SolarPACES 2023, 29th International Conference on Concentrating Solar Power, Thermal, and Chemical Energy Systems

Solar Fuels and Chemical Commodities

https://doi.org/10.52825/solarpaces.v2i.891

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Published: 28 Aug. 2024

Allothermally Heated Reactors for Solar-Powered Implementation of Sulphur-Based Thermochemical Cycles

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Abstract. Catalytic sulphur trioxide splitting is the highest-temperature (650-950°C), endothermic step of several sulphur-based thermochemical cycles targeted to production of hydrogen or solid sulphur. Concentrated solar power tower plants are an attractive renewable energy source to provide the necessary heat. Furthermore, the development of solar receivers capable of delivering solid or gaseous heat transfer fluids at these temperature ranges enable the implementation of such endothermic reactions in allothermally-heated reactors/heat exchangers placed away from the solar receiver. In this context, a 2-kW laboratory-scale shell-and-tube reactor/heat exchanger to perform thermal sulphuric acid decomposition and catalytic sulphur trioxide splitting was in-house designed, built and tested with electrically heated bauxite particles, in the perspective of eventually coupling such a reactor with a centrifugal particle solar receiver. Thermal test runs demonstrated the in-principle feasibility of the concept. The temperatures reached were sufficient to ensure complete sulphuric acid evaporation. However, the ones in the SO_3 splitting zone were of the order of 750°C, high enough to demonstrate SO_3 splitting but not reaching the levels required for close-to-equilibrium conversion of the Fe_2O_3 catalyst system used (~ 850°C). An improved version of the reactor is under construction incorporating design modifications based on lessons learned from the test campaigns, in the perspective of scaling up the process.

Keywords: Thermochemical Cycles, Sulphur, Iron Oxide Catalysts

1. Introduction

Thermochemical cycles are a repeating set of two or more consecutive chemical reactions targeted to the synthesis of a specific chemical product or the storage and on-demand release of energy, involving intermediate compounds that are fully recycled at the end of the process. The cycles of the sulphur family recycle sulphur as the central element that appears in different compounds at changing oxidation state. Such cycles like the Hybrid Sulphur (HyS) and the Sulphur-Iodine (SI) cycle were originally conceived to produce hydrogen via water-splitting [1], but can be also modified to produce solid sulphur (SoSu) that can be combusted in air to produce high-temperature heat and hence can be used both as a renewable fuel as well as a seasonal solar energy storage medium [2]. These three Sulphur-based cycles share the common step of the decomposition reactions of sulphuric acid (Table 1, [3]): first thermally to steam and SO_3 (1a) and subsequently the catalytic SO_3 dissociation (splitting) to SO_2 and oxygen (1b), their highest-temperature (650-950°C) endothermic step. In the HyS cycle these two reactions are followed by the electrochemical oxidation of SO_2 with water to yield sulphuric acid and hydrogen (2a, 2b) in a so-called sulphur dioxide-depolarized electrolyzer (SDE). In the SI cycle the SO_2 produced in the first step reacts with water and iodine via the Bunsen reaction (3) to produce two acids, HI and H_2SO_4, that are subsequently respectively decomposed according to reaction (4) that produces Hydrogen and (1a, 1b) that re-generate SO_2. In the SoSu cycle, reactions (1a), (1b) are followed by the disproportionation of SO_2 into elemental sulphur (S) and H_2SO_4 (5). Elemental sulphur can then be on-demand combusted in air to produce high-temperature heat and SO_2 in (6). The SO_2 from combustion can be converted back into S and H_2SO_4 to close the cycle. That is exactly the reason why the stoichiometry of reactions (1a), (1b) is written differently ion this specific cycle and the industrially practiced reactions for sulphuric acid production (7) and (8), the (catalytic) oxidation of the combustion product SO_2 to SO_3 (reaction 7, the so-called contact process, reverse of 1b) and, finally, absorption of SO_3 in water and its reaction to sulphuric acid (reaction 6, reverse of 1a) are added in Table 1, to “close” the mass balance so that the “net” sum of reactants and products is zero. The valuable outcome of this cycle is not a chemical product, but the high-quality sulphur-combustion heat at temperatures in excess of 1200°C, suitable for combustion in gas turbines and allowing hence efficient combined cycle power generation. The SoSu cycle is flexible enough to be also operated on demand as an “open cycle” (i.e. without “closing” the mass balance in reactions 1a-7) employing/providing material feedstocks (H_2SO_4, S, SO_2) in combination with sulphuric acid production or with desulphurization of flue-gas or natural gas.

