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Improving the Efficiency of Thermochemical Hydrogen Production with Optimal Multiphase Design and Process Integration

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Abstract

The hydrogen economy is one potential avenue to a clean energy system, and a promising option for hydrogen production is thermochemical water decomposition. This process involves multiple steps, some consisting of multiphase reaction systems. This paper examines the importance of design optimization of gas-solid-liquid phase systems and process integration of a thermochemical copper-chlorine cycle for hydrogen production. A specific integration example is the dissolution of copper chloride in hydrochloric acid. This dissolution step is investigated in order to provide a predictive modeling method for more complex multiphase reacting systems.

Keywords: hydrogen production, global warming, clean energy, renewable energy

Résumé

Mots clés :

1. Introduction

Unsustainable human activities and practices are emitting greenhouse gases into the atmosphere, releasing compounds that diminish the Earth's stratospheric ozone layer, and polluting water supplies. Many unsustainable characteristics are exhibited by energy processes, which are one of the largest contributors to climate change.

Climate change occurs when long-term weather patterns are altered. Global warming is one measure of climate change given by a rise in the average global temperature. Earth needs naturally occurring, heat-trapping gases such as water vapour, carbon dioxide (CO₂) and methane to be able to sustain life, as we know it. The threat lies in the rapid increase of greenhouse gases that intensify the greenhouse effect. Carbon dioxide is the main contributor to climate change, which is attributed to the burning of fossil fuels. Another contributor is methane, which is produced naturally when vegetation is burned, digested or rotted without the presence of oxygen. Large amounts of methane are released by farming, waste and the production of oil and gas. Nitrous oxide is another contributor related to burning of fossil fuels that has a much greater global warming potential than that of carbon dioxide.

Due to the increasing impact on the health of humans and ecosystems and the economic costs associated with global warming and other environmental impacts, engineers, scientists and others are increasingly pursuing clean energy technologies. One approach is based on a hydrogen economy, in which the two main energy carriers are hydrogen and electricity.

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Hydrogen production is a key aspect of a hydrogen economy, and one promising renewable option for large-scale hydrogen production is thermochemical water decomposition. The copper-chlorine thermochemical cycle uses mainly thermal energy to split water into hydrogen and oxygen through intermediate copper and chlorine compounds, which are recycled continuously without releasing pollutants into the environment. A thermochemical hydrogen production cycle usually involves multiple steps, some consisting of multiphase reaction systems. To improve the system efficiency, it is important to optimize the multiphase system design and the process integration.

This paper provides alternative methods of hydrogen production that have the potential of addressing some of the aspects related to climate change, introduces an experimental set-up for multiphase design and discusses methodology for improved efficiency.

2. Hydrogen Energy Systems and Hydrogen Production

Many environmental problems are related to the production, transformation and use of energy. Some of the concerns include, but are not limited to, acid rain, ozone depletion and climate change [1-3]. Therefore, “greener” alternatives for energy production are sought. Hydrogen energy systems are potential solutions and can have a significant role in improving environmental sustainability [4-7]. However, current methods of hydrogen production are often unsustainable as they are based primarily on fossil fuels such as natural gas or coal, and release CO₂ into the atmosphere. Promising alternatives for sustainable hydrogen production are electrolytic, photoelectrolytic and thermochemical cycles. Non-fossil fuel based hydrogen production cycles use a series of steps to achieve the splitting of water into hydrogen and oxygen given by Eq. 1:



2.1 Hydrogen production via water electrolysis

One of the most common methods of direct water splitting is conventional water electrolysis. Electrolysis has been used for the production of hydrogen since the early nineteenth century. In this process, the water is split into hydrogen and oxygen using an electric current. It yields hydrogen at about 99.99% purity compared with 98% purity obtained from fossil fuel based methods [8]. Other advantages of electrolysis are that the produced hydrogen is already separated from oxygen without releasing by-products to the atmosphere. However, electrolysis is relatively costly compared to other hydrogen production methods such as steam methane reforming (SMR). The overall efficiency of hydrogen production through water electrolysis depends mainly on two factors: electricity-to-hydrogen efficiency and energy-source-to-electricity conversion efficiency [9]. Efforts to reduce the cost have been reported [10], including developing a proton exchange membrane (PEM) electrolytic hydrogen generator series, which enables renewable technology as a sustainable energy source.

