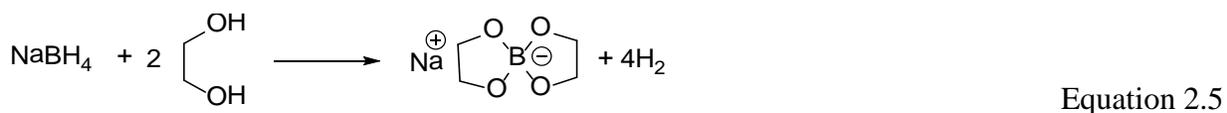
Similarly, reactive alcohols such as methanol and ethanol [6] also reacted with sodium borohydride to release quantitative amounts of hydrogen and sodium tetramethoxyborate and sodium tetraethoxyborate respectively (equations 2.2 and 2.3). The reaction in methanol took place in 15 minutes while the corresponding reaction with ethanol took almost 30 minutes.

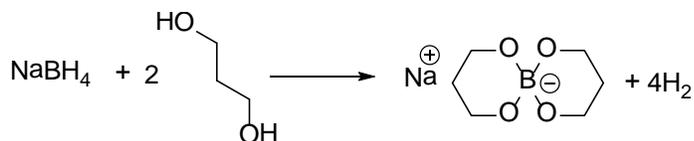


However, the reaction with bulky alcohol such as 2-methyl-2-propanol (t-butyl alcohol) was quite slow and even after 24 hours appreciable quantities of hydrogen gas was not released (Equation 2.4).



Reaction of sodium borohydride with ethylene glycol and propylene glycol also took place in a convenient manner to release quantitative amounts of hydrogen gas and the corresponding borate complexes (equations 2.5 and 2.6). All of the above results have been summarized in Table 2.1 (Appendix A).

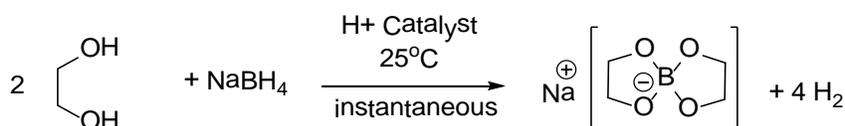




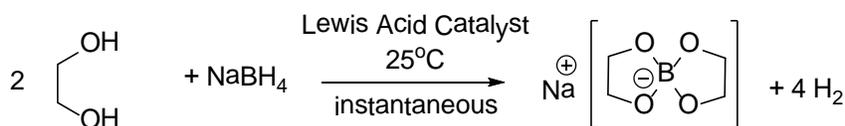
Equation 2.6

### Generation of Hydrogen from Sodium Borohydride Under Catalytic Conditions

The reaction of sodium borohydride with water and alcohols was also carried out in the presence of protic as well as Lewis acid catalysts. The protic acids used include vinegar (acetic acid), tartaric acid, and hydrochloric acid (Equation 2.7). The Lewis acid catalysts used include ruthenium (III) chloride, cobalt (II) chloride, nickel (II) chloride, and iron (III) chloride (Equation 8). We chose ethylene glycol as the representative alcohol for this study. In each of these cases, quantitative amount of hydrogen gas was released almost instantaneously. The results are summarized in Table 2.2 (Appendix A).



Equation 2.7



Equation 2.8

In cold climates such as those in Minnesota, the efficient release of hydrogen gas at low temperatures is also crucial. Hence, we conducted the study of hydrogen evolution in the presence of catalysts at low temperatures as well. For this purpose, we used a 1:1 solution of ethylene glycol and water as this mixture can tolerate temperatures as low as  $-50^\circ\text{C}$  without freezing. Again, in the presence of protic and Lewis acid catalysts, we observed fairly rapid evolution of hydrogen gas even at  $-40^\circ\text{C}$ .

