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CONF-800616--6(Rev)

GA-A15777
(REV)

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This is a preprint of a paper to be presented at the 3rd World Hydrogen Energy Conference, June 23-26, 1980, Tokyo, Japan and to be published in the Proceedings.

Work supported by
Department of Energy
Contract DE-AC02-80ET26225

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GENERAL ATOMIC PROJECT 3260
MAY 1980

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ABSTRACT

A summary of the progress of the overall total development effort of the General Atomic (GA) sulfur-iodine thermochemical water-splitting cycle over the last two years is reported. The major accomplishments have been the following:

1. Significant improvements in the chemistry of the process.
2. Development, review, and revision of an engineering flowsheet, resulting in a thermal process efficiency of 47%.
3. Screening, identification, and testing of potential materials-of-construction for the corrosive process fluids.
4. Small-scale demonstration of the cycle in a closed loop under recycle conditions.
5. Installation of bench-scale equipment and demonstration of parts of the process in this system.
6. Development of a conceptual, preliminary flowsheet for the GA sulfur-iodine cycle driven by solar energy.

The results of the work carried out during the last two years have demonstrated that thermochemical water splitting by the sulfur-iodine cycle is a feasible process and have provided confidence that thermal efficiencies in the range of 50% are achievable.

KEYWORDS

Thermochemical water splitting; sulfur-iodine process; hydrogen iodide; solar-water splitting.

INTRODUCTION

Thermochemical water splitting potentially provides a nonfossil renewable source of hydrogen. The General Atomic Company (GA) sulfur-iodine cycle appears to be compatible with the heat characteristics projected for three of the major energy sources to achieve the necessary temperature conditions required for the process. These heat sources are: the high-temperature gas-cooled reactor (HTGR), the fusion reactor, and systems for concentrating solar energy. GA is working on the development of all three of these heat sources.

The sulfur-iodine cycle has been under investigation at GA since 1974. The process can be described by the following three equations:

$$xI_2 + SO_2 + 2H_2O \rightarrow H_2SO_4 + 2HI_x \qquad 370\ K\ , \qquad (1)$$

$$2HI_x \rightarrow H_2 + xI_2 \qquad 393\ K\ , \qquad (2)$$

$$H_2SO_4 \rightarrow H_2O + SO_2 + 1/2\ O_2 \qquad 1144\ K\ . \qquad (3)$$

In these equations, HI_x represents the average of several HI_n compounds formed in the initial solution reaction. Separation of the H_2SO_4 and HI_x takes place under gravity, since the two acids are nearly immiscible. The upper phase contains most of the H_2SO_4, and the lower phase contains most of the HI_x.

The main attributes of this cycle are its expected high thermal efficiency (50%) and that it can be conducted as an all-liquid and a gas-phase process.

The total process development at GA has been historically divided into several areas:

1. Basic chemical investigations.
2. Material investigations.
3. Process engineering studies.
4. Bench-scale testing.

Recently, GA has initiated engineering studies to couple the existing sulfur-iodine process to solar heat sources.

The chemical investigations are under the joint sponsorship of the Gas Research Institute (GRI) and GA. The materials investigations are sponsored by the Metal Properties Council (MPC). The process engineering studies and the bench-scale testing are sponsored by the U.S. Department of Energy (DOE), Division of Energy Storage Systems. The solar studies are sponsored by the Advanced Technology Branch, Solar Thermal Power Office of DOE.

BASIC CHEMICAL INVESTIGATIONS

The major effort in the chemistry area during the last two years was focused on developing new process methods, improving the existing technology, and collecting detailed data for the process engineering design effort. Figure 1 illustrates the sulfur-iodine process. It consists of four distinct sections:

1. Section I produces the acids H_2SO_4 and HI from H_2O, SO_2, and I_2. The SO_2 that is fed to this section contains all the O_2 generated by the decomposition of the H_2SO_4. The O_2 product is removed from this section.

2. Section II concentrates and decomposes H_2SO_4 received from Section I.

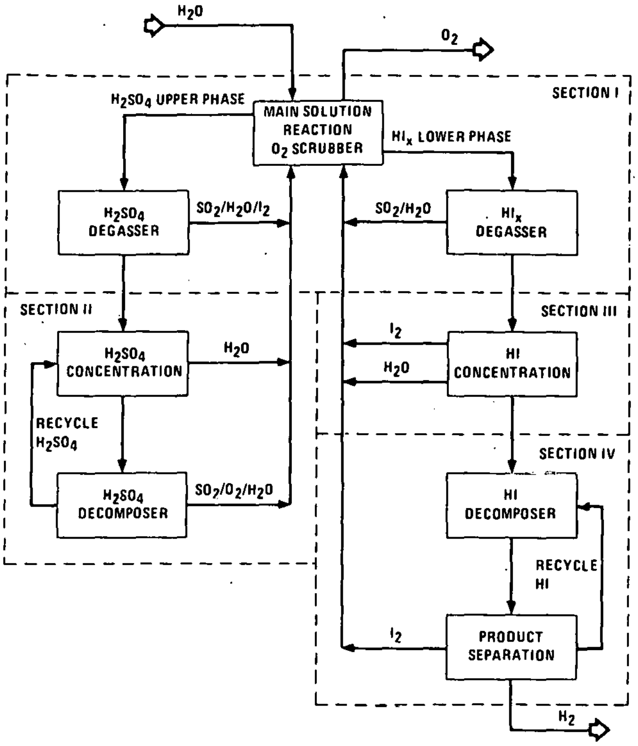

Fig. 1. Sulfur-iodine cycle process model.