Table 1. *Chemical reactions of the Hybrid Sulphur (HyS), Sulphur-Iodine (SI) and Solid Sulphur (SoSu) thermochemical cycles (adapted from [3])*

Cycle Reactions	No	Stoichiometry	Temperature (°C)	ΔH^0 (kJ/mol S)
HyS cycle				
H_2SO_4 dissociation	1a	$H_2SO_4\,(g) \rightarrow H_2O(g) + SO_3(g)$	450-500	+98
SO_3 splitting	1b	$SO_3(g) \rightarrow SO_2(g) + ½\ O_2\,(g)$	650-950	+99
Electrolysis	2	$2\ H_2O + SO_2 \rightarrow H_2SO_4 + H_2\,(g)$	50	
Anode	2a	$SO_2 + 2\ H_2O \rightarrow H_2SO_4 + 2H^+ + 2e^-$		
Cathode	2b	$2H^+ + 2\ e^- \rightarrow H_2$		
SI cycle				
H_2SO_4 dissociation	1a	$H_2SO_4\,(g) \rightarrow H_2O(g) + SO_3(g)$	450-500	+98
SO_3 splitting	1b	$SO_3(g) \rightarrow SO_2(g) + ½\ O_2\,(g)$	650-950	+99
Bunsen reaction	3	$2H_2O + SO_2 + I_2 \rightarrow H_2SO_4 + 2HI$	25-125	-75
HI decomposition	4	$2HI \rightarrow I_2\,(g) + H_2\,(g)$	125-725	+12
SoSu cycle				
H_2SO_4 dissociation	1a	$3H_2SO_4(aq) \rightarrow 3H_2O(g) + 3SO_3(g)$	450 – 500	826
SO_3 splitting	1b	$3SO_3(g) \rightarrow 3/2\ O_2\,(g) + 3SO_2(g)$	650 – 1000	-300
SO_2 disproportionation	5	$3SO_2(g)+2H_2O(l)\rightarrow 2H_2SO_4(aq) + S(s)$	50 – 200	-254
S combustion	6	$S(l) + O_2(g) \rightarrow SO_2(g)$	500 – 1500	-297
Contact process	7	$SO_2(g) + 1/2\ O_2\,(g) \rightarrow SO_3(g)$		-99
Absorption	8	$SO_3(g) + H_2O(g) \rightarrow H_2SO_4(aq)$		-176

The two endothermic steps (1a), (1b) are performed together in sequence and can be driven by heat supplied via concentrated solar energy (CSE). In this context, various CSE-heated reactor concepts have been explored, including tubular reactors absorbing solar radiation on their external surface and transferring the heat to their internal one containing the catalyst wherein the reactant gas mixture flows [4] or with the SO_3 splitting catalysts coated on stationary, structured, direct absorbing, volumetric solar receivers from silicon carbide (SiC) foams and honeycombs [1]. Even though the latter concept was in principle operational, drawbacks included high re-radiation losses from the front surface of structured bodies and capability to maintain the high temperatures achieved only within a limited length along the porous receiver body, necessitating implementation of complex heat recovery architectures.

Alternatively, the solar receiver and the sulphuric acid dissociation reactor can be spatially de-coupled; the receiver can be employed to heat a heat transfer fluid (HTF) that can in turn be transported via insulated tubes to the reactor where the endothermic chemical reactions take place, in an approach known as "allothermal" heating, first coined by the nuclear energy research community to exploit the high temperature (in the range of 900°C) of the Helium gas stream from nuclear very high temperature reactors (VHTRs). Obviously, for analogous such solar operation, the receiver should be capable of heating the HTF at a similar level exceeding the temperature required for SO_3 splitting, i.e. ~ 850 - 900°C. Two recently developed such solar receivers employ as HTFs solid ceramic particles and gases, respectively. The first is a developed and patented by DLR centrifugal particle solar receiver (Centrec®) that has demonstrated capability of heating flowing sintered bauxite particle streams at temperature levels of the order of 900-950°C [5]. The other, developed by company SUNHELION, is based on direct absorption of thermal radiation by a gaseous HTF similarly to the well-known greenhouse gas effect [6] that can reach temperatures in the range of 1500°C. These developments can render the implementation of the two sulphuric acid decomposition steps feasible "allothermally" via the enthalpy of solar receiver-heated solid particles in a catalytic reactor/heat exchanger placed on ground level; in such a case structural materials and reactor design issues relevant to the corrosive characteristics of sulphuric acid chemistry at high temperatures can be much easier addressed.