The yield of hydrogen was observed to be  $>90\%$  for the temperature range between  $25^\circ\text{C}$  to  $-20^\circ\text{C}$  and at  $-40^\circ\text{C}$  the yield was observed to be  $\sim 80\%$ . A few more experiments have been performed using pump oil to make a slurry of sodium borohydride. The reactions have a yield of greater than  $90\%$ , but the reaction seizes at temperatures below  $-20^\circ\text{C}$  as the oil gets viscous with the decreasing temperature.

The detailed temperature dependent hydrogen evolution studies using sodium borohydride and ethylene glycol in the presence of vinegar as the protic acid have been reported in Appendix A [Table 2.3 (study performed at  $25^\circ\text{C}$ ), Table 2.4 (study performed at  $0^\circ\text{C}$ ), Table 2.5 (study performed at  $-10^\circ\text{C}$ ), Table 2.6 (study performed at  $-20^\circ\text{C}$ ), and Table 2.7 (study performed at  $-40^\circ\text{C}$ )].

Further details of these experiments have also been reported in Appendix B [Figure 2.1 (study performed at  $25^\circ\text{C}$ ), Figure 2.2 (study performed at  $0^\circ\text{C}$ ), Figure 2.3 (study performed at  $-10^\circ\text{C}$ ), Figure 2.4 (study performed at  $-20^\circ\text{C}$ ), and Figure 2.5 (study performed at  $-40^\circ\text{C}$ )].

## **Conclusions**

In this project, we have carried out a systematic study of the hydrolysis and alcoholysis of sodium borohydride under catalytic and non-catalytic conditions. Both Lewis acid and protic acid catalysts have been identified to work very well for the efficient generation of hydrogen from sodium borohydride. We have identified the combination of ethylene glycol and water as an ideal mixture for controlled release of hydrogen at low temperatures (as low as  $-40^{\circ}\text{C}$ ) in the presence of vinegar as the catalyst. From this study, we identified the combination of sodium borohydride, ethylene glycol and vinegar as possible chemical hydrogen storage material for fuel cell-based intelligent transportation systems (ITS) applications.



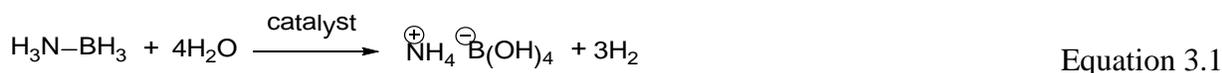
## Chapter 3 Ammonia Borane for Hydrogen Generation

Due to the high content of hydrogen (19.6% by weight), ammonia-borane is one of the most actively pursued chemicals for hydrogen storage [7]. Unlike sodium borohydride, ammonia-borane could be used for the generation of hydrogen under thermal conditions and hence is an attractive source of hydrogen. However, the use of ammonia-borane for these purposes is severely limited because of the high temperatures (>300°C) that are required for the evolution of hydrogen as well as the harsh conditions required for the regeneration of spent fuel [8]. We studied and optimized the conditions for the controlled release of hydrogen from ammonia-borane under mild hydrolytic and alcoholic conditions using water and alcohols respectively [9].

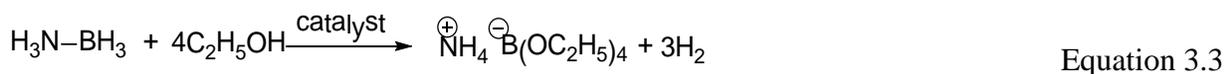
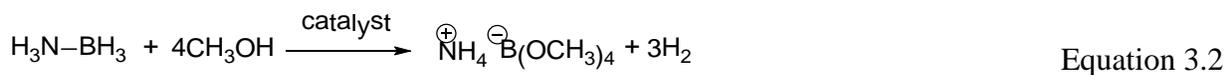
### Hydrogen Generation from Ammonia-Borane (AB) via Hydrolysis and Alcoholysis under Catalytic Conditions

Although 3 moles of hydrogen gas could be released from each mole of ammonia borane, the release of third equivalent of hydrogen requires high heat (>300°C) and hence is not practical. Moreover, the product obtained as a result of complete removal of hydrogen from ammonia borane is boron nitride, a highly stable compound from which the regeneration of ammonia borane is an extremely arduous process. To circumvent both these problems, as mentioned above, we carried out the hydrolysis or alcoholysis of ammonia borane.