3. Section III removes the very small amount of dissolved H_2SO_4 and unreacted SO_2 from lower phase product [HI_x (sol)] and then separates it into HI, I_2, and H_2O using H_3PO_4.

4. Section IV decomposes HI into H_2 and I_2 and separates products. The H_2 product is taken from this section.

5. Section V, not shown in the figure, is the intermediate helium loop that combines the helium-related parts for the delivery of nuclear heat and the generation of process power.

A number of significant process innovations have resulted from the chemistry effort:

1. The main solution reaction of the process is now being carried out at 368 K (95°C), which results in a higher process yield and an improved separation of the reaction products.

2. The separation of SO_2 and O_2 is no longer carried out cryogenically, but rather, in the main solution reaction where the SO_2 is reacted and O_2 leaves the system.

3. A sulfuric acid boost reaction has been added to the process. In this reaction, upper phase from the main solution reaction is reacted with molten iodine and additional SO_2. This increases the concentration of sulfuric acid from 50 to $\sim$57 wt %.

4. The purification of HI from the lower phase is now being carried out using phosphoric acid. Chemical research concentrating on the

optimization of the phosphoric acid step has resulted in a significant reduction in equipment and an increase in process thermal efficiency.

5. Also, a new separation system has been identified which may further improve the process economics. In the $HI/I_2/H_2O/H_3PO_4$ phase diagram, a region exists with two liquids, where one of the liquids is almost pure HI. Work is underway to incorporate this two-phase separation system into the flowsheet.

6. HI decomposition, which was previously carried out in the gas phase at ∿700 K, is now being developed for the liquid phase at ∿400 K. This has significant advantages for the efficiency of the process.

The process innovations and improvements made in the last two years are mainly responsible for the increased thermal efficiency of the sulfur-iodine process.

MATERIAL INVESTIGATIONS

The main solution reaction of the sulfur-iodine process produces the immiscible HI_x and H_2SO_4 acid phases from I_2, SO_2, and H_2O. These acids are then purified and decomposed to yield the original reaction products and reactants ($H_2 + O_2$). In a process plant of commercial size, the different process fluids will require large containment vessels, heat exchangers, and pumping hardware. Industrial experience in handling some of these corrosive fluids is limited. Therefore, material compatibility data need to be developed before a realistic process plant design can be carried out. GA has placed its main emphasis on materials testing in solutions which are specific to the sulfur-iodine process, the HI_x solutions. Limited corrosion tests with sulfuric acid have also been carried out. A large number and types of materials were screened at room temperature, and the best candidates were then subjected to extensive tests at elevated temperatures.

Sufficient numbers and types of materials have shown good compatibility with HI_x solutions. Thus, realistic, first-order materials selections can now be made for use in the conceptual design of both a pilot and a commercial process plant. Also, with these materials identified, it is anticipated that conventional hardware design concepts of the chemical industry can be utilized.

Table 1 identifies materials that have demonstrated satisfactory compatibility with HI_x and are now prime candidates for use in a conceptual design of process plant components. In Fig. 2, weight change values are presented graphically for six of the alloys investigated. Zirconium, tantalum, Durichlor 51, and Hastelloy B-2 are considered to be candidates for design applications. At the currently shown corrosion rate, zirconium is showing a decrease in thickness of 0.044 mm/yr, while Hastelloy B-2 is exhibiting a loss of 0.70 mm/yr. In contrast, most iron-base and nickel-base alloys exhibited moderate to rapid general corrosion even at 295 K, and thus, they are not viable candidates.

A comparison of corrosion rates for candidate materials is shown in Table 2.

Since the bench-scale system has now been installed, an effort is underway to test the main candidates for materials-of-construction in the bench-scale system. The bench-scale equipment originally installed was glass. Now some of this equipment has been replaced with metal vessels from candidate materials, and corrosion testing will be part of the bench-scale effort.

TABLE 1 Prime Candidate Materials for Component Design Applications in HI_x at 370 K

Material	General Rating After 1000 h of Test Exposure
Metallic	
Refractory Metals	
Tantalum	Excellent
Molybdenum	Excellent
Niobium - 1% zirconium	Excellent
Zirconium Base	
Zirconium 702; 4.5 wt % max hafnium	Excellent
Zirconium (as cast)	Excellent
Zirconium 702 (as welded); 4.5 wt % max hafnium	Excellent
Zircaloy 2; 1.5 wt % tin, 0.12 wt % iron, 0.10 wt % chromium, 0.05 wt % nickel	Good
Iron Base	
Durichlor 51; 14 wt % silicon, 4 wt % chromium (as-cast)	Good
Nickel Base	
Chlorimet 2; 31 wt % molybdenum (as cast)	Fair
Hastelloy B-2; 30 wt % molybdenum	
Plastics	
Fluorocarbons	
Teflon TFE	Good
Teflon PFA	Good
Kel-F81	Good
Teflon FEP	Good
Teflon ETFE	Good
Kynar 450	Fair
Halar E-CTFE	Fair
Elastomers	
Fluorocarbon	
Kalrez 1050	Fair
Fluorocarbon V-834	Fair
Ceramics and Glasses	
Glass	
Borosilicate	Excellent
Vitrecarb	Excellent
Ceramic	
Silicon carbide (CVD)	Excellent
Alumina (high purity)	Excellent
Zirconia (low porosity)	Good

