

## THERMAL DECOMPOSITION OF HYDROGEN PEROXIDE IN THE PRESENCE OF SULFURIC ACID

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Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) is popularly employed as a reaction reagent in cleaning processes for the chemical industry and semiconductor plants. By using differential scanning calorimetry (DSC) and vent sizing package 2 (VSP2), this study focused on the thermal decomposition reaction of H<sub>2</sub>O<sub>2</sub> mixed with sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) with low (0.1, 0.5 and 1.0 N), and high concentrations of 96 mass%, respectively. Thermokinetic data, such as exothermic onset temperature ( $T_0$ ), heat of decomposition ( $\Delta H_d$ ), pressure rise rate ( $dP/dt$ ), and self-heating rate ( $dT/dt$ ), were obtained and assessed by the DSC and VSP2 experiments. From the thermal decomposition reaction on various concentrations of H<sub>2</sub>SO<sub>4</sub>, the experimental data of  $T_0$ ,  $\Delta H$ ,  $dP/dt$ , and  $dT/dt$  were obtained. Comparisons of the reactivity for H<sub>2</sub>O<sub>2</sub> and H<sub>2</sub>O<sub>2</sub> mixed with H<sub>2</sub>SO<sub>4</sub> (lower and higher concentrations) were evaluated to corroborate the decomposition reaction in these systems.

**Keywords:** DSC, hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), thermal decomposition reaction, vent sizing package 2 (VSP2)

### Introduction

Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) is the most common peroxide in many applications throughout a wide variety of industries, as pure H<sub>2</sub>O<sub>2</sub> and its aqueous solutions resemble water in physical appearance. It is also widely employed in semiconductor manufacturing processes because it is essentially active to oxidizing agents for organic and inorganic cleaning processing [1]. Many thermal runaway incidents have been caused by H<sub>2</sub>O<sub>2</sub> due to the peroxy function group, –O–O–, which is essentially unstable and active. H<sub>2</sub>O<sub>2</sub> is also sensitive to thermal sources and is incompatible with many materials, such as acids, bases, metals and ions [2–5].

Table 1 gathers accidents caused by H<sub>2</sub>O<sub>2</sub> in the USA and New Zealand [6]. Hydrogen peroxide containing impurities, which can exist in a non-controlled environment, has caused many explosions [7]. H<sub>2</sub>O<sub>2</sub> was mixed with sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) in a vacuum truck and an explosion occurred at that moment. The cause of the accident is suspected to be that H<sub>2</sub>SO<sub>4</sub>, which was mixed, accelerated catalytic decomposition of H<sub>2</sub>O<sub>2</sub> and led to a runaway reaction. When the self-heating rate by the decomposition exceeds a heat radiation rate toward the outside of the container, thermal storage occurs. Rising temperature

accelerates the decomposition, leading to a thermal explosion after an induction period [8, 9].

The piranha solution (Caro's solution), which is applied to remove organic residues from substrates, has caused many accidents, because of its dangerous chemical mixtures. However, the H<sub>2</sub>O<sub>2</sub> should not be added a great quantity more than the acid to avoid explosion of the mixture. Typical application is a 3:1 mixture of H<sub>2</sub>SO<sub>4</sub> (96 mass%) with H<sub>2</sub>O<sub>2</sub> (20 mass%) [10]. In this study, the mechanism contrasted with thermokinetics of decomposition reaction for H<sub>2</sub>O<sub>2</sub> in the presence of H<sub>2</sub>SO<sub>4</sub> did compare with the thermal analysis and adiabatic runaway data, exothermic onset temperature ( $T_0$ ), heat of decomposition ( $\Delta H_d$ ), self-heating rate ( $dT/dt$ ), pressure rise rate ( $dP/dt$ ), etc., in these systems.