Hydrolysis of ammonia borane with water in the presence of Lewis acid catalysts such as ruthenium (III) chloride, nickel (II) chloride, iron (III) chloride, cobalt (II) chloride, etc. proved very facile and >95% of hydrogen evolution was observed at vigorous rate (Equation 3.1).



Similarly, alcoholysis with methanol and ethanol also proved to be equally fast under these catalytic conditions to obtain essentially quantitative yields of hydrogen gas along with ammonium tetramethylborate and ammonium tetraethyl borate respectively (Equations 3.2 and 3.3). Further details about these reactions could be found in Appendix A (Table 3.1)



### Conclusions

In conclusion, we have studied the generation of hydrogen from ammonia borane under hydrolysis and alcoholysis conditions in the presence of transition metal catalysts. In the absence of the catalysts, hydrogen evolution proved to be too slow to be of any practical use. The above methodologies offer an easy entry in to the preparation of hydrogen storage materials, generation of hydrogen on demand and importantly they also provide a strategy to regenerate the spent materials.



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## **Appendix A.**

### **Generation of Hydrogen from Sodium Borohydride and Ammonia Borane**

**Table 2.1: Reaction of sodium borohydride with alcohols under non-catalytic conditions**

#	Alcohol	Temperature	Time	Hydrogen evolved
1	Water	25°C	24 hours	~50%
2	Methanol	25°C	15 minutes	>95%
3	Ethanol	25°C	30 minutes	>95%
4	2-Methyl-2-propanol	25°C	24 hours	Trace
5	Ethylene glycol	25°C	15 minutes	>95%
6	Propylene glycol	25°C	15 minutes	>95%

**Table 2.2: Reaction of sodium borohydride with ethylene glycol in the presence of acid catalysts**

#	Acid Catalyst	Temperature	Hydrogen evolved
1	Ruthenium (III) Chloride	25°C	>95%
2	Cobalt (II) Chloride	25°C	>95%
3	Nickel (II) Chloride	25°C	>95%
4	Iron (III) Chloride	25°C	>95%
5	Vinegar (Acetic Acid)	25°C	>95%
6	Tartaric Acid	25°C	>95%
7	Hydrochloric Acid	25°C	>95%
8	Aluminum foil	25°C	>95%
9	Silica Gel	25°C	>95%
10	Copper metal	25°C	>95%

**Table 2.3: Reaction of sodium borohydride with vinegar dissolved in ethylene glycol/water mixture at room temperature (25°C)**

#	Volume of Hydrogen gas evolved (mL)	Time
1	133.0	1.0
2	164.0	2.0
3	164.0	3.0

**Table 2.4: Reaction of sodium borohydride with vinegar dissolved in ethylene glycol/water mixture at 0°C.**

#	Volume of Hydrogen gas evolved (mL)	Time
1	99.0	1.0
2	157.0	2.0
3	180.0	3.0
4	183.0	4.0
5	183.0	5.0

**Table 2.5: Reaction of sodium borohydride with vinegar dissolved in ethylene glycol/water mixture at -10°C.**

#	Volume of Hydrogen gas evolved (mL)	Time
1	94.0	1.0
2	154.0	2.0
3	193.0	3.0
4	201.0	4.0
5	204.0	5.0
6	207.0	6.0

**Table 2.6: Reaction of sodium borohydride with vinegar dissolved in ethylene glycol/water mixture at -20°C.**

#	Volume of Hydrogen gas evolved (mL)	Time
1	110.0	1.0
2	155.0	2.0
3	183.0	3.0
4	190.0	4.0
5	201.0	5.0
6	205.0	6.0
7	215.0	7.0