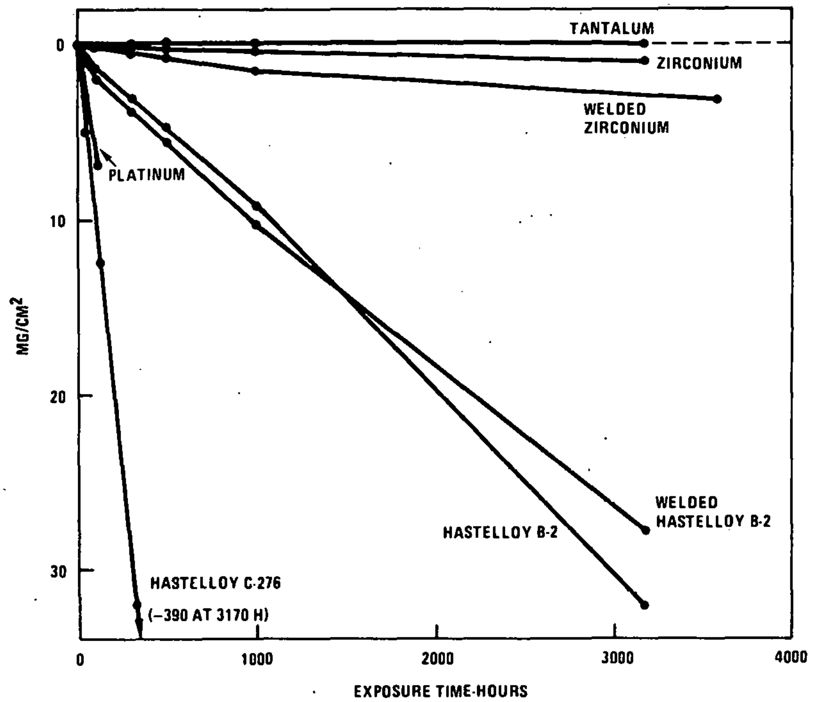

Fig. 2. Weight loss due to general corrosion for metallic materials exposed to liquid HI_x.

TABLE 2 Comparison of Corrosion Rates Corrosive Solution: HI_x at 370 K

Material	Corrosion Rate (mm/yr)
Ta, Mo, Nb-1% Zr	Nil
Zr-702[(a)]	0.044
Hastelloy B-2 (Ni-30% Mo)	0.700

(a) The prime candidate metal for heat exchangers and containment vessels handling HI_x is Zr-702.

PROCESS ENGINEERING STUDIES

The process engineering design effort for the water-splitting cycle at GA was based on the following objectives and constraints:

1. Energy is supplied by a process heat HTGR having a helium inlet temperature of 772 K and an outlet temperature of 1265 K.
2. Process power is supplied from the helium loop and from a low-temperature, process-bottoming cycle.
3. The temperature match-up of the helium heat delivery line and the process heat demand line must be good to maintain efficiency.

4. Heat must be reused within the process until it is at such low quality that it must be rejected.
5. Reasonable estimates are used for unavailable thermochemical data.

In 1979, a revision of the total flowsheet was completed. In this revised version, several of the new process improvements were incorporated in the flowsheet. The result of this revision was a significantly improved efficiency of 47% (from 42%). The main process improvements included in the design were the following:

1. A higher temperature in the main solution reaction.
2. An increase in sulfuric acid concentration to 57% (from 50%).
3. Utilization of phosphoric acid of lower concentration in the HI purification system.
4. Decomposition of HI in the liquid phase instead of in the gas phase.

These process improvements were mainly responsible for the increased thermal efficiency of the process. A schematic of the sulfur-iodine process is shown in Fig. 1, and a description of the process via the simplified flow diagrams of Sections I through V is given below. The flow diagrams for Sections I through V are shown in Figs. 3 through 7.

