Ibrahim Dincer · Adnan Midilli
Haydar Kucuk *Editors*

Progress in Sustainable Energy Technologies: Generating Renewable Energy

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Volume 1

Ibrahim Dincer • Adnan Midilli • Haydar Kucuk
Editors

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Preface

Renewable energy sources are of vital importance for future of humanity due to the issues and problems caused by mainly fossil-based energy systems and applications. Renewable energy sources are treated as sustainable energy sources while fossil fuels are considered unsustainable. This fact has attracted many researchers, scientists, practicing engineers, etc. to work on tirelessly and develop better renewable energy-based technologies for a sustainable future. The study domain is quite diverse, covering many engineering disciplines, such as mechanical, civil, physical, chemical, biotechnology, environmental, industrial, geological, electrical, etc. and non-engineering areas, such as chemistry, biology, physics, mathematics, business, informational technology, economy, medicine, etc.

Everyone agrees that sustainable energy technologies are necessary for solving current and potentially future energy problems and achieve environmentally benign solutions. This volume primarily concerns the largest energy domain under sustainable energy technologies, covering all relevant disciplinary areas, ranging from current problems, projections, new concepts, modeling, experiments and measurements to simulations, and discusses recent research findings on solar energy, wind energy, biomass, geothermal energy, hydro energy, wave energy, hydrogen production, fuel cells, energy storage, heat pump, integrated energy systems, etc.

This volume includes some invited contributions and the selected papers from the 11th International Conference on Sustainable Energy technologies (SET-2012) held in Vancouver, Canada on September 2–5, 2012. The conference had a multidisciplinary nature, covering main areas of sustainable energy technologies, and aimed to provide a forum for researchers, scientists, engineers and practitioners from all over the world to exchange information, to present high-quality research results and new developments in the wide domain covered by sustainable energy technologies, and discussed the future direction and priorities in the field.

In conclusion, the editors of this volume gratefully acknowledge the assistance provided by Dr. Ugur Akbulut in reviewing and revising several chapters, checking for consistency and finalizing them for publication. The editors also register their sincere appreciation to the authors for their contributions which have made this

unique book possible. Furthermore, Dr. Dincer acknowledges the support provided by the Turkish Academy of Sciences in Ankara, Turkey.

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Chapter 30
Hydrogen Production by Reforming Clathrate Hydrates Using the in-Liquid Plasma Method

Andi Erwin Eka Putra, Shinfuku Nomura, Shinobu Mukasa and Hiromichi Toyota

Abstract Clathrate hydrates, which were formed from methane and cyclopentane, were decomposed by plasma at atmospheric pressure. Methane hydrate was synthesized by injecting methane into shaved ice in the reactor at a pressure of 7 MPa and a temperature of 0 °C. In addition, cyclopentane hydrate was formed by adding surfactant into cyclopentane-water emulsion at 0.1 MPa and a temperature of 0 °C. The process of plasma decomposition of clathrate hydrates has been carried out by irradiating high frequency plasma at the tip of the electrode in clathrate hydrates. 2.45 GHz MW oven and 27.12 MHz RF irradiation were used. This study results gas production that its content identified by gas chromatograph. High purity of hydrogen would be extracted from clathrate hydrate using the in-liquid plasma method.

Keywords Hydrogen production · Clathrate hydrates · The in-liquid plasma

30.1 Introduction

Clathrate hydrates are formed by the presence of constituent molecules such as CH_4, CO_2, and even cyclopentane as liquid hydrocarbon within the cavity of lattice water. Clathrate hydrates are formed by the exothermic reaction with a combination of

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pressure and temperature accordingly. Clathrate hydrates, mainly methane hydrate, which are estimated exist stable and abundant in the seabed and permafrost at low temperature and high pressure are the source of untapped energy. Methane hydrate is categorized as cubic structure I (sI) with the ideal composition of $CH_4.5.75H_2O$ has the pressure phase equilibrium of 2.3 MPa at 0 °C with the content of ice—liquid water—hydrate and cyclopentane hydrate as cubic structure II (sII) 7.7 °C at atmospheric pressure [1]. Therefore, cyclopentane hydrate is generally preferred in experimental laboratory to identify the mechanism of hydrate formation in oil production pipelines [2, 3] and it also applied to the thermal energy storage for air conditioning system [4].