**Table 2.7: Reaction of sodium borohydride with vinegar dissolved in ethylene glycol/water mixture at -40°C.**

#	Volume of Hydrogen gas evolved (mL)	Time
1	76.0	1.0
2	120.0	2.0
3	140.0	3.0
4	142.0	4.0
5	150.0	5.0
6	153.0	6.0
7	155.0	7.0
8	158.0	8.0
9	160.0	9.0

**Table 3.1: Reaction of ammonia-borane with water and alcohols in the presence of acid catalysts**

#	Acid Catalyst	Proton source	Temperature	Hydrogen evolved
1	Ruthenium (III) Chloride	Water	25°C	>95%
2	Cobalt (II) Chloride	“	25°C	>95%
3	Nickel (II) Chloride	“	25°C	>95%
4	Iron (III) Chloride	“	25°C	>95%
5	Ruthenium (III) Chloride	Methanol	25°C	>95%
6	Cobalt (II) Chloride	“	25°C	>95%
7	Nickel (II) Chloride	“	25°C	>95%
8	Iron (III) Chloride	“	25°C	>95%
9	Ruthenium (III) Chloride	Ethanol	25°C	>95%
10	Cobalt (II) Chloride	“	25°C	>95%
11	Nickel (II) Chloride	“	25°C	>95%
12	Iron (III) Chloride	“	25°C	>95%

## **Appendix B.**

### **Reaction of Sodium Borohydride with Alcohols – Temperature Study**

Figure 2.1: Reaction of sodium borohydride with vinegar dissolved in ethylene glycol/water mixture at 25°C.