2. Experimental

In the perspective of scaling up the H_2SO_4 dissociation/SO_3 splitting process and coupling it to a centrifugal particle solar receiver [5], extensive analysis described in previous works [7, 8] concluded to the selection of a shell-and-tube type reactor/heat exchanger to perform the two reactions downstream of the receiver with solar-receiver-heated bauxite particles as HTF flowing downwards in the shell side and providing the necessary heat for the SO_3 splitting reaction at the upper part of the reactor and for the thermal dissociation of sulphuric acid at the lower. In the tube side, rising sulphuric acid vapours come into contact with a non-moving catalyst bed that can contain the catalyst in structured forms like honeycombs or foams. A proof-of-concept, 2 kW shell-and-tube reactor/heat exchanger designed to be heated through bauxite particles flowing in the shell through an electrically- heated inclined belt feeder (Figure 1a) and comprised of six catalytic tubes (Figure 1b) was constructed. Details on the prior catalytic systems tested and on the reactor's design and construction were provided in previous publications ([3], [9], respectively). Thermocouples were placed on the surface of every tube at different positions to measure temperature distribution along them as shown in Figure 1c. One of the 6 tubes (tube 2) served as a reference tube being equipped with more thermocouples whereas the remaining 5 tubes had the same number of thermocouples placed at the same position. Besides, 12 thermocouples are placed on the parallelepiped shell side of the reactor, co-planar to the thermocouples on the tubes. Non-coated SiC foams of Ø24mm x 40mm were employed in the lower, sulphuric acid thermal dissociation zone as flow diffusers and heat transfer media upon which the H_2SO_4 vapors decompose according to reaction (1a). Similar, yet Fe_2O_3-coated SiC foams comprised the catalytic systems of choice for the shell-and-tube reactor since they have demonstrated reproducible near-equilibrium conversion at 850°C, under a broad range of sulphuric acid flow rates as well as minute pressure drop even under high catalyst loadings (35-45 wt. %) [3]. Three non-coated and five coated foams were placed in each one of the six tubes (Figure 1b). Aspects addressed in the design and construction included materials solutions for the manifolds for the injection of the sulphuric acid solution, evaporator tubes, sealants, special ceramic adhesives to join anti-corrosive metallic-to-ceramic tubing e.g. (stainless steel to SiC) and materials and reactor solutions to compensate for thermal expansion/contraction [9]. During the shutdown or emergency, the hot particles inside the reactor are vented out via an emergency exit (on the reactor door) into a water-cooled emergency container as per the schematic in Figure 1d. The unit was also equipped with an in-house developed customized UV-Vis spectrometry system with a specifically designed corrosion and high-temperature resistant gas cell, for on-line analysis of produced SO_2. The produced gas is analysed and neutralised at the outlet of the reactor. Hereafter, the gas stream flows completely into the scrubber. The particle heating system has been described in detail previously [9]; briefly it consists of a 100-liters-volume particle container and a 1-meter-long, 35°-inclined heater tube wrapped with resistively heated elements and encapsulated with insulation. The overall test rig is shown in Figure 1e.