### Experimental

#### Samples

This reaction was focused on thermal decomposition for H<sub>2</sub>O<sub>2</sub> present with H<sub>2</sub>SO<sub>4</sub>. We selected the decomposition of H<sub>2</sub>O<sub>2</sub> (20 mass%, 30 mL) mixed with H<sub>2</sub>SO<sub>4</sub> (0.1, 0.5 and 1.0 N, 10 mL) and H<sub>2</sub>O<sub>2</sub> (20 mass%, 2, 4 and 6 mL) mixed with H<sub>2</sub>SO<sub>4</sub> (96 mass%, 6 mL).

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**Table 1** Selected accidents caused by H<sub>2</sub>O<sub>2</sub> in USA and New Zealand since 2004 [6]

Date	Location	Fatalities	Injuries	Remarks
01/27/2005	San Bernardino, California, USA	N/A	0	tank-container
01/13/2005	Vacaville, California, USA	N/A	0	road-tanker
01/08/2005	Seabrook, Texas, USA	N/A	0	rail-tanker
12/23/2004	South Dunedin, New Zealand	N/A	N/A	tank-container
04/24/2004	Dallas, Texas, USA	N/A	N/A	tank-container
04/05/2004	Mobile, Alabama, USA	N/A	N/A	waste-storage
02/17/2004	Nr. Natchez, Mississippi, USA	N/A	N/A	transport

N/A: not applicable

### Differential scanning calorimetry (DSC)

A temperature-programmed screening experiment was performed (Mettler TA8000 system). The test cell could withstand high pressure to approximately 100 bar (DSC 821°). The experiment was conducted between 30 to 300°C; the scanning rate selected for the temperature-programmed ramp was 4°C min<sup>-1</sup> to maintain better thermal equilibrium [11].

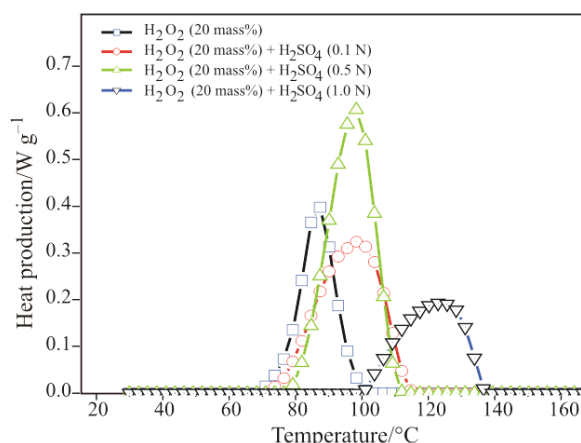
### Vent sizing package 2 (VSP2)

VSP2, developed by Fauske and Associates, Inc. (FAI) [12] is a highly sensitive calorimeter that can obtain thermokinetic and thermal decomposition data, such as temperature and pressure traces with respect to time in an adiabatic calorimeter system by PC-control. Under heating conditions, the main heater will turn on to heat the sample to a pre-set temperature, then turn on a guard heater to maintain an adiabatic environment. The standard operating procedure is repeated by automatic heat–wait–search (H–W–S) mode. To adequately protect the normal operation of this apparatus and avoid bursting the test cell and missing the end of exothermic data, 20 mass% of H<sub>2</sub>O<sub>2</sub> was prudently chosen for the experiments. We injected H<sub>2</sub>O<sub>2</sub> (20 mass%, 30 mL) mixed with H<sub>2</sub>SO<sub>4</sub> (different concentrations, 10 mL) into the test cell and H<sub>2</sub>O<sub>2</sub> (20 mass%, different volumes) mixed with 96 mass% H<sub>2</sub>SO<sub>4</sub> into the spherical bomb.

## Results and discussion

### Thermal analysis by DSC

The  $T_0$  and  $\Delta H_d$  of H<sub>2</sub>O<sub>2</sub> reacting with H<sub>2</sub>SO<sub>4</sub> (lower concentration) could be obtained and calculated from the peak areas by DSC, which was performed to provide different experimental results for thermal decomposition. The DSC scans under continuous heating conditions show the presence of one exothermic peak.