2.2 Solar hydrogen production

Solar irradiance can be utilized to decompose water into hydrogen and oxygen with either indirect or direct water splitting methods. An indirect method is to utilize concentrated solar thermal energy at a high temperature to produce hydrogen in a thermochemical water splitting cycle [11]. Thermochemical cycle will be discussed in the following section. Another indirect method is the combination usage of solar PV-generated electricity and water electrolysis. The

process of a solar PV cell may use two doped semiconductor materials. A p-type and n-type are brought together forming a p–n junction [9, 12]. A permanent electric field is formed at the junction when the charges in the n-type material rearrange. When a photon with energy greater than the semiconductor material's bandgap is absorbed at the junction, an electron is released and a hole is formed [13]. The hole and electron are forced to move in opposite directions due to the electric field. As a result, an electric current is created when an external load or wiring is connected [14]. Then the electricity can be utilized in the conventional water electrolysis [15].

Similarly, the electric potential can also be generated even if the external load or wiring is not present when a photocathode, p-type material with excess holes, or a photoanode, n-type of material with excess electrons, is immersed in an aqueous electrolyte [9, 14, 16]. This potential can be used for direct water splitting if it exceeds the thermodynamic threshold of breaking the chemical bond between hydrogen and oxygen atoms in the water molecules. This direct water splitting method is often termed as photoelectrolysis [9, 14, 16]. Holladay et al. (2009) summarize the process in four steps. First, a photon with greater energy than the bandgap strikes the anode, creating an electron–hole pair. Second, the holes decompose water at the anode's front surface to form hydrogen ions and gaseous oxygen, while the electrons flow through the back of the anode, which is electrically connected to the cathode. Third, the hydrogen ions pass through the electrolyte and react with the electrons at the cathode to form hydrogen gas. In the last step, the oxygen and hydrogen gases are separated by the use of a semi-permeable membrane. The separated gases are then processed and stored [13].

The efficiency is directly related to the semiconductor band gap. This is the energy difference between the bottom of the conduction band and the top of the valence band. Efficiency is also related to the band edge alignments, since the material or device must have sufficient energy to split water [13]. In order to increase the efficiency of the system, electron transfer catalysts may be used. However, these can minimize the surface over-potentials in relation to the water and facilitate the reaction kinetics, decreasing the electricity losses in the system [13]. As a result, appropriate surface catalysts for the systems are being sought. Such catalysts must remain active for as many as 10^8 redox reaction cycles, which is the equivalent of 20 years of operation [9]. Researchers also suggested the use of suspended metal complexes in solution as the photochemical catalysts [17]. Typically, nano-particles of ZnO, Nb₂O₅ and TiO₂ have been used [13, 18].

2.3 Thermochemical cycles for hydrogen production

Thermochemical water decomposition generally involves at least three distinct steps: hydrogen production, oxygen production, and re-cycling of other byproducts and compounds internally. One of the advantages of thermochemical cycles over direct one-step thermal water decomposition is their ability to split water into hydrogen and oxygen at much lower temperatures (usually below 1,000 °C) [19]. One of the promising thermochemical cycles that operates at even lower temperatures (under 600 °C) is the thermochemical copper-chlorine (Cu-Cl) cycle. The Cu-Cl cycle splits water into hydrogen and oxygen through intermediate copper and chlorine compounds. The chemical reactions form a closed internal loop and re-cycle all chemicals on a continuous basis, without emitting any greenhouse gases to the atmosphere. An example of a proposed Cu-Cl cycle is shown in Figure 1.

