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Experimental validation of an optimal operating window in the Bunsen reaction section of the iodine–sulfur cycle

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ABSTRACT

The Bunsen reaction section of I–S cycle has two parts, reaction and phase separation. Some previous work focused on phase separation using a simulated reaction mixture with four components (H_2SO_4 , HI, H_2O and I_2). Based on phase separation data an operating window was suggested. This previous work had the limitation that reaction was not included. The present work includes both reaction and separation. We demonstrated that the Bunsen reaction ratio is higher than 2, and the azeotropic ratio (mol of HI/mol of $\text{HI} + \text{H}_2\text{O}$) is higher than 0.4 at 293 K and 2 mol of I_2 in the feed. Bunsen reaction was promoted at a lower temperature and higher I_2 content, while phase separation became worse since the generated H_2SO_4 and HI remained in the HIx phase without separation. We validated the previously suggested optimal window and examined phase separation at the lower temperature region.

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Introduction

Hydrogen is considered as a future energy carrier that can substitute for hydrocarbon fuel. It is currently produced by coal gasification and natural gas reforming, which are still highly dependent on fossil fuel. In the consideration of the depletion of natural resources and global warming due to greenhouse gas, alternative clean and sustainable hydrogen production methods are taken into account as one of the

future energy strategies. Especially, water-splitting methods are one of the promising processes for producing hydrogen. These water-splitting methods are mainly classified as three categories: electrolysis, a hybrid thermochemical cycle, and a thermochemical cycle. Among them, a thermochemical cycle is suitable for massive production of hydrogen, since it is easy to scale up and requires only heat source for the process. The sulfur–iodine cycle proposed by General Atomics Co. (GA) was identified as the most promising among 115 thermochemical hydrogen cycles considered in a 1981 survey [1].

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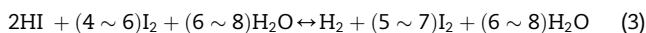
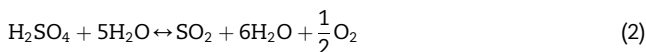
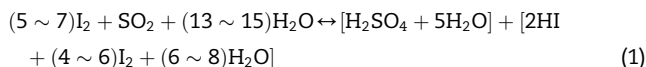
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Nomenclature

HI	hydrogen iodide
HI _x	poly-hydriodic acid
I ₂	iodine
L–L	liquid–liquid
S.A.	sulfuric acid
SO ₂	sulfur dioxide
α	Bunsen reaction ratio = dissolved mol of SO ₂ /13 mol of H ₂ O
ω	H ₂ O distribution ratio between SA and HI _x phase

The I–S cycle is a water-splitting process as illustrated in Fig. 1. It consists of three sections: Bunsen reaction section (1), sulfuric acid decomposition section (2), and Hydrogen iodide decomposition section (3) as shown in the following equations.



When SO₂ and I₂ are added to H₂O, H₂SO₄ and HI are generated spontaneously through Bunsen water-splitting reaction. Oxygen is produced from decomposition of H₂SO₄; hydrogen is produced from decomposition of HI. SO₂ and I₂ from both decomposition processes are recovered and reused in the Bunsen reaction section; thus the cycle is closed. In contrast to spontaneous generation of H₂SO₄ and

HI from the reaction, separation between two acids does not proceed by itself. Thus, the conventional way to achieve good phase separation is adding excessive amounts of H₂O and I₂ to the Bunsen reaction section. This treatment enhances a good phase separation characteristics in Bunsen reaction: H₂SO₄ and parts of H₂O move to upper phase; HI and I₂ and parts of H₂O moves to lower phase due to density difference. However, excessively supplied H₂O for phase separation reacts with acid makes an azeotrope. Azeotropic constraint makes it difficult to separate pure H₂SO₄ and HI from H₂O. Thus, unnecessary H₂O flows through an entire cycle and undermines the efficiency of total cycle. This is the first drawback of conventional I–S cycle. The second drawback is material integrity in highly corrosive condition. The I–S cycle contains strong acids(HI, H₂SO₄), it operates in high temperature and pressure region in accordance with original suggestion from GA Co. Only few noble metals such as tantalum and platinum are candidate for structural materials. But those are not a perfect corrosion resistance material but uneconomical.[2]

As mentioned above, the conventional I–S cycle has many technical challenges to resolve [3]. To cope with underlying drawbacks of conventional I–S cycle, a hydrogen research team at Korea Advanced Institute of Science and Technology(KAIST) proposed new operating windows of the Bunsen reaction section and flowsheet which includes flash distillation. The proposed optimal operating point for Bunsen reaction is represented by 4 mol of excess iodine and 11 mol of excess water in the stoichiometry at temperature of 330 K while allowable operating window ranges over 4–6 mol for the excess iodine, 11–13 mol for the excess water, and 330–350 K as expressed in Eq (1).[4] This proposal was based on parametric studies of 70 sets of existing experimental data. If the Bunsen reaction section is operating within a proposed optimal range, a concentration of HI becomes overazeotropic condition in lower HI_x phase. Then,

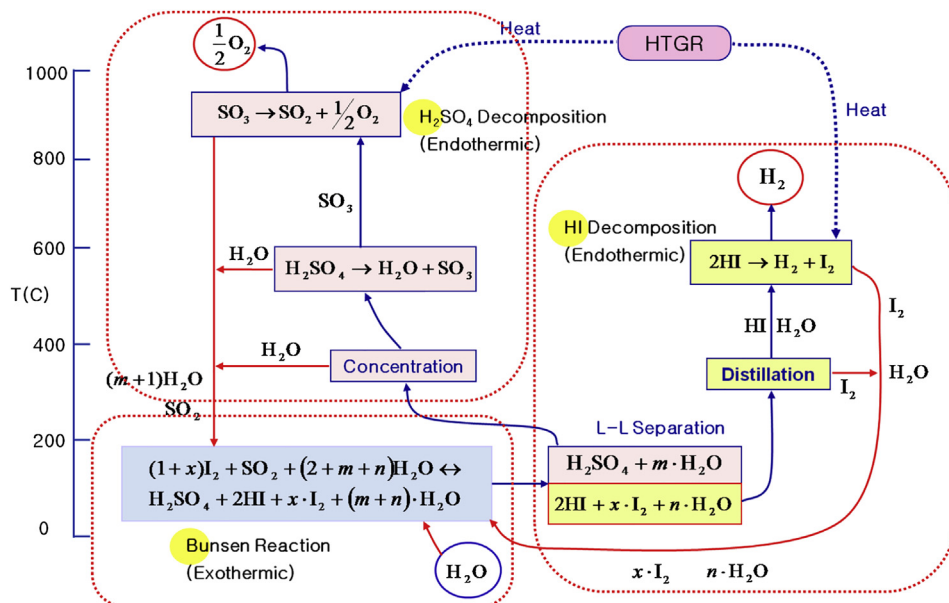


Fig. 1 – Iodine–Sulfur thermochemical water-splitting process and an azeotropic constrain.

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