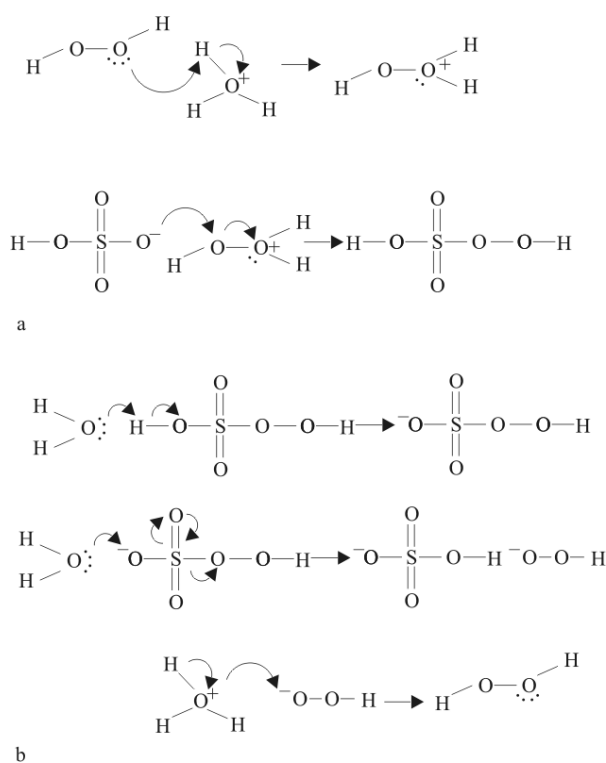


**Fig. 1** Comparison of heat production vs. temperature by DSC tests on H<sub>2</sub>O<sub>2</sub> and various concentrations of H<sub>2</sub>SO<sub>4</sub> at heating rate of 4°C min<sup>-1</sup>

Figure 1 depicts the typical heat flow curve that was obtained through DSC of the thermal decomposition for H<sub>2</sub>O<sub>2</sub> with various concentrations of H<sub>2</sub>SO<sub>4</sub>. The  $T_0$  value of H<sub>2</sub>O<sub>2</sub> acquired was about 67°C which is lower than the solutions of H<sub>2</sub>O<sub>2</sub> mixed with H<sub>2</sub>SO<sub>4</sub> (0.1, 0.5 and 1.0 N). Table 2, which is obtained through DSC data, displays the exothermic data of H<sub>2</sub>O<sub>2</sub> compared with various concentrations of H<sub>2</sub>SO<sub>4</sub> (lower concentration). According to the data from Fig. 1 and Table 2, the H<sub>2</sub>O<sub>2</sub> mixed with various concentrations of H<sub>2</sub>SO<sub>4</sub> (0.1, 0.5 and 1.0 N) reveals that  $\Delta H_d$  was lower than H<sub>2</sub>O<sub>2</sub> alone. From thermal analysis by DSC trials, we found that H<sub>2</sub>O<sub>2</sub> itself will react more dangerously than with H<sub>2</sub>SO<sub>4</sub> (0.1, 0.5 and 1.0 N). Figures 2a and b delineate our proposed mechanisms of the reaction when the decomposition of H<sub>2</sub>O<sub>2</sub> is mixed with the H<sub>2</sub>SO<sub>4</sub> (lower concentration). Figure 2a illustrates that the H<sub>2</sub>O<sub>2</sub> decomposed to H<sub>3</sub>O<sub>2</sub><sup>+</sup> and H<sub>3</sub>O<sup>+</sup>, in the solution and H<sub>2</sub>SO<sub>4</sub> led to HSO<sub>4</sub><sup>-</sup> in the solution. The H<sub>2</sub>SO<sub>4</sub><sup>-</sup>, integrated with H<sub>3</sub>O<sub>2</sub><sup>+</sup>, shifted to H<sub>2</sub>SO<sub>5</sub> [8]. Figure 2b shows that the solution, which included the H<sub>2</sub>O<sub>2</sub> and the H<sub>2</sub>SO<sub>4</sub>, had a great quantity of H<sub>2</sub>O. The H<sub>2</sub>SO<sub>5</sub> combined with the H<sub>2</sub>O shifted to HSO<sub>5</sub><sup>-</sup> in a moment. The HSO<sub>5</sub><sup>-</sup> combined with H<sub>2</sub>O and decom-