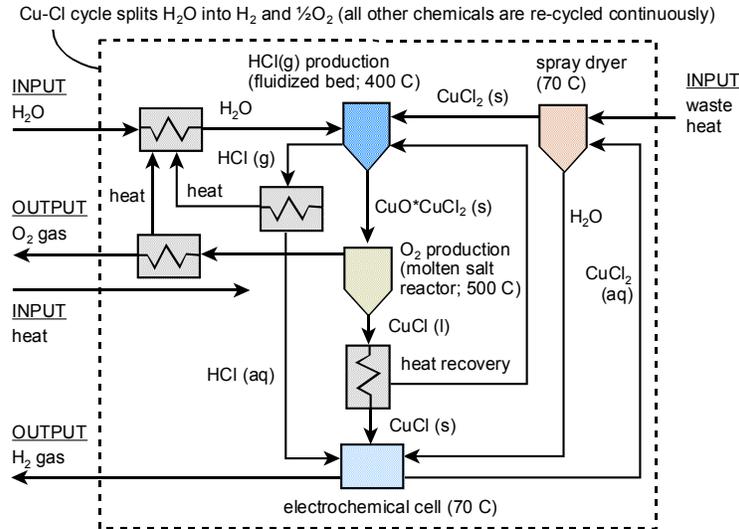


Figure 1. Schematic of a thermochemical copper-chlorine cycle [20]

There are several variations of the Cu-Cl cycle reported in past literature. Such variations include 5-step, 4-step and 3-step cycles [20,21]. A 4-step cycle with its chemical reactions is summarized in Table 1.

Table 1. Steps and chemical reactions in a four-step Cu-Cl cycle [20]

Step	Reaction	Temp. Range (°C)	Feed	Output*
1	$2\text{CuCl}(\text{aq}) + 2\text{HCl}(\text{aq}) \rightarrow \text{H}_2(\text{g}) + 2\text{CuCl}_2(\text{aq})$	<100 (electrolysis)	Feed: Output:	Aqueous CuCl and HCl + V + Q $\text{H}_2 + \text{CuCl}_2(\text{aq})$
2	$\text{CuCl}_2(\text{aq}) \rightarrow \text{CuCl}_2(\text{s})$	<100	Feed: Output:	Slurry containing HCl and $\text{CuCl}_2 + \text{Q}$ Granular $\text{CuCl}_2 + \text{H}_2\text{O}/\text{HCl}$ vapours
3	$2\text{CuCl}_2(\text{s}) + \text{H}_2\text{O}(\text{g}) \rightarrow \text{CuO} \cdot \text{CuCl}_2(\text{s}) + 2\text{HCl}(\text{g})$	400	Feed: Output:	Powder/granular $\text{CuCl}_2 + \text{H}_2\text{O}(\text{g}) + \text{Q}$ Powder/granular $\text{CuO} \cdot \text{CuCl}_2 + 2\text{HCl}(\text{g})$
4	$\text{CuO} \cdot \text{CuCl}_2(\text{s}) \rightarrow 2\text{CuCl}(\text{l}) + 1/2\text{O}_2(\text{g})$	500	Feed: Output:	Powder/granular $\text{CuO} \cdot \text{CuCl}_2(\text{s}) + \text{Q}$ Molten CuCl salt + oxygen

* Q = thermal energy, V = electrical energy

The first step of the cycle is electrolysis, in which hydrogen is generated as a result of copper (I) chloride (CuCl) oxidation in the presence of hydrochloric acid (HCl) during an electrochemical reaction. The overall cell cycle reaction is given by [20]:



Several relatively inexpensive electrode materials have been tested and hydrogen production has been achieved at potentials as low as 0.5 V [19]. Also, three different membranes were investigated to identify membranes with lower copper diffusion rates but similar proton conductivities [21].

The second step of the cycle consists of removing the water from aqueous copper (II) chloride [18]. Once the water is removed, the solid CuCl_2 is supplied to the hydrolysis reactor to produce copper oxychloride (Cu_2OCl_2) and HCl gas [21], via an endothermic non-catalytic gas-liquid or gas-solid reaction.