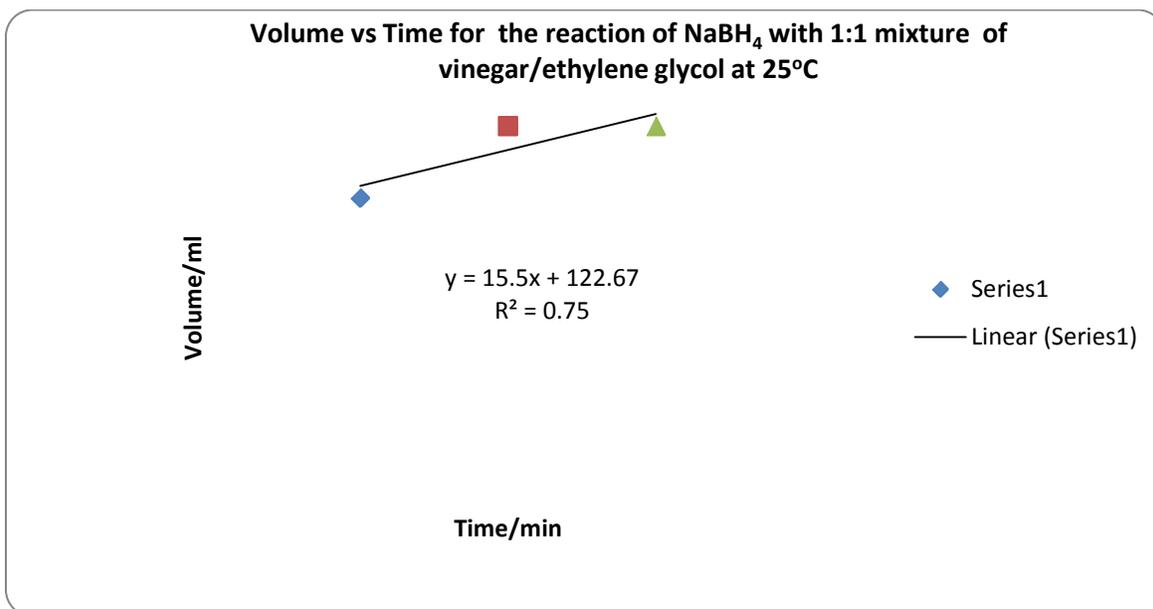
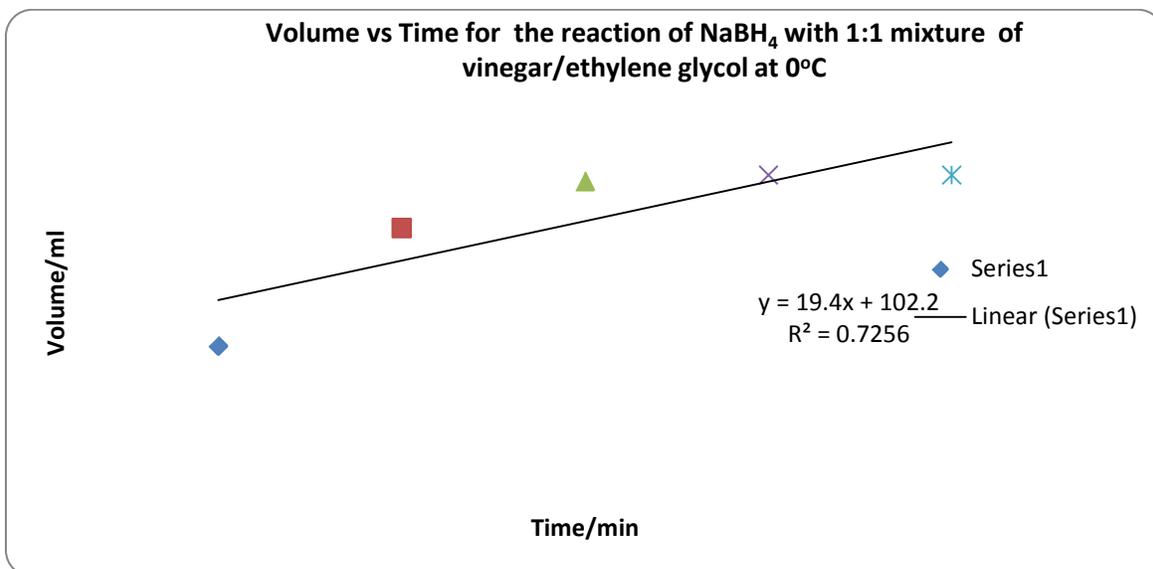
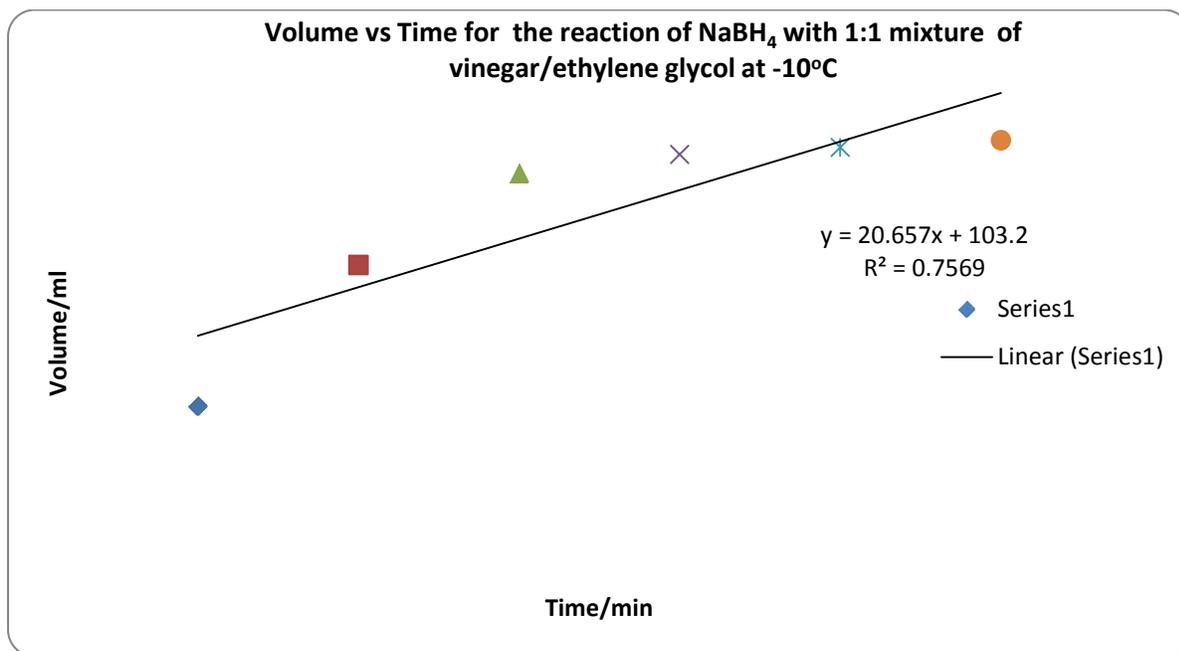


