J Therm Anal Calorim (2010) 102:563–568
DOI 10.1007/s10973-010-0959-6

Thermal hazard analyses and incompatible reaction evaluation of hydrogen peroxide by DSC

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NATAS2009 Special Issue

Abstract Hydrogen peroxide (H_2O_2), historically, due to its broad applications in the chemical industries, has caused many serious fires and explosions worldwide. Its thermal hazards may also be incurred by an incompatible reaction with other chemical materials, and a runaway reaction may be induced in the last stage. This study applied thermal analytical methods to explore the H_2O_2 leading to thermal accidents by incompatibility and to discuss what might be formed by the upset situations. In this study, the thermal hazard analyses were conducted with various solvents, propanone (CH_3COCH_3), Fe_2O_3, $FeSO_4$, H_2SO_4, HCl, HNO_3, H_3PO_4, NaOH, LiOH, and KOH which were deliberately selected to individually mix with H_2O_2 for investigating the degree of hazard. Differential scanning calorimetry (DSC) was employed to evaluate the thermal hazard of H_2O_2-mixed ten chemicals. The results indicated that H_2O_2 is highly hazardous while separately mixed with ten materials, as a potential contaminant. Fire and explosion hazards could be successfully reduced if the safety-related data are suitably imbedded into manufacturing processes.

Keywords Differential scanning calorimetry (DSC) · Hydrogen peroxide (H_2O_2) · Incompatible reaction · Thermal hazards · Potential contaminant

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Introduction

Hydrogen peroxide (H_2O_2) was applied and isolated firstly in 1818 by Louis Jacques Thenard by reacting barium peroxide with nitric acid (HNO_3). An improved version of this process used hydrochloric acid (HCl), followed by sulfuric acid (H_2SO_4) to precipitate the barium sulfate ($BaSO_4$), a typical byproduct. Thenard's process was applied from the end of the nineteenth century until the middle of the twentieth century. Since then, for a long time it was believed that pure H_2O_2 was unstable, because attempts to separate the H_2O_2 from the water, which is present during synthesis, failed.

This was because traces of solids and heavy metal ions led to a catalytic decomposition or explosions of the H_2O_2. In 1894, 100 mass% H_2O_2 was first obtained through vacuum distillation by Richard Wolffenstein in 1894. Before the end of nineteenth century, Petre Melikishvili and his pupil L. Pizarjevski presented that the many proposed formulas of H_2O_2, the correct one was H–O–O–H. Oxygen (O_2) and hydrogen (H_2) are applied to produce H_2O_2 and is releasing H_2O and O_2 in decomposition reaction [1]. H_2O_2 decomposition mechanism [2, 3] is displayed as follows:

$$H_2 + O_2 \rightarrow H_2O_2 \tag{1}$$

$$2H_2O_2 \rightarrow 2H_2O + O_2. \tag{2}$$

H_2O_2 is employing to oxidize in organic and inorganic manufacturing process, polymerization reaction [4], wet bench reaction in semiconductor process [5], industrial wastes application, and so on [6–8].

Common hazardous characteristics of organic peroxides (OPs) and inorganic peroxides (IPs) are mainly attributed to its peroxide molecule (–O–O–) existing [9–13]. H_2O_2 is one of IPs group that is considered as hazardous material in Globally Harmonized System of Classification and Labelling of Chemicals (GHS). H_2O_2, dimethyl phthalate (DMP), and methyl ethyl ketone (MEK) are applied to a manufacture the methyl ethyl ketone peroxide (MEKPO) product in Taiwan, Korea, and China. Such a manufacturing process had caused many thermal explosion and runaway reaction accidents [14]. The difference between heat generation rate and heat removal rate exceeds zero value that is leading to heat accumulation, eventually triggering a runaway reaction in the reactor [15, 16].