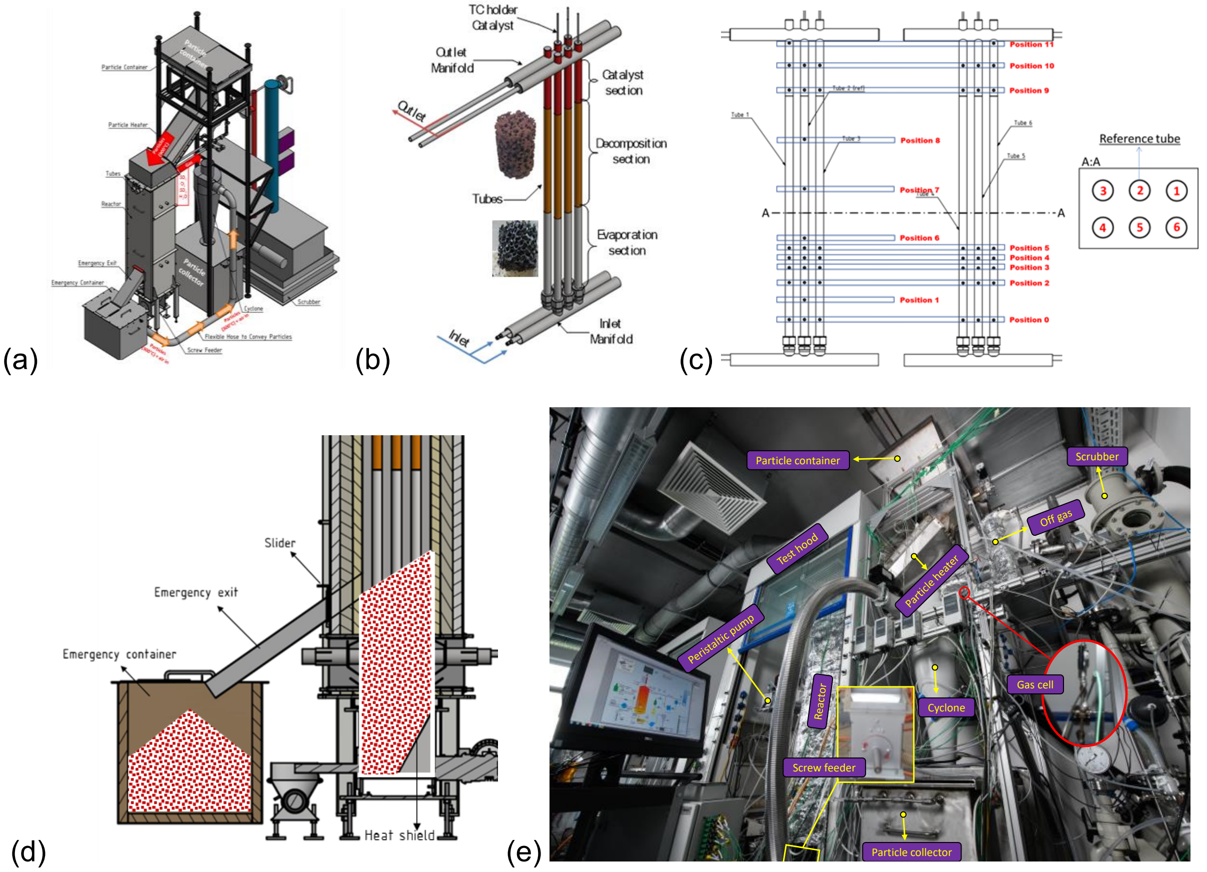


Figure 1. *(a) CAD of lab-scale particles-heated reactor/heat exchanger test setup (from [9]); (b) schematic of six reactor tubes included in the actual reactor within two 3-tube bundles and photographs of typical non-coated and* Fe_2O_3*-coated SiC foams used in the evaporation and catalytic sections respectively; (c) thermocouple positions for temperature measurements along the tubes; (d) operation schematic of side safety exit gate for hot particles stream drain (from [9]); (e) overall lab-scale insulated particles-heated reactor/heat exchanger test setup unit with particles heater and feeder and all necessary peripherals.*

3. Results

3.1 Thermal-only tests

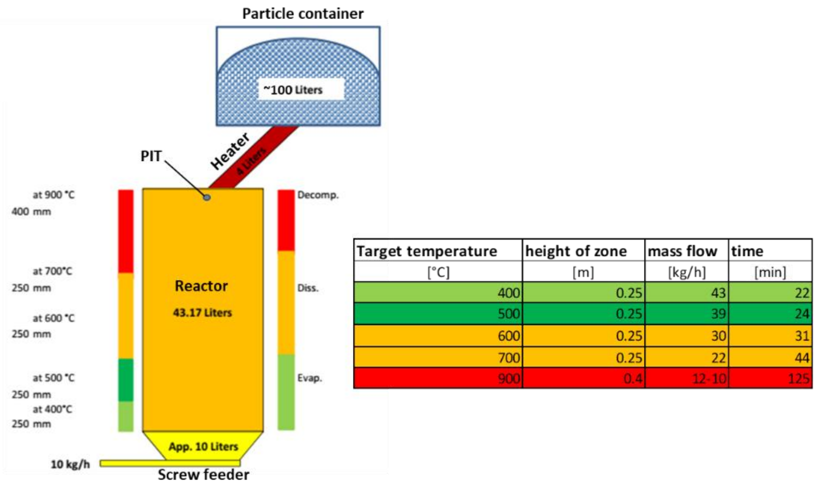
To study the temperature distribution within the reactor and to verify the target temperatures in the evaporation and catalyst zones, several thermal tests were conducted with air as process fluid within the tubes prior to any chemical tests. The particle heater and the reactor performance were thermally tested and the critical temperatures like particle inlet temperature

(PIT), temperatures at position 1 (beginning of the tubes), position 5 (end of the evaporator zone) and position 11 (top tip of the catalyst) were reviewed during multiple thermal tests. During the start-up of every experiment, the reactor shell is filled completely with cold particles. Later when the particle heater is turned on, the screw feeder (at the bottom of the reactor, Figure 1a) enables the hot particles into the reactor by replacing the cold particles. The speed of the screw feeder defines the residence time of the hot particles falling into the reactor. The test plan has been evolved from test plan 1 to test plan 3 (TP1 – TP3) as shown in Table 2 and explained in detail below, according to the results obtained and the lessons learned at each stage.