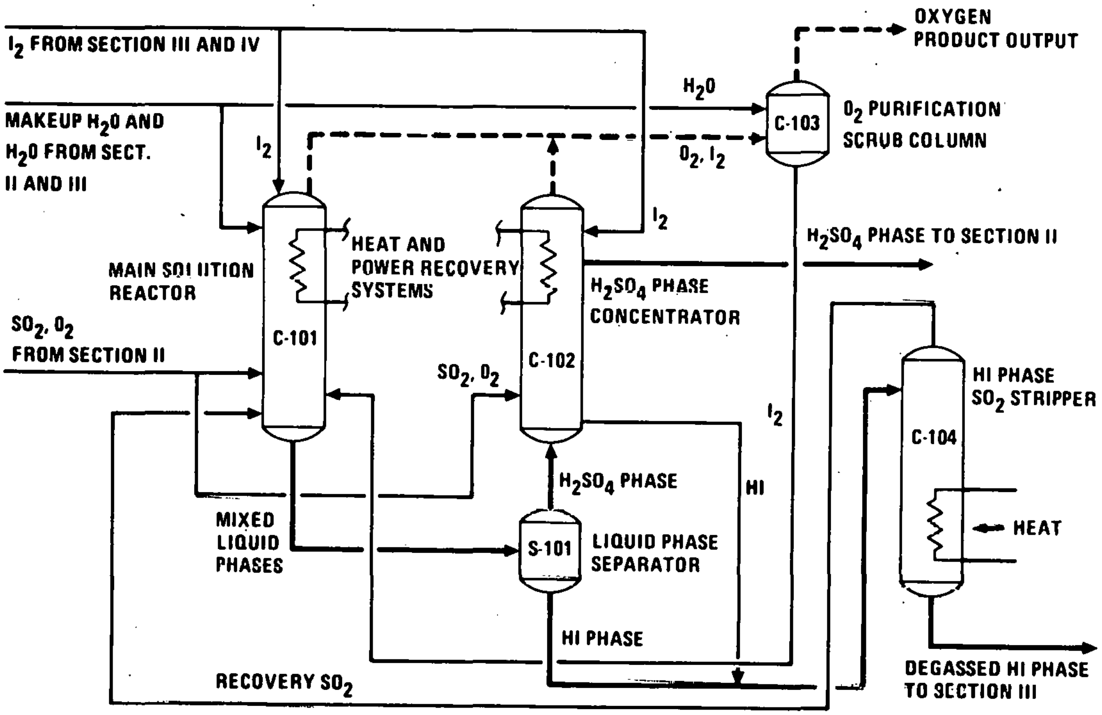

Fig. 3. Section I, Main Solution Reaction (H_2SO_4-HI Production and Separation and O_2 Removal).

Flowsheet Section I - H_2SO_4-HI Production and Separation; O_2 Purification (Fig. 3)

In the main reaction, recycle iodine from Sections III and IV reacts with H_2O and SO_2 from a mixture of gaseous SO_2/O_2 in a countercurrent reactor (C-101). The reaction results in the formation of the two acids, H_2SO_4 and HI, in solution. The discovery of the formation of two phases and the natural separation of these phases made the sulfur-iodine cycle feasible. The lower density phase (upper phase) contains all the H_2SO_4 at a concentration of ∿50 wt %, with traces of iodine and a small amount of dissolved SO_2. The higher density phase (lower phase) contains all the HI with considerable amounts of iodine in an H_2O solution. A small quantity of SO_2 and a trace of H_2SO_4 is also present.

The phases are separated (S-101), and the sulfuric acid phase is reacted with molten iodine and SO_2. This increases the H_2SO_4 concentration to ∿57 wt % and generates additional reaction product HI. The 57% sulfuric acid is transferred to Section II for concentration and decomposition. The lower phase goes through a degassing step which removes practically all the SO_2 (C-104). This lower phase, containing HI, H_2O, and I_2, is then transferred to Section III for purification and HI separation.

The SO_2 entering the main reaction (C-101) and the H_2SO_4 boost reaction (C-102) is a mixture of SO_2 and O_2 coming directly from the SO_3 decomposition reaction of Section II. As this gas mixture passes through the reactor, SO_2 is removed by reaction with I_2 and H_2O, and the gas leaving the top of the main solution reactor is practically pure oxygen with small amounts of iodine. The iodine is removed in a scrub column (C-103), and pure oxygen leaves the system as a product.

Flowsheet Section II - H_2SO_4 Concentration and Decomposition (Fig. 4)

Dilute sulfuric acid (57 wt %) from Section I is concentrated in a series of flash evaporators (V-201 through V-212). The concentrated H_2SO_4 is decomposed (E-221) to H_2O and SO_3, and the sulfur trioxide is decomposed (E-214) to sulfur dioxide and oxygen. The SO_3 decomposition is the highest temperature step in the total process with operating temperatures of up to 870°C. The gaseous mixture of SO_2 and O_2 is separated from the H_2O and unreacted H_2SO_4 and transferred to the main solution reactor in Section I. The condensate from this separation is recycled to the first flash evaporator.

The concentration and decomposition of H_2SO_4 is common to a number of water-splitting cycles and has been evaluated in several laboratories. GA has developed proprietary catalysts and processes which have been demonstrated at the quoted temperature.