The last step of the cycle involves the multiphase reaction, in which oxygen gas and molten CuCl are obtained from decomposition of solid copper oxychloride.

Several gaseous products exit the reactor such as oxygen, CuCl vapour and side-reaction products such as HCl gas, Cl_2 gas and water vapour [20, 21].

It can be found that the CuCl produced in the last step of the cycle is in a molten state, by comparison, the existing state of the CuCl used in the first step is the dissolved state of the CuCl in hydrochloric acid. This means, the following physical process is needed in order to link the first and last steps of the cycle:



The solidification of molten CuCl will not be discussed in this paper. The research reported in this paper has been motivated by the need to determine the optimal dissolution rate of solid CuCl particles in HCl for improved efficiency of the cycle. The solution obtained is then supplied to the first step of the cycle (i.e. electrolysis reactor) for the system integration.

3. Experimental Set-up

In order to better understand the physics of liquid-solid systems and the dissolution rate of copper chloride (CuCl) in hydrochloric acid (HCl), an experimental apparatus has been designed and built at the University of Ontario Institute of Technology (UOIT) in the Clean Energy Research Laboratory (CERL). The experimental apparatus consists of an 8-in high, 4-in diameter clear pyrex vessel filled with liquid and a camera system (see Figures 2 and 3).

In the first set-up of the experimental loop (Figure 2) CuCl particle dynamics through water are examined so that the descending velocity of the particles can be determined.

In the second set-up of the experimental loop (Figure 3) the dissolution rate of CuCl particles in stagnant HCl is examined. Since the dissolution rate of the CuCl particles is affected by mixing, nitrogen gas is supplied from the bottom of the pyrex vessel and the flow generates a steady mixing of the liquid without disturbing the particle. The flow rate of nitrogen is set manually using rotameters.

A FlowSense 2ME camera with an effective sensor size of 1600 by 300 pixels was used to capture images. Images were captured in single frame mode with a trigger frequency of 91 Hz when observing the particle dynamics and 1 Hz when observing the dissolution rate.