***Table 2**. Test plan development*

Plan	Description
Test plan 1 (TP1)	Stepwise particle heated bed
Test plan 2 (TP2)	Overnight heating + TP1
Test plan 3 (TP3)	Overnight heating + Day operation (constant test parameters)

As per the very initial test plan TP1, a stepwise particle heated bed was planned by regulating the particle flow with respect to time and distance covered along the reactor (height) in such a way that the particles to be sufficiently hot when reaching the evaporation zone as shown in Figure 2. By adjusting the particle flow rate, the target PIT temperature can be achieved and by holding the particle flow for certain duration, the particles can fill required heights within the reactor. In reality, the measured target temperatures might deviate from the below calculated target temperatures because of the process fluid (in case of these tests this fluid was air rather than sulphuric acid) and heat losses.



Target temperature	height of zone	mass flow	time
[°C]	[m]	[kg/h]	[min]
400	0.25	43	22
500	0.25	39	24
600	0.25	30	31
700	0.25	22	44
900	0.4	12-10	125

***Figure 2**. Test plan 1 (TP1) – Stepwise particle heated bed.*

The temperature distribution on reactor tube 2 (used as reference as already mentioned) during TP1 is shown in Figure 3. The particle flow rate was reduced from 43 kg/h to 10 kg/h in a timespan of approx. 2:30 hours and the PIT at the outlet of the particle heater has recorded a corresponding progressive increase reaching a maximum temperature of 890°C with a particle flow rate of approximately 10 kg/h. The temperature difference between PIT and RT2-pos. 11 (tip of catalyst) was approx. 150°C by the end of the test run due to the heat loss at the interface between the heater and the reactor. After approximately 4 operational hours, the lowermost thermocouple on the reactor tube in the evaporator zone, RT-Pos.0 has recorded a temperature of 100°C, still exhibiting a rising trend. Although the temperatures at RT-Pos.11 are higher than 700°C, the temperature in the evaporation section needs to be higher than the boiling point of sulphuric acid (approx. 337°C for 98 wt% concentrated H_2SO_4). It was clear that under these conditions the temperature reached was insufficient for the sulphuric acid splitting and moreover the heat of the particles is essentially used to preheat the reactor. Therefore, the test plan was modified to the one denoted with TP2.

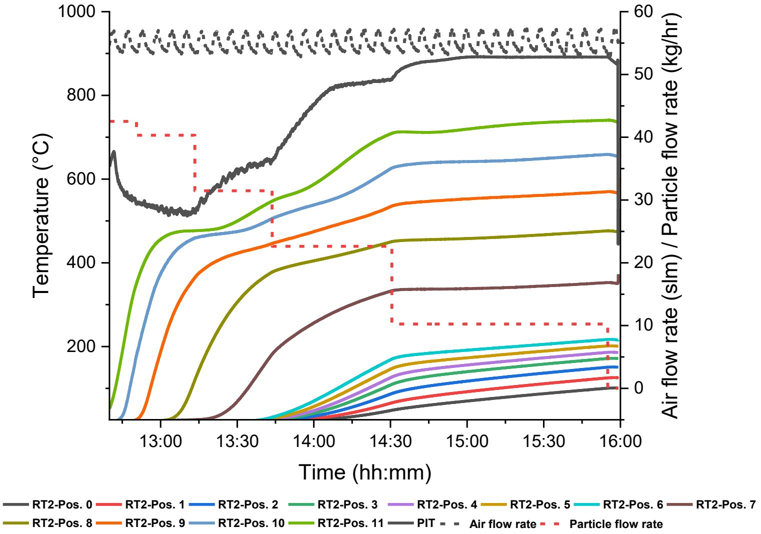


***Figure 3**. Temperature distribution on Tube 2 according to TP1*

Test plan 2 (TP2) is a combination of TP1 with so-called “overnight” heating. The latter refers to running the test setup overnight with a constant, low flow rate of particles heated at a relatively low-to-intermediate temperature, namely 400°C, followed by TP1 (stepwise particle heated bed) next day morning. The operation parameters are tabulated in Table 3.