**Table 2** Experimental data of decomposition reaction for H<sub>2</sub>O<sub>2</sub> mixed with H<sub>2</sub>SO<sub>4</sub> (0.1, 0.5 and 1.0 N) by DSC

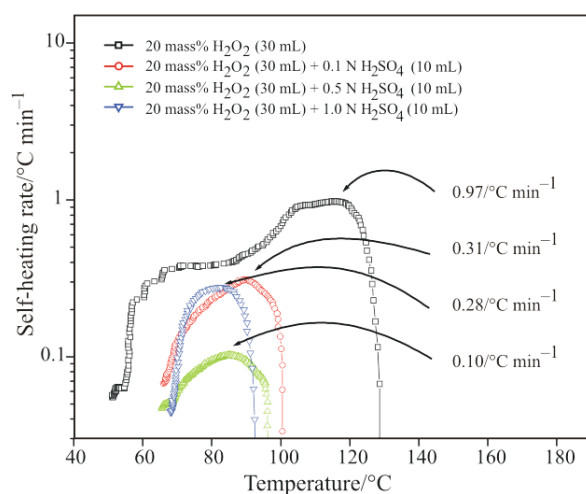
Thermal analysis						
Sample				Thermal data		
H <sub>2</sub> O <sub>2</sub>		H <sub>2</sub> SO <sub>4</sub>		Scanning rate/ °C min <sup>-1</sup>	T <sub>0</sub> / °C	ΔH <sub>d</sub> / J g <sup>-1</sup>
Conc./mass%	Mass/mg	Conc./N	Mass/mg			
20	2.47	0.0	0.00	4	67	162
20	2.01	0.1	0.91	4	70	154
20	2.54	0.5	0.70	4	76	99
20	2.25	1.0	0.73	4	98	110


**Fig. 2** Proposed reaction mechanisms for H<sub>2</sub>O<sub>2</sub> mixing with the lower concentrations of H<sub>2</sub>SO<sub>4</sub> (0.1, 0.5 and 1.0 N):  
 a – H<sub>2</sub>O<sub>2</sub> combined with H<sub>2</sub>SO<sub>4</sub> shifts to H<sub>2</sub>SO<sub>5</sub>;  
 b – H<sub>2</sub>SO<sub>5</sub> shifts to H<sub>2</sub>O<sub>2</sub> [10]

posed to HSO<sub>4</sub><sup>-</sup> and HOO<sup>-</sup>, quickly. The final step indicated the HOO<sup>-</sup>, after being mixed with H<sub>3</sub>O<sup>+</sup>, returned to H<sub>2</sub>O<sub>2</sub>. According to Figs 1 and 2, the concentration of H<sub>2</sub>SO<sub>4</sub> could rise and the reaction rate became slower than the H<sub>2</sub>O<sub>2</sub> alone.

#### Thermal decomposition of adiabatic experiments

Adiabatic thermal decomposition reaction data could be detected by VSP2 to obtain the thermokinetic parameters such as T<sub>0</sub>, ΔH<sub>d</sub>, dP/dt, and dT/dt, and so on. The characteristic curves of self-heating rate and pressure rise rate of H<sub>2</sub>O<sub>2</sub> reacting with H<sub>2</sub>SO<sub>4</sub> (0.1, 0.5 and 1.0 N) are recorded in Figs 3 and 4. From Figs 3