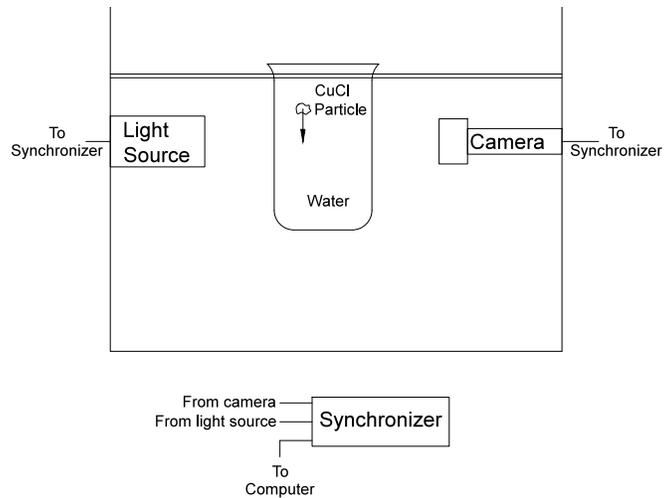


Figure 2. Experimental set-up for particle dynamics

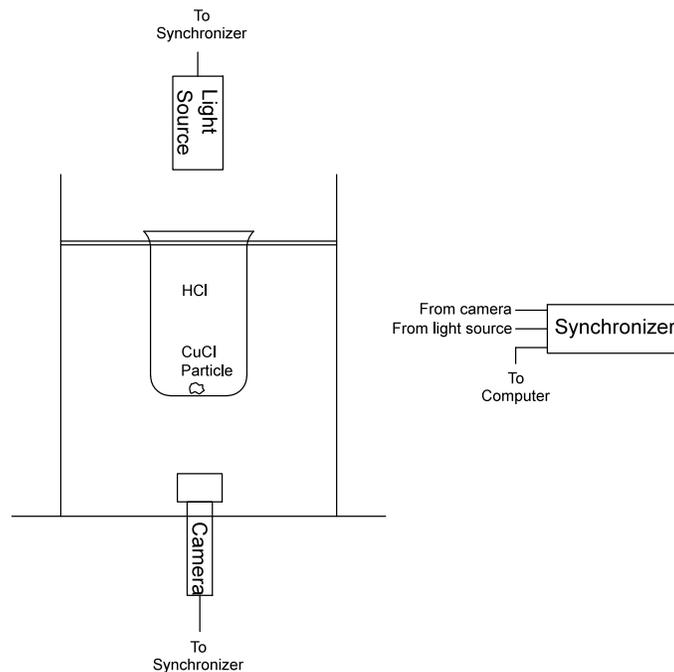


Figure 3. Experimental set-up for dissolution rate

The particle in motion was frozen with the aid of a short light flash and a synchronizer. A glass diffuser was used to obtain an even light sheet so that the camera can acquire a shadow image of the particles. DynamicStudio software, an advanced edge detection algorithm to extract the bubble shape information, is used to acquire and process the images. The “shadow sizer processing” method is used to extract information such as size, position, shape and velocity of particles. Prior to image processing, a set of calibration images was acquired. This step was necessary as it enabled the measurement of a scale factor. The scale factor was used to determine the conversion of pixel units into metric units. The velocity information is then extracted through combining a correlation algorithm and measuring displacement between two successive images via a dedicated particle-tracking algorithm.

4. Results and Discussion

For successful operation of the Cu-Cl cycle for hydrogen production, it is important to carefully design intermediate phases that aid in the integration of the main steps. As depicted in Figure 1 and Table 1, molten CuCl salt leaves the oxygen reactor, enters an intermediate step which recovers the heat thus changing its state to solid and it finally reaches the electrolysis reactor where it aids in the production of hydrogen. Similarly, HCl gas leaves the hydrolysis reactor and enters the electrolysis reactor as hydrochloric acid. However, for improved performance of the cycle, it is necessary to dissolve the solid CuCl particles in aqueous HCl before reaching the electrolysis cell. Hence, a second intermediate step is introduced between the oxygen reactor and electrolysis reactor. Before introducing this step, a series of experiments have been performed to understand the dynamics of solid CuCl particles in aqueous HCl and to study the dissolution rates in solution of different molarities such as 12M, 9M and 6M HCl. A clear understanding of the dynamics of the particles and the dissolution rates will aid in the design of the mixing step and effective integration of the cycle. Two different sets of experiments have been performed. The first consists of dropping CuCl(s) into water to determine the velocity of the particle. In this set of experiments, HCl(aq) was replaced by water since they have similar densities and the dynamics of the particle would not be altered. However, a source of error is introduced as the CuCl(s) particles begin dissolving immediately when dropped in HCl(aq) as opposed to when dropped in water and no dissolution takes place. Therefore, the dissolution of the particle would affect its dynamics as well as the density of the solution. In this paper it is assumed that the CuCl(s) particle does not begin dissolving until it settles on the bottom of the vessel.

To better understand the dynamics of the particles in water, Figure 4 depicts the processed images of a descending particle with velocity vectors starting from the center of the particle and marked in red, taken in the same reference frame at 0.956 s and 1.011 s, respectively. It is observed that the particle's velocity changes as it changes orientation (see Table 2). Due to the irregular shape, the particle changes orientation until it settles on the bottom of the vessel with a settling velocity [22-23].

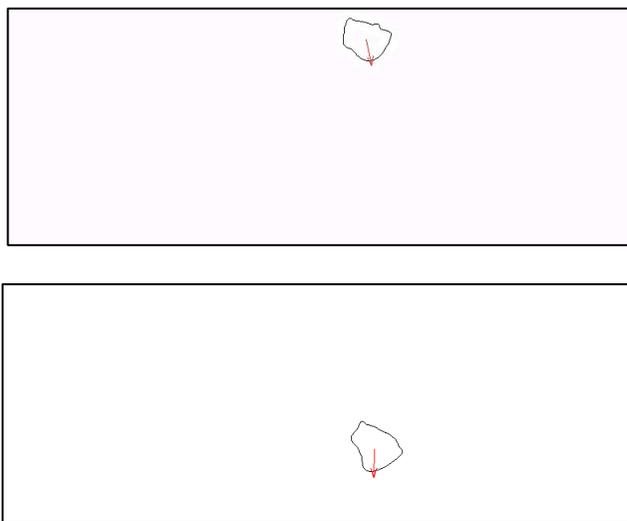


Figure 4. Processed images showing velocity vector for a 45 mg particle at 0.956 s (top) and 1.011 s (bottom)