Flowsheet Section III - HI Concentration (Fig. 5)

Lower phase from Section I containing ∿4 mole of iodine and 5 mole of water for each mole of HI is treated with concentrated phosphoric acid (C-302), and a major portion of the iodine (∿95%) is separated from the solution. This iodine is scrubbed with water to remove small amounts of HI and H_3PO_4 (C-301) and returned to the main solution reaction of Section I. The overhead solution containing HI, H_2O, H_3PO_4, and some I_2 is subjected to an extractive distillation (C-303), where most of the H_2O (99%) remains with the phosphoric acid and the HI and I_2 are removed as overhead vapor. Minor amounts of H_2S may be formed in these steps from a reaction of trace quantities of H_2SO_4 with HI. The dilute phosphoric acid is concentrated in a series of concentrators (E-318 through E-321) and reused for the iodine separation, as discussed above. The overhead containing HI, some I_2, and a very

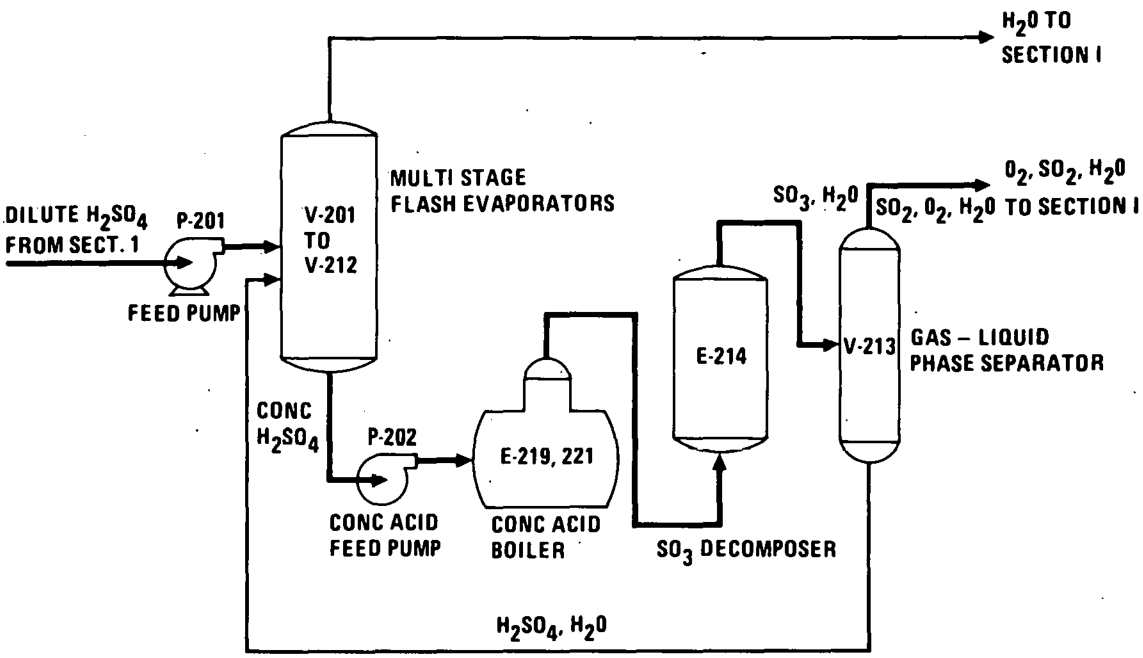

Fig. 4. Section II, H_2SO_4 Concentration and Decomposition

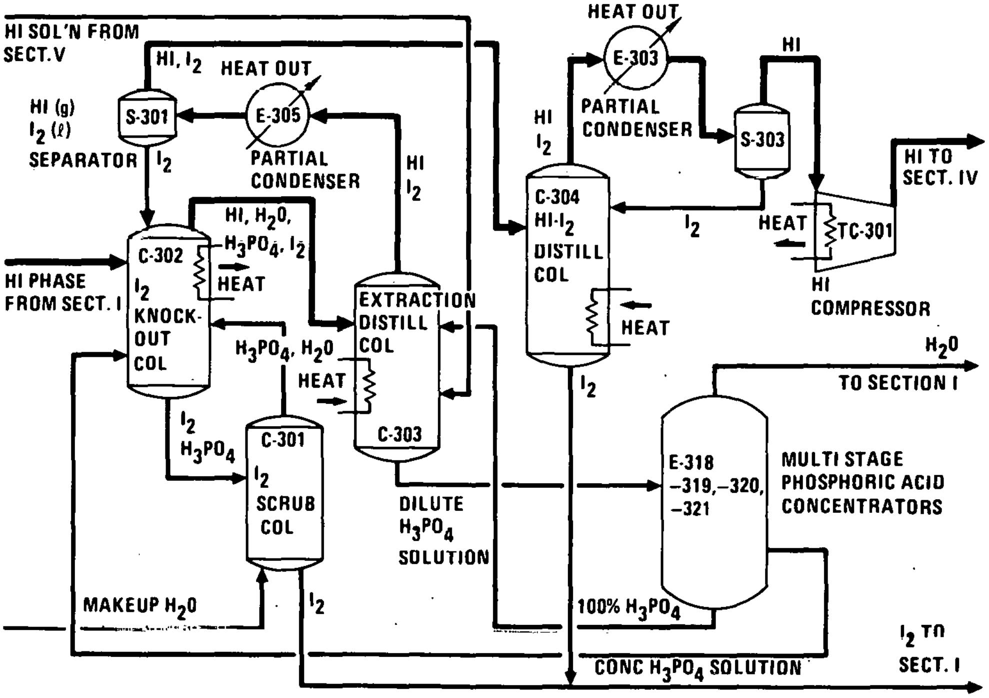

Fig. 5. Section III, HI Concentration

small amount of water is cooled (E-305) to condense and separate (S-301) some of the iodine and then subjected to another distillation (C-313). Here the HI is purified to a level where it can be sent to Section IV for decomposition after compression to 50 atm.