Because of the potential for being economically viable, methane hydrate has been exploited for the recovery of natural gas through dissociation process. One of the hydrate dissociation process is the involving of heating hydrate fields through thermal stimulation at above hydrate equilibrium temperature. Thermal stimulation method is typically by injecting hot water (steam and hot brine) into hydrate fields. Unfortunately, this method requires high production costs due to high-energy losses during the injection of hot water. On the other hand, the use of high frequency waves is irradiated directly to hydrate fields can be more rapidly than the hot water injection [5].

The other application of hydrate technologies for the storage and transportation of hydrocarbon fuels, mainly natural gas, has been investigated. This is because large amount of gas so that they can facilitate the transportation of natural gas [6], and hydrate storage and transport processes became feasible at low pressure [7, 8, 9].

The potential energy source mentioned above can support the availability of fuel gas. However, the release of methane and carbon dioxide into the atmosphere should be considered as a major contribution to global warming. Therefore, as the response to the environmental impact of fossil fuels, hydrogen which is currently used extensively in the chemical industry can be optimized as environmentally energy. Generally, the need of commercial hydrogen is produced by steam methane reforming.

The other process that could be applied to hydrogen industry is plasma generation technology because its high temperature of more than 1000 K may accelerate the reaction rate. On the other hand, a technology for generating plasma in liquid by radio frequency (RF) or microwave (MW) irradiation has been conducted to produce 70–80 % of hydrogen at atmospheric pressure from waste oil and n-dodecane [10, 11]. RF irradiation could be easy to generate plasma in the water at high pressure [12] and to produce hydrogen, oxygen, and hydrogen peroxide from water [13].

In this study, high frequency plasma decomposition of clathrate hydrates at atmospheric pressure consist of methane hydrate and cyclopentane hydrate is conducted. This process is a first step towards the ultimate goal to produce hydrogen from hydrate fields with in-liquid plasma method. The in-liquid plasma makes it possible to stimulate plasma into hydrate fields using transmission cable to connect the power generation in the floating board and the electrode in hydrate fields. The in-liquid plasma method is easily generated with a high localized temperature at high pressure where the plasma exist mostly around the tip of the electrode. That's way this method is considered suitable for this purpose.

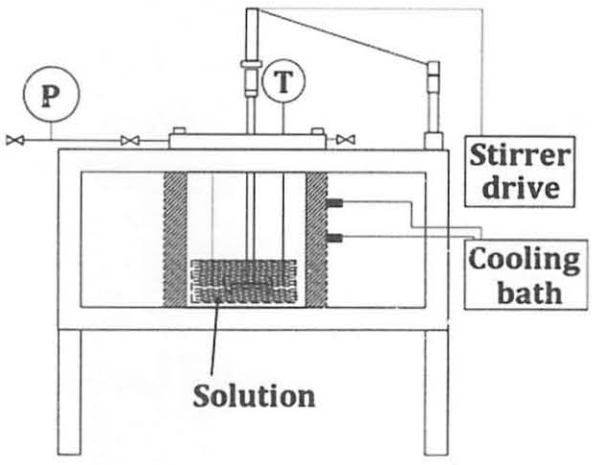

Fig. 30.1 The structure of the clathrate hydrates formation apparatus

30.2 Experimental Procedures

Clathrate Hydrates Formation

Figure 30.1 depicts the experimental equipment used in this research. The volume of the reaction vessel is 400 mL with a height of 140 mm and an inner diameter of 60 mm. The maximum pressure is 15 MPa, and the temperature of the reactor casing is maintained by an ethylene glycol cooling medium. A magnetic stirrer with a diameter of 40 mm and a gas injection tube are positioned 30 mm from the reactor bottom.