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Time stamp (s)	Area ^a (mm ²)	EqDia ^b (mm)	MajAxis ^c (mm)	MinAxis ^d (mm)	Shape ^e	U vel ^f (m/s)	V vel ^g (m/s)
0.956	18.559	0.418	0.249	0.184	0.896	0.005	-0.022
1.011	19.166	0.425	0.24	0.2	0.909	0	-0.023

^a Cross-sectional area; ^b Diameter of the circle with an area equivalent to the cross-sectional area; ^c Longest axis of particle; ^d Shortest axis of particle; ^e Shape of the particle relative to circle; ^f Horizontal velocity; ^g Vertical velocity

Table 2. Parameters obtained using DynamicStudio

The instantaneous velocities for different mass particles are tabulated in Table 3. It is important to note that the particles are irregular in shape and the area is the cross sectional area of the particle parallel to the direction of motion. Thus the mass and area values in Table 3 are not necessarily correlated, explaining why the particle in Table 3 with a mass of 33 mg has a smaller cross sectional area than the one with a mass of 31 mg.

Area ^a (mm ²)	U vel (m/s)	V vel (m/s)	Mass (mg)
3.841	-0.003	-0.012	4.000
3.293	0.000	-0.017	7.000
5.042	-0.007	-0.020	7.000
6.971	-0.002	-0.016	8.000
7.060	-0.001	-0.017	8.000
12.950	-0.001	-0.020	21.000
9.228	0.005	-0.017	22.000
15.562	-0.003	-0.018	26.000
20.816	0.000	-0.026	31.000
13.764	-0.001	-0.023	32.000
8.591	0.004	-0.020	33.000
19.795	0.000	-0.023	45.000

^a Projected area of the particle parallel to the direction of motion

Table 3. Areas and velocities of particles of varying masses

The second set of experiments consisted of dropping CuCl(s) particles in HCl(aq) and recording the time required to dissolve. The mass of the particle was kept constant at 7 mg, however the molarity of the HCl(aq) varied between 6M to 12M. The cross sectional area of the particle was recorded with the aid of the camera system from the time the particle was dropped in the acid until it fully dissolved. Initially, the particle was dropped in stagnant HCl(aq) and local saturation could be observed. To speed up the dissolution rate and to avoid local saturation, nitrogen was injected from the bottom of the container. The flow of nitrogen provided mixing without disturbing the particle so that the camera can capture the same view of the particle as when

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dropped in the acid. As expected, mixing increased the dissolution rate and prevented local saturation.

These findings coupled with the dynamic data can provide valuable information regarding the size of the dissolution vessel, the type of mixing required and molarity of the solution to increase the dissolution rate and ultimately improve the efficiency of the hydrogen production cycle.

6. Conclusions

Various techniques are summarized for small- and large-scale production of hydrogen, a potentially “green” energy carrier, and advantages and disadvantages are highlighted. The focus was on hydrogen production via the thermochemical Cu-Cl cycle, which has the ability to produce large amounts of hydrogen without releasing greenhouse gases to the atmosphere. Shortcomings regarding the integration of the first and last steps of the cycle were discussed and a method to overcome the shortcomings for efficiency improvement has been proposed. As a follow up, a correlation will be developed relating the velocity of the particles and concentration of the solution to the change in mass, the change in mass due to dissolution as the particle is falling will be investigated, and an optimum mixing technique for improved efficiency will be proposed and implemented.

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9. Biography

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