H_2O_2, propanone (so-called acetone), and H_2SO_4 are employing to generate diacetone diperoxide (DADP) or triacetone triperoxide (TATP) [17]. DADP or TATP is used as an initiator for explosive materials for terrorist attacks worldwide [18–23]. Although H_2O_2 is widely applied in industry; however, it had caused many serious explosions and runaway reactions in Asia [24].

Many investigations [25, 26] are applying to describe incompatible reactions of H_2O_2 mixed with various contaminations, such as iron, copper, sulfuric acid (H_2SO_4), propanone, heavy mental ion, sodium hydroxide (NaOH), 2-propanol (2-PropOH), 2-methyl-2-propanol (TBA), 2-methyl-2-butanol (TAA), 2-methyl-2-pentanol (THA) [27], etc. Therefore, this study was applied to assess thermal hazard of NaOH, potassium hydroxide (KOH), and lithium hydroxide (LiOH) that were usually employed in chemical industries.

The aim of this study was to analyze the thermal decomposition behaviors of H_2O_2 individually mixing with propanone, ferric oxide (Fe_2O_3), ferrous sulfate ($FeSO_4$), H_2SO_4, HCl, HNO_3, phosphoric acid (H_3PO_4), NaOH, KOH, and LiOH by differential scanning calorimetry (DSC). Most of the reports regarding thermal hazard analysis are using calorimetric technique that includes glass vessel, DSC, vent sizing package 2 (VSP2), reaction calorimeter (RC), reactive system screening tool (RSST), thermal activity monitor (TAM), accelerating rate calorimeter (ARC), and so on [28–30]. Exothermic onset temperature (T_0) of H_2O_2 comparing with various contaminations were determined at 50 °C by DSC.

Experimental

Samples

Thirty-one and 45 mass% H_2O_2 were purchased directly from the Aldrich Co. and both density and concentration were measured. Various chemicals includes propanone (CH_3COCH_3), 6 N NaOH, 6 N KOH, 6 N LiOH, 6 N HCl, 6 N H_2SO_4, 6 N HNO_3, 6 N H_3PO_4, Fe_2O_3, $FeSO_4$ that could be assumed to be encountered in process or storage conditions, was deliberately chosen to be in the DSC experiments. In this study, the ratio of 31 mass% H_2O_2 compared with incompatible materials were calculated to be 3. As planned, H_2O_2 samples with 10, 20, 31, and 45 mass% were selected, as shown in Fig. 1.

Differential scanning calorimetry (DSC)

Differential scanning calorimetry is a technical tool in which the differential in energy inputs into the sample and reference sample required to keep their temperature equal is measured as a function of temperature while the sample and reference material are subjected to a controlled increase in temperature. Temperature-programed screening experiments were performed on a Mettler TA8000 system DSC821^e: the test cell (Mettler ME-26732) could withstand high pressure of about 100 bar. Heating rate chosen for the temperature-programed ramp was 4 °C min^{-1}, to maintain better thermal equilibrium. The tests were conducted between 30 and 300 °C with a series of incompatible materials. The test used a pipet to inject at about 3–10 mg, with 10, 20, 31, and 45 mass% H_2O_2 dripped for few different incompatible samples, respectively, and then was injected into the test cell. The test cell was sealed manually, and dynamic scanning by DSC tests was conducted.

Results and discussion

H_2O_2 decomposition investigation

Practically, H_2O_2 is used as the oxide provider in chemical industries. H_2O_2 decomposed at 40 °C and released O_2 and H_2O. In parallel, it produced tremendous amount of heat