***Table 3.** Test plan 2 (overnight heating and TP1)*

Overnight heating			**Day operation**		
Particle flow rate (kg/h)	**Air flow rate (SLM)**	**Particle heater set point**	**Particle flow rate (kg/h)**	**Air flow rate (SLM)**	**Particle heater set point**
4	21	400 °C	43 to 10	42 to 43	900 °C

The temperature distributions on reactor tube 2 (RT2) during the overnight heating and the subsequent day operation according to TP2 are shown in Figure 4 and Figure 5 respectively. As the speed of the particle flow is low, the time required for the hot particles from heater to reach pos.0 is also longer. As shown in Figure 4, the evaporator section was not exposed to high enough temperatures even after overnight operation of the test rig. Soon after initiating the day operation according to the test plan, the temperatures on the surface of the reactor tube 2 started to rise as shown in Figure 5. The PIT has raised from 400°C to 890°C due to change in flow rate and it remained constant from and below 10 kg/h particle flow rate. The temperatures of pos.11 are suitable for sulphuric acid splitting but those at pos.6 are still not favourable for evaporation. Overall, the temperature measurements from TP2 concluded that the overnight heating had a comparatively positive effect on the day operation enabling to attain higher temperatures along the overall length of the tube but the repeated change in flow rates (particularly particle flow rate) is affecting the temperature distribution within the test rig. Therefore, TP2 was further modified to a final test plan (TP3) in which the optimal parameters are kept constant throughout the experimental test.

Test plan 3 (TP3) is actually a fine-tuned version of TP2. The particle flow rate in TP3 is always kept constant to approx. 10 kg/hr unlike in TP1 and TP2 because all the set of tests and calculations determined that with this rate of 10 kg/h, the heater could deliver constant PIT of 890°C, higher than the targeted 850°C for the SO_3 splitting step where the employed catalysts have demonstrated near-equilibrium conversion. The operation parameters of TP3 are shown in Table 4; they are identical during overnight heating and day operation.

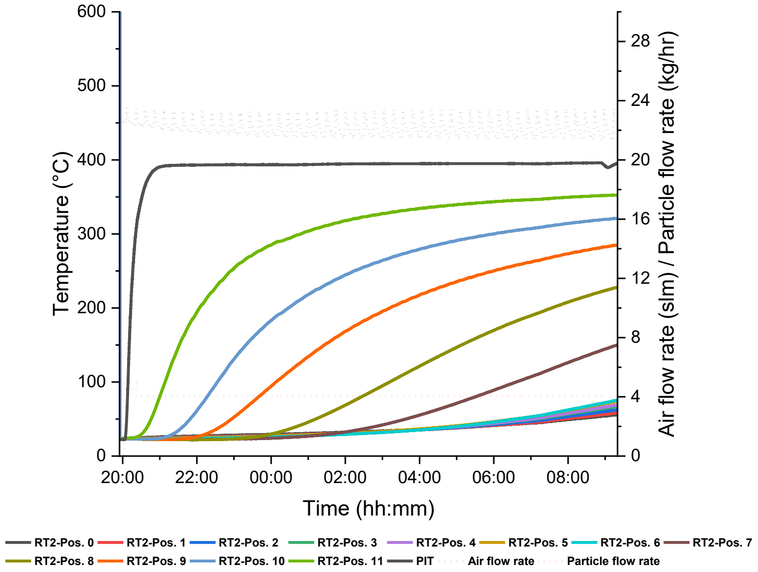


Figure 4. *Temperature distribution during overnight heating on reactor tube 2 at every position according to TP2 with particles flow rate of 4 kg/h and air flow rate of 22.5 slm.*