Methane hydrate formation has been synthesized by injecting pressurized methane into shaved ice in the formation reactor. one hundred grams of shaved ice were put into the formation reactor that had been cleaned with water. The air in the reactor was then purged by decompression using an aspirator and substituted with methane. The temperature of the cooling bath was kept constant at 0 °C, the methane was pressurized to about 7 MPa and the stirrer was activated to agitate the solution at 400 rpm. The temperature of clathrate hydrates formation was monitored by a thermocouple placed at at 30 mm from the reactor bottom. The temperature and pressure during the process was recorded every 15 minutes.

In addition, 33.5 gm of cyclopentane were dissolved into 146.5 gm of water with 0.2 gm of surfactant. The solution composition is $C_5H_{10}.17H_2O$ and it categorized as cubic structure II (sII). Then, cyclopentane (CP) hydrate was injected into the reactor at atmospheric pressure and was cooled by an ethylene glycol cooling medium at 2 °C. The solution was stirred at 120 rpm during the formation. Hydrate crystal grains are added to the solution when its temperature reaches about 2 °C in order to trigger the formation of hydrates.

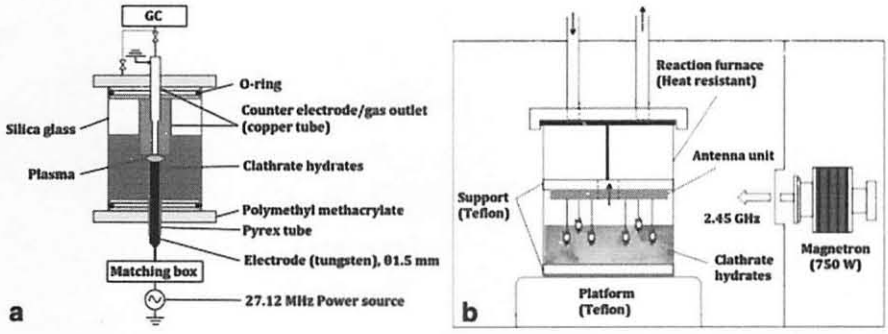

Fig. 30.2 Experimental apparatus for decomposition of clathrate hydrates

Plasma Decomposition of Clathrate Hydrates

27.12 MHz radio frequency plasma and 2.45 GHz MW plasma were used to decompose clathrate hydrates, as shown in Fig. 30.2. Before plasma generated at the tip of the electrode decomposed clathrate hydrates in the reactor vessel at atmospheric pressure, Helium was injected into the reactor vessel to expel air.

27.12 MHz Radio Frequency Plasma Decomposition

A transparent silica glass pipe was used as the reactor vessel with an inner diameter of 55 mm, a thickness of 2 mm, and a height of 85 mm. An electrode consisting of 2 mm of a tungsten rod protruding from a silica glass tube with an outer diameter of 6 mm and a thickness of 1.5 mm as dielectric substance was inserted from the bottom of the reactor and connected to a 27.12 MHz RF power source (T161–5766LQ, Thamway) via a matching box (T020–5766M, Thamway). Additionally, a copper tube was inserted from the top of the reactor to a distance of 4 mm from the lower electrode that served not only as a counter electrode but also as gas outlet as well. Thirty grams of clathrate hydrates were poured into the reactor vessel. The generation of RF plasma in clathrate hydrates was conducted at 150 W at atmospheric pressure. The power values were calculated by subtraction of the reflected power from the forward power. The reflected power was kept constant at the lowest possible level.

2.45 GHz MW Plasma Decomposition of Clathrate Hydrates

The microwave oven was used as a MW source to generate the in-liquid plasma. Fifty grams of clathrate hydrates was placed below the tip of the antennas in the reactor vessel. The reactor vessel was irradiated by microwaves from the magnetron received by antennas. Then, plasma is generated at the tip of antennas.