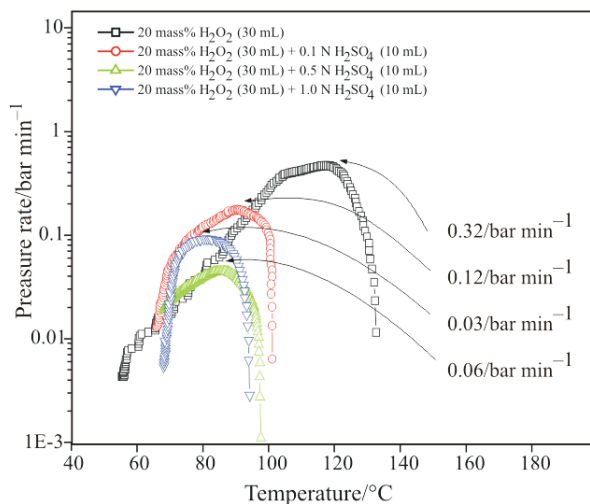

**Fig. 3** Self-heating rate vs. temperature for thermal decomposition of H<sub>2</sub>O<sub>2</sub> and H<sub>2</sub>O<sub>2</sub> reacting with H<sub>2</sub>SO<sub>4</sub> (0.1, 0.5 and 1.0 N), respectively

and 4, when H<sub>2</sub>SO<sub>4</sub> (0.1, 0.5 and 1.0 N) concentrations are increased, the self-heating rate and pressure rise rate can be lower than that of H<sub>2</sub>O<sub>2</sub>. VSP2 experimental data that were acquired by the tests of H<sub>2</sub>O<sub>2</sub> mixed with different concentrations H<sub>2</sub>SO<sub>4</sub> are summarized in Table 3. According to Table 3, the H<sub>2</sub>SO<sub>4</sub> could raise T<sub>0</sub> of the thermal decomposition for H<sub>2</sub>O<sub>2</sub>, and thermokinetic parameters of maximum temperature (T<sub>max</sub>), maximum pressure (P<sub>max</sub>), dP/dt<sub>max</sub>, and dT/dt<sub>max</sub> became smaller with increasing concentration of H<sub>2</sub>SO<sub>4</sub> (0.1, 0.5 and 1.0 N).

There are many possible reaction pathways with respect to the decomposition of H<sub>2</sub>O<sub>2</sub>. In experiments, the H<sub>2</sub>O<sub>2</sub> decomposition reaction was mixed with the H<sub>2</sub>SO<sub>4</sub> (0.1, 0.5 and 1.0 N) solution. Meanwhile, it was verified that the H<sub>2</sub>O<sub>2</sub> reacting with the H<sub>2</sub>SO<sub>4</sub> (0.1, 0.5 and 1.0 N) shifted to H<sub>2</sub>SO<sub>5</sub>. According to Fig. 2, we assumed that the decomposition of the H<sub>2</sub>SO<sub>5</sub> reacts with water and changes to H<sub>2</sub>O<sub>2</sub>. This is the reason why the reaction was slower. By comparison of the data for thermal analysis and adiabatic decomposition reactions from DSC and VSP2 calorimetry, we proposed the decomposition rate of H<sub>2</sub>O<sub>2</sub>

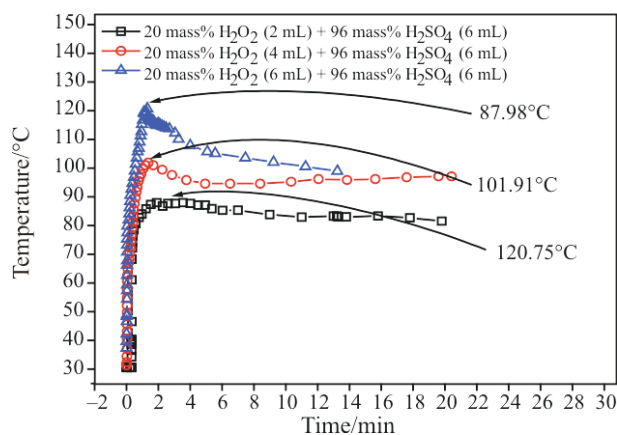
was increased with the  $\text{H}_2\text{SO}_4$  under the lower concentration. According to Figs 2–4 and Table 3, it could have raised the concentration of  $\text{H}_2\text{SO}_4$  and the reaction rate became slower than the  $\text{H}_2\text{O}_2$  alone.