Flowsheet Section IV - HI Decomposition (Fig. 6)

Purified liquid HI (50 atm) from Section III is catalytically decomposed at ~120°C (R-401). The degree of decomposition is only ~30% in one pass. Therefore, the recycle step has to be used in this section. The hydrogen product is separated from most of the I_2 and some HI in a liquid gas separator (S-401). This gas is then cooled (E-408, -409, -410) to condense out most of the HI which is recycled to the HI decomposer (R-401). The gaseous H_2 product is scrubbed with H_2O, and pure hydrogen is the resulting end product. The liquid from the gas-liquid separator (S-401) contains mostly iodine and HI. The HI is removed by distillation (C-401) and returned to the HI decomposer (R-401). The iodine is returned to the main solution reaction in Section I.

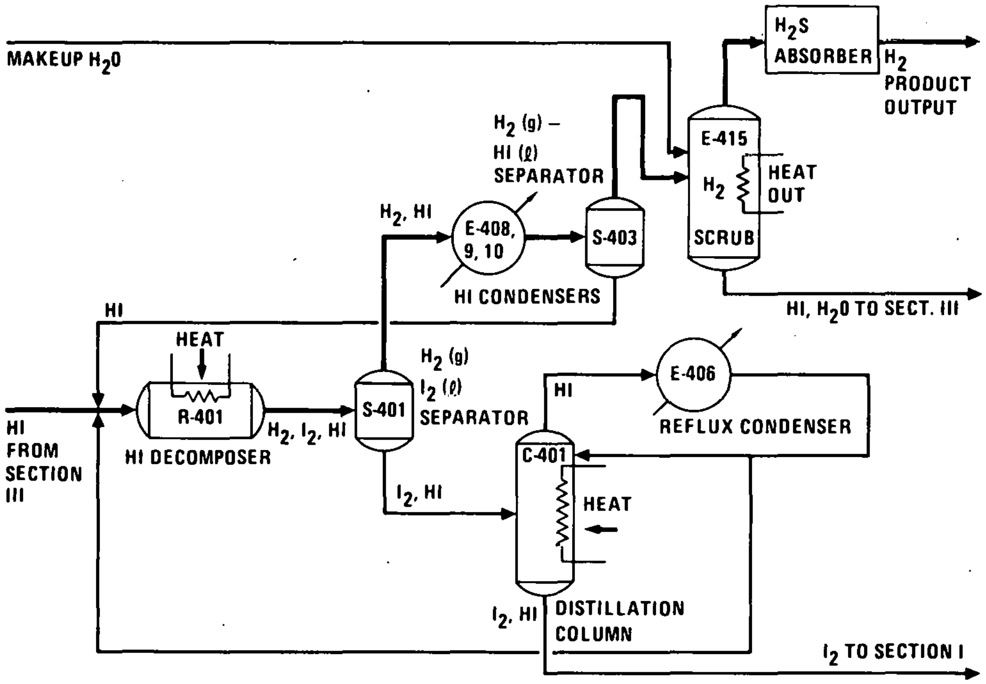

Fig. 6. Section IV, HI Decomposition

Flowsheet Section V - Power Generation and Heat Transfer (Fig. 7)

Section V of the flowsheet describes the generation of heat and power needed in some of the processing sections. The basic assumption has been that an HTGR, similar to the one designed by GA, would be available.

Helium from the primary loop transfers its heat to three secondary helium loops through heat exchangers which operate at high, intermediate, and low temperatures. The high temperature loop provides the heat for the sulfuric acid decomposition