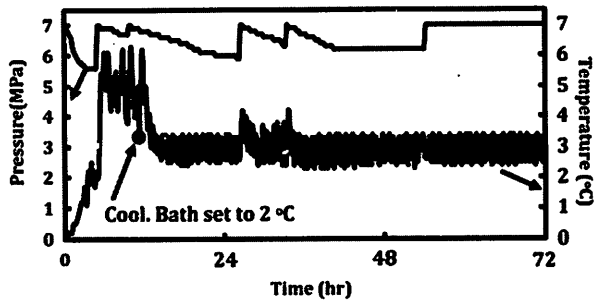

Fig. 30.3 Pressure and temperature along methane hydrate formation. (adapted from [15])

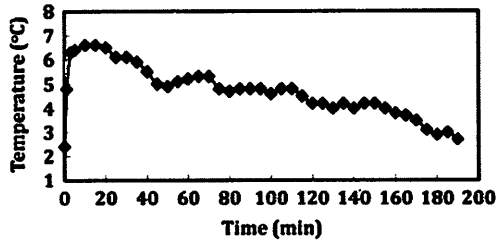

Fig. 30.4 The cyclopentane hydrate formation at atmospheric pressure. (adapted from [16])

A gas chromatograph (Shimadzu 8 A) with a column temperature 60 °C (6 min hold) to 160 °C and Helium as the carrier gas was used to identify the contents of product gases.

30.3 Results and Discussion

Methane hydrate began to be formed immediately after the stirrer was activated at the beginning of the process, which was characterized by the pressure drop to approximately 5.6 MPa and an increase in temperature to approximately 2 °C. This was due to an exothermic reaction, as shown in Fig. 30.3. The methane was pressurized to 7 MPa with a corresponding increase in temperature to approximately 6 °C. The formation of methane hydrate then occurred continuously from the melting ice. The remaining ice was melted to form hydrates by a change in the temperature of cooling bath to 2 °C after the stirrer was stopped. Pressurization with methane to 7 MPa was performed in several times. After the methane hydrate formation process was complete, when the formation pressure was constant at 7 MPa, it was reduced rapidly to atmospheric pressure. The further cooling of the hydrate is required to prolong the completion of hydrate dissociation [14].

Figure 30.4 shows temperature along cyclopentane hydrate formation at atmospheric pressure. The adding of the hydrate crystal grains triggers the hydrate formation that is characterized by an increase in the solution temperature to about 7 °C. The cyclopentane hydrate formation is complete when the solution temperature remained constant at about 2 °C.

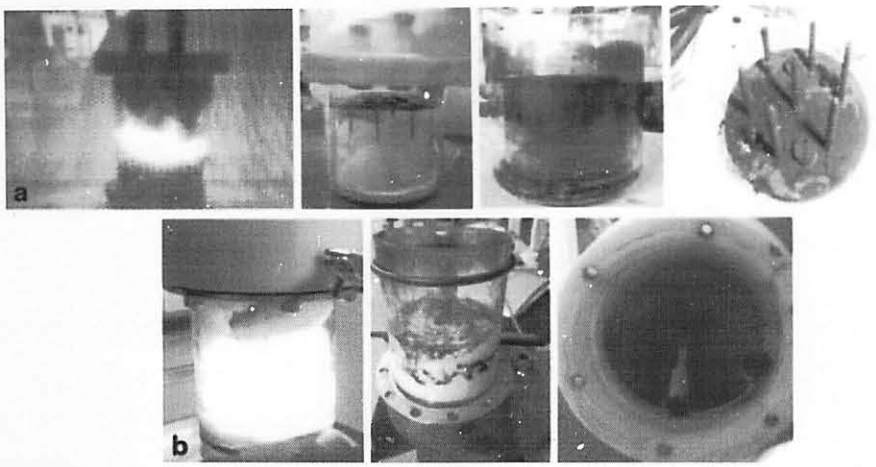

Fig. 30.5 Photo of the high frequency plasma decomposition of clathrate hydrates. **a** 2.45 GHz MW plasma decomposition. **b** 27.12 MHz RF plasma decomposition