During the VSP2 test,  $\text{H}_2\text{O}_2$  mixed with the  $\text{H}_2\text{SO}_4$  (96 mass%) caused a violent reaction in a short period resulting in bursting along the weld of the test cell, only the adiabatic data was influenced by  $\text{H}_2\text{SO}_4$  (lower concentration). Accordingly, other sets of data were obtained by using spherical test bombs equipped with VSP2. Figures 5 and 6 show that the reaction of  $\text{H}_2\text{O}_2$  mixed with  $\text{H}_2\text{SO}_4$  instantly generated high temperature and pressure during the test. VSP2 experimental data of the spherical bomb was received by the tests of  $\text{H}_2\text{O}_2$  mixed with  $\text{H}_2\text{SO}_4$  (96 mass%), as summarized in Table 4. According to Table 4, maximum of pressure rise rate  $(dP/dt)_{\max}$  and maximum of temperature rise rate  $(dT/dt)_{\max}$  of the  $\text{H}_2\text{SO}_4$  (96 mass%) could rise rapidly during a runaway reaction. Figures 7a–c show the proposed and modified reaction mechanisms of the decomposition for  $\text{H}_2\text{O}_2$  mixed with the  $\text{H}_2\text{SO}_4$  (higher concentration). Figure 7a illustrates the  $\text{H}_2\text{SO}_4$ , when integrated with  $\text{H}_3\text{O}_2^+$  shifted to  $\text{H}_2\text{SO}_5$  [8]. Figure 7b shows that the solution, which

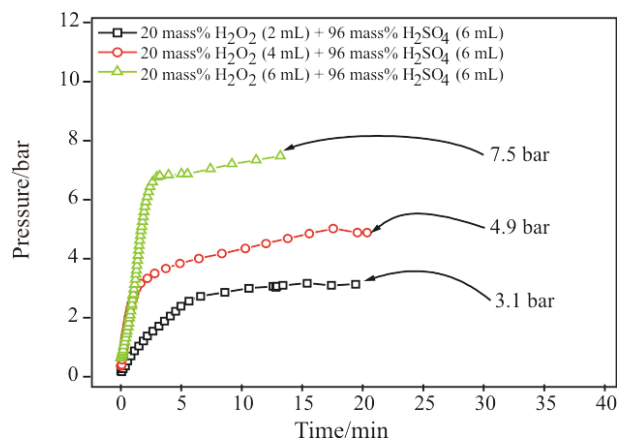


**Fig. 4** Pressure rise rate vs. temperature for thermal decomposition of  $\text{H}_2\text{O}_2$  and  $\text{H}_2\text{O}_2$  reacting with  $\text{H}_2\text{SO}_4$  (0.1, 0.5 and 1.0 N)

included the  $\text{H}_2\text{O}_2$  and the  $\text{H}_2\text{SO}_4$  (higher concentration), had a great quantity of  $\text{H}_2\text{SO}_4$ . The  $\text{H}_2\text{SO}_5$  shifted to  $\text{HSO}_5^-$  and  $\text{OH}^-$ . The  $\text{HSO}_4^-$  decomposed to form  $\text{SO}_4^{2-}$  and  $\text{OH}^-$  quickly. Figure 7c depicts the  $\text{H}_2\text{SO}_5$  integrated with  $\text{H}_3\text{O}_2^+$  shifted to  $\text{H}_3\text{SO}_5^-$  in another reaction. The  $\text{H}_3\text{SO}_5^-$ , combined the  $\text{HSO}_4^-$  shifted to  $\text{H}_2\text{S}_2\text{O}_8$  and  $\text{H}_2\text{O}$ . The  $\text{H}_2\text{S}_2\text{O}_8$  shifted to  $\text{S}_2\text{O}_8^{2-}$  and  $\text{H}_2\text{O}$  in a moment. The  $\text{S}_2\text{O}_8^{2-}$  decomposed to  $2\text{SO}_4^{2-}$  quickly.  $\text{H}_2\text{O}_2$  mixing with  $\text{H}_2\text{SO}_4$  could