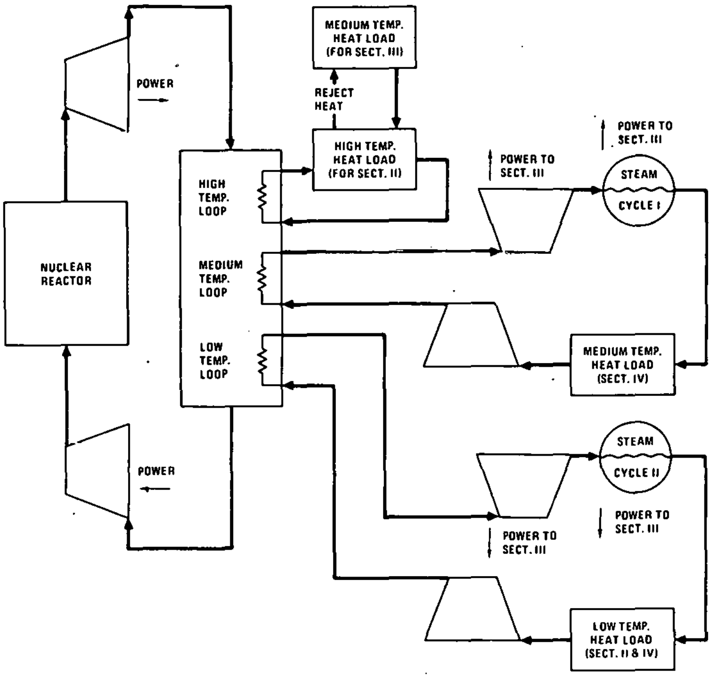

Fig. 7. Section V, Power Generation and Heat Transfer System

reaction of flowsheet Section II. Recovered heat from Section II is utilized to provide heat for the HI distillation and phosphoric acid concentration of flowsheet Section III. The intermediate temperature loop provides the heat for flowsheet Section IV, the HI decomposition. Power for flowsheet Section III is generated through a helium turbine and a steam cycle. The low temperature loop provides low value heat to flowsheet Sections II and IV, sulfuric acid concentration and HI distillation. Additional power for flowsheet Section III is generated through a helium turbine and a second, low-temperature steam cycle. Section I is heat self-sufficient and exports power to other sections.

BENCH SCALE TESTING

Bench-scale studies are the logical first step in the scaleup of the sulfur-iodine water-splitting process. The design of the bench-scale system is based on the results of extensive chemistry studies of the three basic reactions described earlier. The main objective of the bench-scale unit is the study of the cycle under continuous flow conditions and verification of flowsheet criteria by modeling the main solution reaction, product separation, and concentration and decomposition of H_2SO_4 and HI. The unit is divided into three subunits: Subunit I represents the main solution reaction, Subunit II represents the sulfuric acid concentration and decomposition, and Subunit III represents the HI concentration and decomposition. When completed, the system will operate at a production rate of $\sim 6.6 \times 10^{-5}$ m^3/s ($\sim$4 liters/min).

Subunit I

In this subunit, the main solution reaction is carried out. The H_2O, I_2, and SO_2 are injected in a contact reactor where the two acid phases are formed. The products are then passed into a gas separator, where the excess SO_2 is removed for recycle, and eventually into a liquid-liquid separator, where the two phases are separated and collected.

Subunit II

The H_2SO_4 phase from Subunit I is purified, concentrated, and pyrolized at temperatures up to 1144 K. Uncracked H_2SO_4 is recycled to the concentration column, and wet SO_2-O_2 product may then either be passed to a caustic scrub prior to metering and discharging or recycled to Subunit I without removal of O_2.

Subunit III

This subunit separates HI from the Lower Phase product of the main solution reaction (containing HI, I_2, and H_2O) by a treatment with concentrated H_3PO_4. The HI is then catalytically decomposed at moderate temperature.

All three subunits have been installed and data gathering has started. Results of the bench-scale experiments today indicate that flowsheet conditions can be achieved.

Closed Loop Cycle Demonstrator (CLCD)

As part of the bench-scale testing, DOE requested construction and operation of a separate, smaller unit, aimed at an earlier, simple demonstration of the cycle in a closed loop using recycled materials. The unit was designed and installed in 1978. Production of hydrogen was demonstrated in 1979, and recycle of all reaction products was achieved.

SOLAR STUDIES

Solar energy is receiving wide-spread attention as a means of reducing the demand for conventional fuel sources. Most activity has centered on the use of solar energy to provide heat and electricity. However, if solar energy could be utilized in the production of fuels and chemicals, the potential for solar energy to displace conventional sources of energy would be greatly expanded.

A study has been conducted at GA to examine the requirements for producing hydrogen by driving the sulfur-iodine thermochemical water-splitting cycle using solar energy. The process design involved conducting the high-temperature portion of the cycle (H_2SO_4 decomposition) during daylight and storing sufficient SO_2 and O_2 to provide enough recycle materials to run the other portions of the cycle continuously. Various types of solar collectors were evaluated for supplying heat for H_2SO_4 decomposition. A comparison of molten salt sensible heat storage and additional SO_2/O_2 storage was completed. The preferred energy storage method for driving the lower-temperature portions of the process at night is molten salt storage.

The purpose of the GA study was to evaluate coupling the sulfur-iodine cycle to solar energy sources. The specific objectives included the following:

1. Evaluating various types of solar energy collectors to determine those most suitable for supplying heat for the water-splitting cycle.
2. Designing a conceptual flowsheet that utilizes solar energy to drive the cycle, paying particular attention to coupling the

diurnal solar source to the continuous process energy demand.

3. Making recommendations for future development work.

PROCESS CONCEPT

The basic problem in coupling a solar heat source to water splitting is that of devising systems that can use the time-varying solar heat input to permit a uniform level of hydrogen production over a 24-h day. A thermochemical approach may readily lend itself to solar heating, because many intermediate compounds are storable. Figure 8 illustrates a possible system arrangement for the sulfur-iodine cycle. The mirror field is divided into high- and intermediate-temperature portions. The low-temperature solution reaction is conducted 24 h/day, producing H_2SO_4 and HI. The H_2SO_4 is stored, and during daylight, is pumped from storage, preheated, and then decomposed in the heat receiver of the high-temperature mirror field. The acid decomposition products are then heat exchanged with the incoming acid, the oxygen and water are removed, and the SO_2 is stored for use in the low-temperature solution reaction.