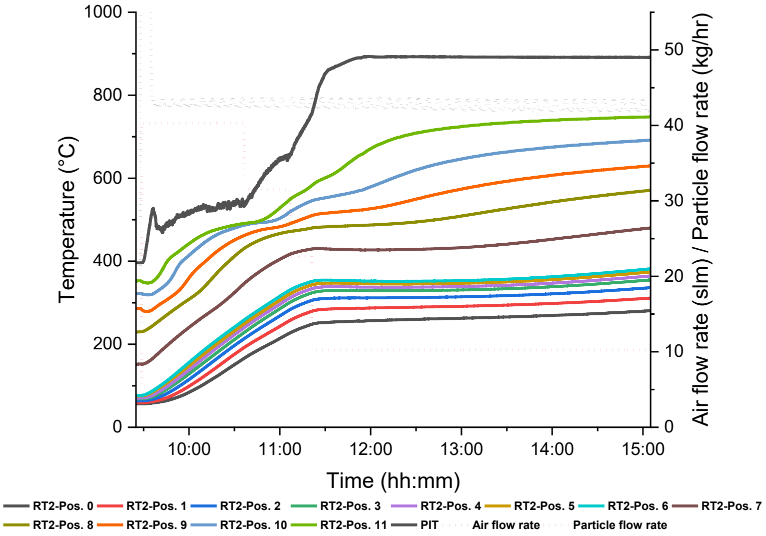


Figure 5. *Temperature distribution on reactor tube 2 at every position according to TP2 during day operation.*

Table 4. *Test plan 3*

Overnight heating			**Day operation**		
Particle flow rate (kg/h)	**Air flow rate (SLM)**	**Particle heater set point (H1-H4)**	**Particle flow rate (kg/h)**	**Air flow rate (SLM)**	**Particle heater set point (H1-H4)**
10	35	900 °C	10	35	900 °C

The temperature distribution on reference tube RT2 is shown in Figure 6. The air flow was interrupted soon after 2 hours of test start due to the malfunction of the air supply system. There was no air flow into the tubes during the overnight heating. This was an unfortunate harsh test on the reactor which however, resulted in a positive result in the sense that no physical damage of tubes occurred, as confirmed with a leak test. The air flow was manually resumed again during the day operation which resulted in the small drop of temperatures recorded throughout the tube. The decomposer zone had already reached more than 750°C during overnight operation. The temperature of pos. 11 (tip of catalyst) was close to 800°C during the day operation, whereas the pos. 0 of the evaporator zone was above 400 °C during the start of the day operation. In conclusion, the temperature distribution of the evaporation zone was improved by a factor of 2 using TP3 compared to TP2.

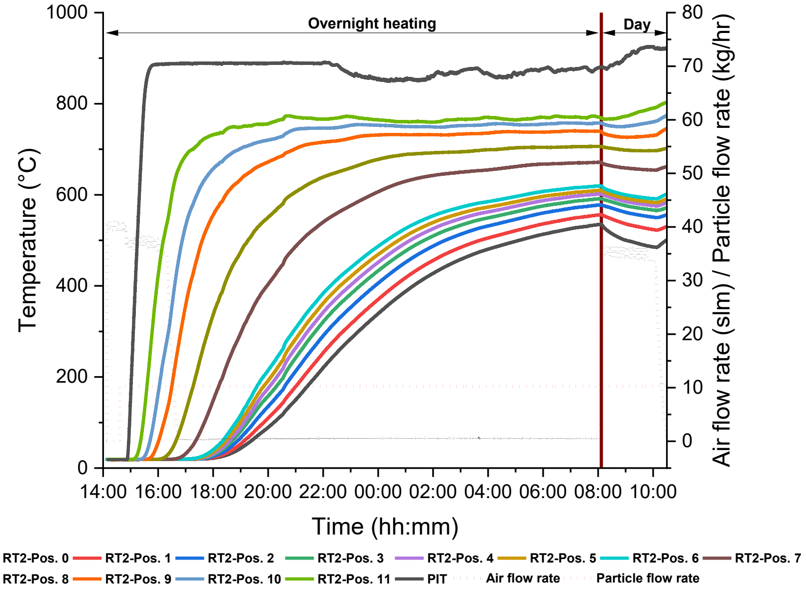
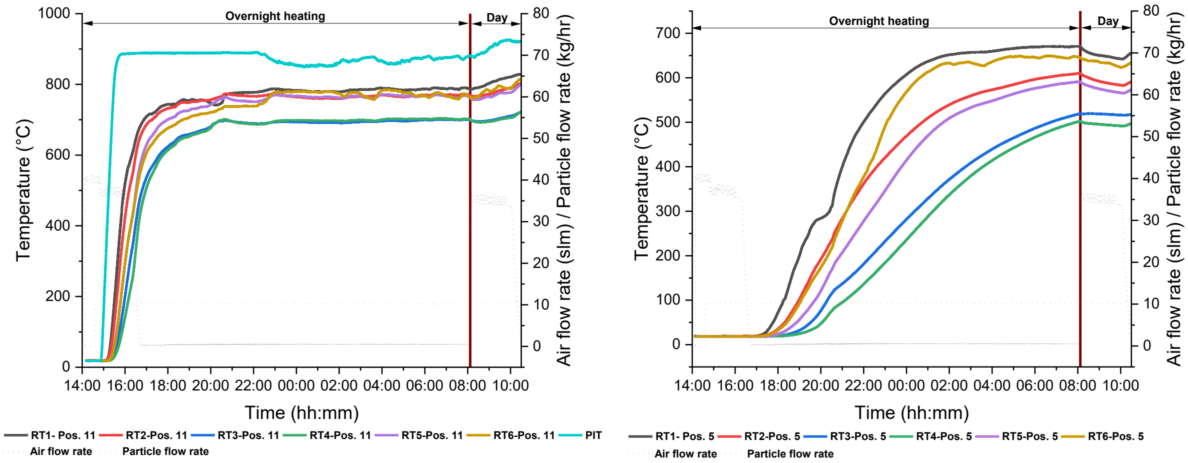