**Fig. 5** Temperature vs. time for thermal reaction of  $\text{H}_2\text{O}_2$  reacting with  $\text{H}_2\text{SO}_4$  (96 mass%) by VSP2



**Fig. 6** Pressure vs. time of  $\text{H}_2\text{O}_2$  reacting with  $\text{H}_2\text{SO}_4$  (96 mass%) by VSP2

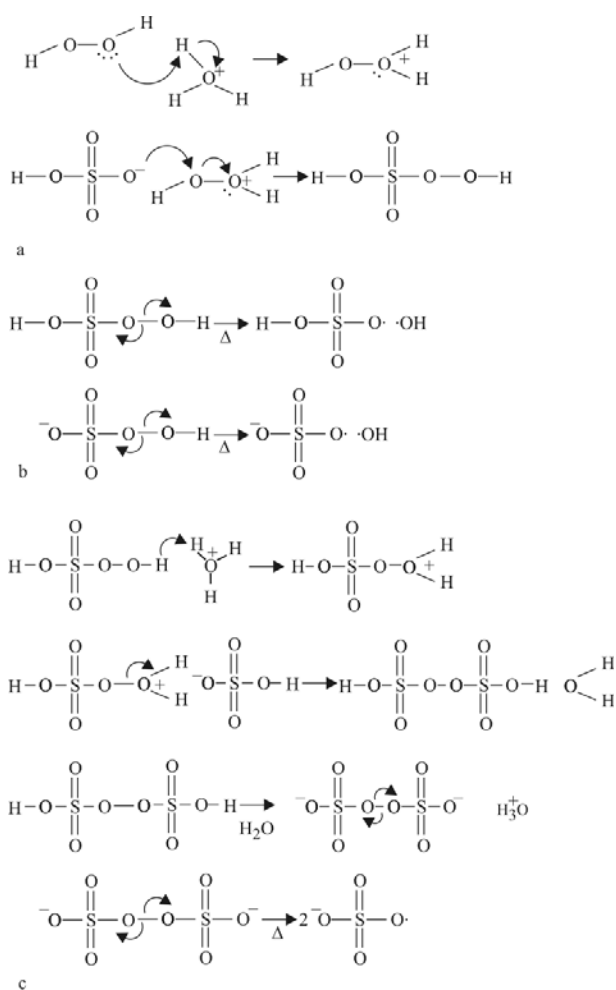
**Table 3** VSP2 experimental data of incompatibilities on  $\text{H}_2\text{O}_2$  with  $\text{H}_2\text{SO}_4$  (0.1, 0.5 and 1.0 N)

Sample	$T_0/^\circ\text{C}$	$T_{\max}/^\circ\text{C}$	$P_{\max}/\text{bar}$	$(dT/dt)_{\max}/^\circ\text{C min}^{-1}$	$(dP/dt)_{\max}/\text{bar min}^{-1}$
$\text{H}_2\text{O}_2$ (20 mass%, 30 mL)	51.1	132.6	16.0	1.0	0.32
$\text{H}_2\text{O}_2$ (20 mass%, 30 mL)+ 0.1 N $\text{H}_2\text{SO}_4$ (10 mL)	66.0	100.8	13.5	0.3	0.12
$\text{H}_2\text{O}_2$ (20 mass%, 30 mL)+ 0.5 N $\text{H}_2\text{SO}_4$ (10 mL)	68.4	98.3	10.3	0.1	0.03
$\text{H}_2\text{O}_2$ (20 mass%, 30 mL)+ 1.0 N $\text{H}_2\text{SO}_4$ (10 mL)	68.1	94.3	5.3	0.3	0.06

**Table 4** VSP2 experimental data of spherical bomb on H<sub>2</sub>O<sub>2</sub> and H<sub>2</sub>SO<sub>4</sub> (96 mass%)