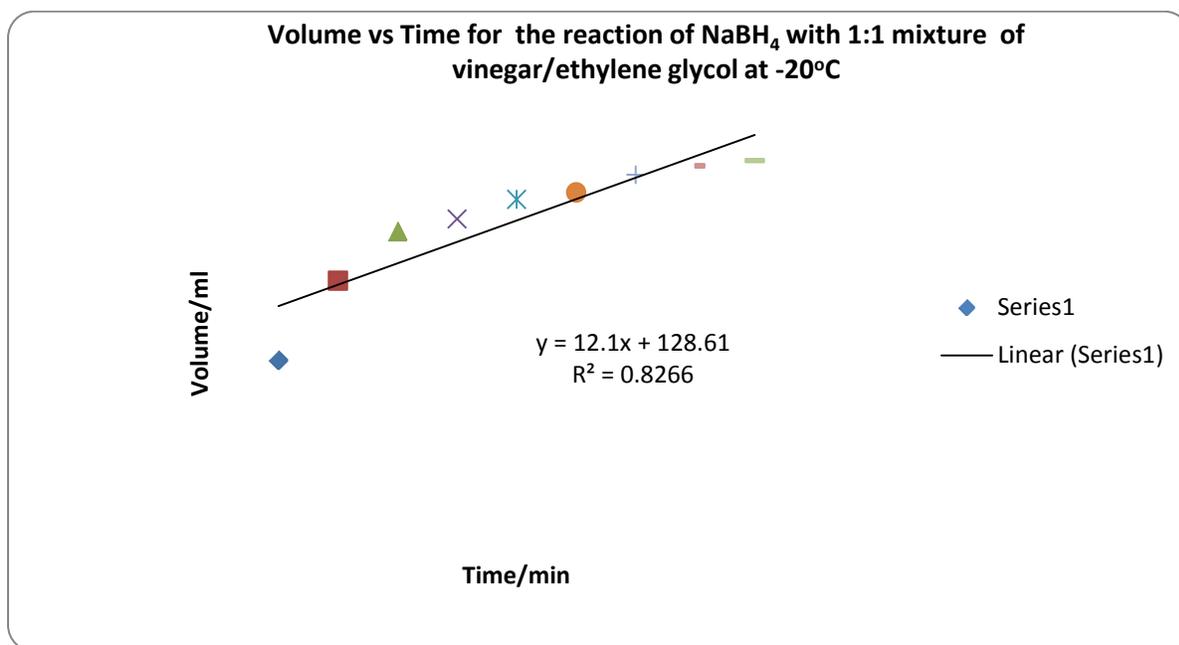
Figure 2.2: Reaction of sodium borohydride with vinegar dissolved in ethylene glycol/water mixture at 0°C.



**Figure 2.3: Reaction of sodium borohydride with vinegar dissolved in ethylene glycol/water mixture at -10°C.**



**Figure 2.4: Reaction of sodium borohydride with vinegar dissolved in ethylene glycol/water mixture at -20°C.**



**Figure 2.5: Reaction of sodium borohydride with vinegar dissolved in ethylene glycol/water mixture at -40°C.**