The intermediate-temperature mirror field heats a heat-transport fluid, such as a eutectic salt. During daylight the salt charges a heat-storage reservoir, generates steam to drive compressors and pumps, and provides heat for concentration and cracking of the HI to yield the hydrogen product and iodine for recycle to the main reaction. At night, the heat-transport fluid continues to perform these functions, but bypasses the heat receiver and is heated by the heat-storage reservoir.

A plant heat requirement of 400 MW(t) was selected, because it corresponded approximately to commercial-size solar plants being studied for electric power application, and it would result in a hydrogen plant of acceptable capacity with an estimated hydrogen output in the range of 4.64×10^5 m^3/day (1.64×10^7 ft^3/day).[1] This energy would be absorbed during daylight hours and used as necessary, with the balance being stored for use at night. Of this energy, 100 MW(t) would be needed during the day for high-temperature cracking of H_2SO_4 in the high-temperature solar receiver. As part of this study, several high-temperature central receivers were evaluated. A comparison of the central receivers considered, summarized in Table 3, showed that the cavity design was the preferred concept from the standpoint of thermal efficiency. However, structural requirements indicated a preference for designs with relatively small temperature gradients and large thermal capacitance. Hence, the fluidized bed concept may be competitive when both performance and structural aspects are considered. Therefore, these two concepts will be studied further in a second conceptual design phase to define specific receiver geometries. This would allow determination of the structural integrity of the concepts and the economic tradeoffs between the cavity and fluidized bed receivers.

A second phase of these design studies is underway. It will more clearly define the optimal plant size and carry out detailed evaluations of the energy storage systems.

CONCLUSIONS

Work carried out at GA on the sulfur-iodine thermochemical water-splitting process during the last year has resulted in significant accomplishments. Total recycle capability was demonstrated for the cycle, and major process improvements have

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[1] Based on 47% process efficiency, the higher heating value of hydrogen, standard conditions, and a nominal 8-h heat collection period.

resulted in an efficiency increase to 47%. It appears that 50% efficiency is well within reach.

The significant progress made in the development of the GA process gives confidence that this process will be shown as a viable, economic alternative for hydrogen production.

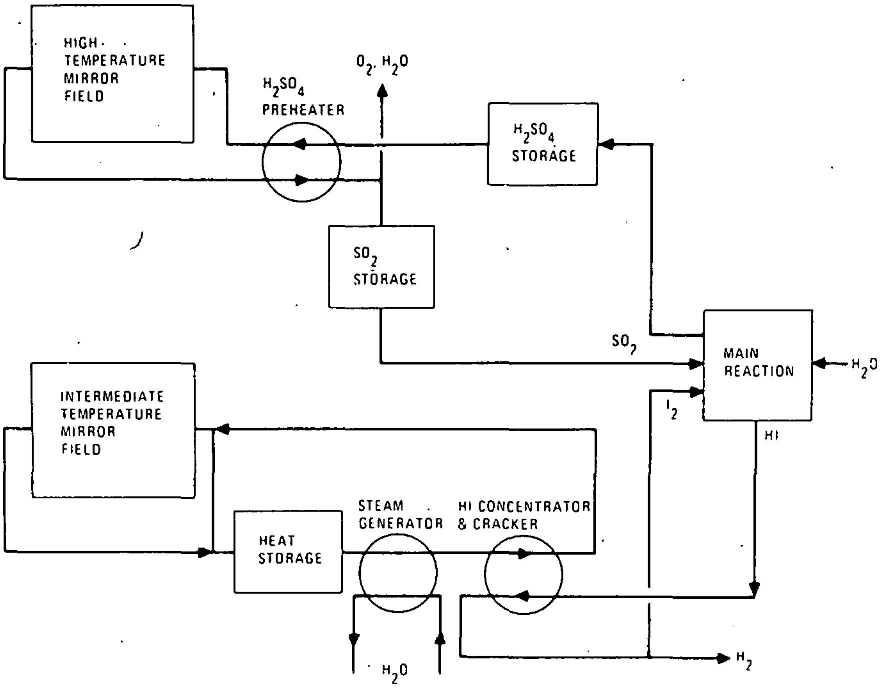

Fig. 8. Possible solar/thermochemical water-splitting configuration.

TABLE 3 Results for Central Receiver Designs

Receiver Design	Efficiencies (%)				Temperature Difference[a] (°C)			Thermal Capacitance, ρVC_p	Overall Receiver Size[b] (m)	Tube Size[c] (cm)
	Overall Efficiency	Reflective Loss	Reradiation Loss	Convective Loss	Tube Wall	Tube Diametral	Tube to Tube			
External	88	5	6	1	11	28	11	250	3.9D x 7.9H	1.27d x 0.025t
Cavity	97	1.5	1	0.5	11	28	10	250	3.0D x 5.5H	1.27d x 0.025t
Fluidized bed	88	9	1	2	6	0	3	50,000	7.0D x 9.1H	1.27d x 1.27t
Direct Absorption	68	5	25	2	6	0	3	50,000	7.0D x 10.9H	1.27d x 1.27t

(a) Estimates based on mean expected heat flux levels at tube outlet.

(b) D = receiver diameter; H = receiver height.

(c) d = tube diameter; t = tube wall thickness.

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