Sample	$T_0/^\circ\text{C}$	$T_{\text{max}}/^\circ\text{C}$	$P_{\text{max}}/\text{bar}$	$(dT/dt)_{\text{max}}/^\circ\text{C min}^{-1}$	$(dP/dt)_{\text{max}}/\text{bar min}^{-1}$
H <sub>2</sub> O <sub>2</sub> (20 mass%, 2 mL)+ H <sub>2</sub> SO <sub>4</sub> (96 mass%, 6 mL)	N/A	88.0	3.1	34.7	0.32
H <sub>2</sub> O <sub>2</sub> (20 mass%, 4 mL)+ H <sub>2</sub> SO <sub>4</sub> (96 mass%, 6 mL)	N/A	101.9	4.9	60.4	0.12
H <sub>2</sub> O <sub>2</sub> (20 mass%, 6 mL)+ H <sub>2</sub> SO <sub>4</sub> (96 mass%, 6 mL)	N/A	120.8	7.5	70.9	0.03

N/A: not applicable


**Fig. 7** Proposed reaction mechanisms for H<sub>2</sub>O<sub>2</sub> mixed with higher concentrations of H<sub>2</sub>SO<sub>4</sub> (96 mass%): a – H<sub>2</sub>O<sub>2</sub> combined with H<sub>2</sub>SO<sub>4</sub> shifts to H<sub>2</sub>SO<sub>5</sub>; b – H<sub>2</sub>SO<sub>5</sub> shifts to SO<sub>4</sub><sup>2-</sup>; c – H<sub>2</sub>SO<sub>5</sub> mutates to H<sub>2</sub>S<sub>2</sub>O<sub>8</sub> and shifts to SO<sub>4</sub><sup>2-</sup> [10]

result in a violent heat decomposition and pressure rise. Attention was focused on the H<sub>2</sub>O<sub>2</sub> forming an unstable compound when it reacted with the high concentration of H<sub>2</sub>SO<sub>4</sub> (96 mass%), which had a great quantity of H<sub>2</sub>SO<sub>4</sub> and with increasing the mass

of H<sub>2</sub>O<sub>2</sub> shifted quickly to H<sub>2</sub>O<sub>2</sub> decomposition reaction. We proposed that the reaction rate of H<sub>2</sub>O<sub>2</sub> mixed with H<sub>2</sub>SO<sub>4</sub> (96 mass%) was increasing with the mass of H<sub>2</sub>O<sub>2</sub>. According to the data from Figs 4, 5, 7 and Table 4, the reaction rate increased quickly with increasing mass of H<sub>2</sub>O<sub>2</sub>.

## Conclusions

The reactivity of H<sub>2</sub>O<sub>2</sub> mixed with H<sub>2</sub>SO<sub>4</sub> (0.1, 0.5 and 1.0 N) undergoing thermal decomposition was examined. When an H<sub>2</sub>O<sub>2</sub> solution contained lower concentration of H<sub>2</sub>SO<sub>4</sub>, the reaction rate was observed to shift to slower than pure H<sub>2</sub>O<sub>2</sub> (20 mass%). The H<sub>2</sub>O<sub>2</sub> reacting with a lower concentration of H<sub>2</sub>SO<sub>4</sub> had a great amount of H<sub>2</sub>O and H<sub>2</sub>SO<sub>4</sub>, resulting in shifting to the formation of H<sub>2</sub>O<sub>2</sub> reaction. With increasing concentration of acid, this shift translates into an increase in reaction rate that was much slower than pure H<sub>2</sub>O<sub>2</sub>, depending on the concentration of H<sub>2</sub>SO<sub>4</sub>. When the H<sub>2</sub>O<sub>2</sub> mixture contained H<sub>2</sub>SO<sub>4</sub> (higher concentration), the reaction was observed to be violent. The H<sub>2</sub>O<sub>2</sub> reacted with higher concentration of H<sub>2</sub>SO<sub>4</sub> (96 mass%), which has a great quantity of H<sub>2</sub>SO<sub>4</sub>, and led to the reaction rate of the H<sub>2</sub>O<sub>2</sub> decomposition reaction shifting quickly. Under high concentration of H<sub>2</sub>SO<sub>4</sub> (96 mass%), the reaction rate can rise faster, followed by increasing the amount of H<sub>2</sub>O<sub>2</sub>.

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