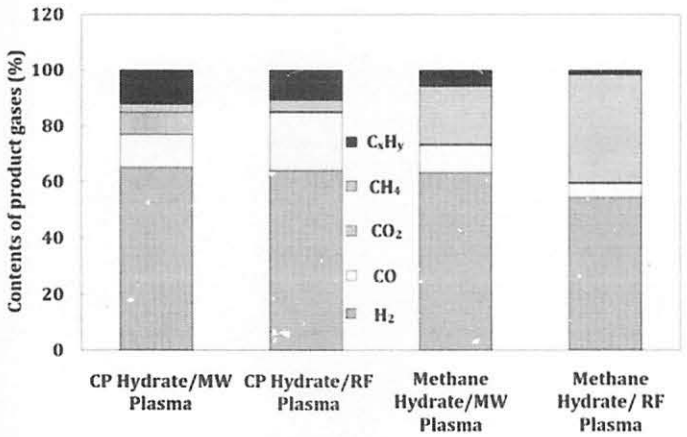

Fig. 30.6 Content of product gases from clathrate hydrate decomposition. C_xH_y consists of C_2H_2, C_2H_4

Clathrate hydrates decomposed by the plasma into the product gases. The product gases are hydrogen (H_2) and carbon monoxide (CO) as the main products, and byproducts are carbon dioxide, methane, carbon (C), and C_2-hydrocarbon. Carbon was found adhered in the reactor walls, the antennas/counter electrodes, and in the remnant of solution as shown in Fig. 30.5.

At plasma decomposition of methane hydrate, the significant methane content in the product gases was identified as the unconverted methane release. It was found about 21% in the MW plasma and 39% in the RF plasma as shown in Fig. 30.6.

Table 30.1 Basic reactions for clathrate hydrates

Reactions	DH (kJ/mol)
Methane hydrate	
$CH_4 \cdot 6H_2O \rightarrow CH_4(g) + 6H_2O$	53.5
$CH_4 + H_2O \rightarrow 3H_2 + CO$	206.16
$CO + H_2O \rightarrow H_2 + CO_2$	-41.2
Direct decomposition	
$CH_4 \rightarrow 2H_2 + C$ (s)	74.87
Cyclopentane hydrate	
$C_5H_{10}.17H_2O \rightarrow C_5H_{10} + 17H_2O$	82.3
$C_5H_{10} + 5H_2O \rightarrow 10H_2 + 5CO$	952.95
$CO + H_2O \rightarrow H_2 + CO_2$	-41.2
Direct decomposition	
$C_5H_{10} \rightarrow 5H_2 + 5\,C$ (s)	76.45

The composition of the methane hydrate is $CH_4.6H_2O$ [17, 18] and it also refers to the hydrate number of Handa [1]. In addition, dissociation enthalpy of methane hydrate (ΔH) in this study is 53.5 kJ/mol [18].

First, methane hydrate dissociated by plasma, and then some of water is turned into the steam by evaporation and the reaction of steam methane reforming results H_2, CO, CO_2 as shown in Table 30.1. In addition, H_2, C and light hydrocarbon as byproduct were resulted from direct decomposition of methane. Similarly, dissociation enthalpy of cyclopentane hydrate is 82.3 kJ/mol [19] and the reaction of plasma decomposition of cyclopentane hydrate is also shown in Table 30. The direct decomposition of cyclopentane also produced the light hydrocarbon as a byproduct.

27.12 MHz RF plasma decomposition of clathrate hydrates resulted the highest H_2/CO mole ratio for methane hydrate and the lowest mole ratio of H_2/CO for cyclopentane hydrate as shown in Fig. 30.7. In addition, enthalpy of H_2 formation from methane hydrate is lower than that from cyclopentane hydrate as shown in Fig. 30.8.

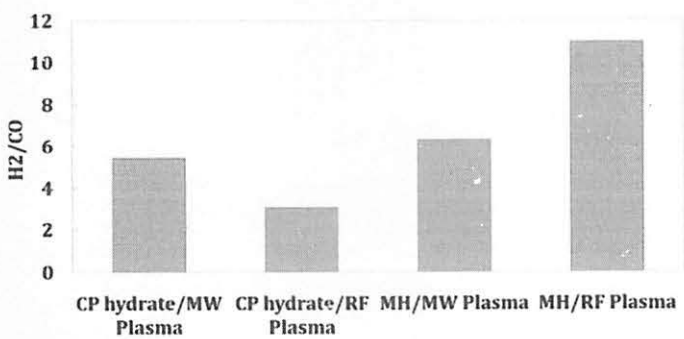

Fig. 30.7 The mole ratio of H_2/CO In the product gases from clathrate hydrate decomposition