***Figure 6**. Temperature distribution on reactor tube 2 at every position according to TP3*

However, despite the fact that the reference reactor tube reached the targeted temperature, the temperatures of the six tubes at the same “vertical” position were not equal as shown on the relevant graphs of Figures 7a and 7b, where the temperatures at the top of the catalytic zone (TC at position 11) and at the tip of evaporation zone (TC at position 5) are compared. At position 11, tube 1 has reached the highest temperature (approx. 800°C), but these of tubes 3 and 4 are almost 100°C lower than that of tube 1. The particle flow pattern affects the temperatures of different reactor tubes at a single vertical position of plane. The flow pattern of the reactor setup is shown in Figure 1d and the temperature differences of the tubes at the same horizontal position can be minimised by altering the flow pattern (which can be achieved by modifying the heat shield in Figure 1d.



(a) At Position 11

(b) At position 5

***Figure 7**. Temperature distribution on all reactor tubes (RT1-RT6) for an experiment according to TP3.*

4. Conclusions and outlook

The development of solar receivers capable of delivering high-temperature (> 850°C) heat transfer fluids, provides for implementation of endothermic reactions via concentrated solar energy in suitably designed allothermally-heated reactors/heat exchangers placed away from the solar receiver. Such a 2-kW laboratory-scale reactor to perform thermal sulphuric acid decomposition and catalytic sulphur trioxide splitting was in-house designed, built and tested with electrically heated bauxite particles, demonstrating the in-principle feasibility of the proposed concept.

The reactor successfully underwent multiple thermal test runs demonstrating the in-principle feasibility of both sulphuric acid decomposition as well as of SO_3 splitting. The temperatures reached in the lower sulphuric acid evaporation zone (~ 400°C) were sufficient to ensure complete sulphuric acid evaporation. However, the ones reached in the upper SO_3 splitting zone were of the order of 750°C, high enough to demonstrate SO_3 splitting but not reaching the levels required for satisfactory conversion (~ 850°C). An improved version of the reactor is under construction incorporating design modifications based on lessons learned from the test campaigns, in the perspective of scaling up the process and coupling it to a centrifugal particle solar receiver operating on a solar tower.

Data availability statement

Data will be made available on request.

Author contributions

V. K. Thanda: Conceptualization, methodology, software, validation, formal analysis, investigation, resources; D. Thomey: Conceptualization, methodology, formal analysis, visualization, writing—review and editing, project administration, funding acquisition; M. Wullenkord: Conceptualization, methodology, software, validation, formal analysis, investigation, resources; K.-P. Eßer: Investigation, validation; C. Agrafiotis: Conceptualization, methodology, visualization, writing—original draft preparation, writing—review and editing, project administration, funding acquisition; D. Dimitrakis: Conceptualization, methodology, investigation, visualization, writing—review and editing; M. Roeb: Resources, funding acquisition, supervision; C. Sattler: Resources, supervision.

Competing interests

The authors declare that they have no competing interests.

Funding

This work was performed within the project “PEGASUS: Power Generation by Solar Particle Receiver Driven Sulphur Storage Cycle”. The project has received funding from the European Union’s Horizon 2020 research and innovation programme under grant agreement No 727540. The preparation of the paper was performed within the project “HySelect: Efficient water splitting via a flexible solar-powered Hybrid thermochemical-Sulphur dioxide depolarized Electrolysis Cycle”. The project receives funding from the European Horizon Europe research and innovation programme under grant agreement No 101101498.

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