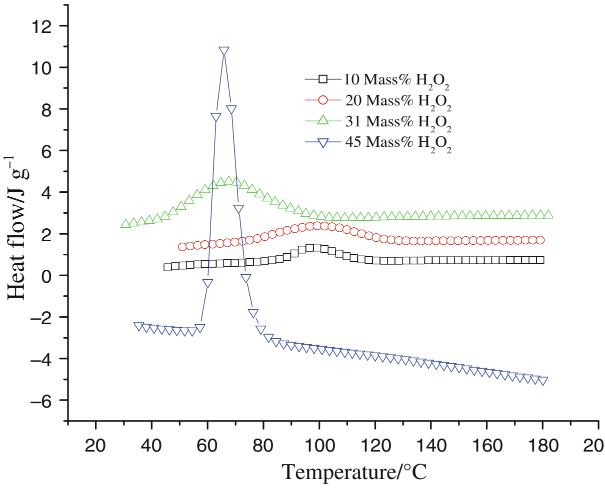

Fig. 1 Thermal analysis curves of various H_2O_2 concentrations by DSC at 4 °C min^{-1} of heating rate

and pressure in the reactor. Various H_2O_2 concentrations, such as 10, 20, 31, 45 mass%, were applied to determine the thermal hazard in storage, handling, and other applications. According to Fig. 1, T_0 of 31 and 45 mass% H_2O_2 were evaluated to be 40 °C because it exist more percentage of H_2O_2. Therefore, T_0 of 10 and 20 mass% H_2O_2 were evaluated to be 60 °C because it exist more percentage of H_2O. Maximum temperature (T_{max}) of H_2O was determined to be 100 °C (boiling point).

According to curve data, higher concentration was more dangerous than lower one. All of experimental data including T_0, activation energy (E_a), frequency factor (A), heat of decomposition (ΔH_d), maximum temperature (T_{max}) were listed in Table 1.

Thermal hazard analyses of H_2O_2 mixed with various acids

Various acids, such as HCl, HNO_3, H_2SO_4, H_3PO_4 were broadly used as catalyst in manufacturing process. To prevent handling hazard, this study was employed to analyze H_2O_2 mixing with those acid by DSC. H_2O_2 mixed with H^+ decomposed at 50 °C. Thirty-one mass% H_2O_2 comparing with various acids were displayed in Figs. 2, 3, 4 and 5. When H_2O_2 mixed with H_2SO_4, exothermic reaction occurred at 70 °C and released energy about 473 J g^{-1}. At the same situation, ΔH_d of H_2O_2 added H_3PO_4 and HNO_3 were fewer than 31 mass% H_2O_2. Figure 3 shows thermal hazard analysis of H_2O_2 mixed with H_3PO_4. H_2O_2 compared with HCl was evaluated in Fig. 4. Endothermic reaction phenomenon at high temperature circumstance was discovered in H_2O_2 joining H_2SO_4, H_3PO_4, and HNO_3 in this study.

H_2O_2 mixed with HCl was applied to scrutinize the differential result from other acids in Fig. 4. There are two decomposition peaks of H_2O_2 mixed with HCl in Fig. 4. H_2O_2 decomposed in the first peak and released energy about 512 J g^{-1}. The ΔH_d of second peak was determined as 1,535 J g^{-1}. Hypochlorous acid (HClO) and H_2O were produced in H_2O_2 mixed with HCl reaction. This reaction mechanism (Eqs. 3–5) was shown as follows:

$$H_2O_2 \rightarrow H^+ + HO_2^- \quad (3)$$

$$HCl \rightarrow H^+ + Cl^- \quad (4)$$

$$H_2O_2 + HCl \rightarrow HClO + H_2O. \quad (5)$$

Thermal decomposition of H_2O_2 mixed with various alkalis

To prevent runaway reaction of acid catalyst in manufacturing process, alkali, such as NaOH, KOH, and LiOH, was widely used as a neutralizer with H^+ at emergency situation that generated H_2O. Incompatible reaction analysis was employed to prevent fire, explosion, or toxic release from occurring. This study used three bases mixed with H_2O_2. Strong alkali released hydroxyl (OH^-) and metal positive ion (M^+). Runaway phenomena incurred as H_2O_2 mixed with three alkalis were shown in Fig. 6. Alkali mixed with H_2O_2 decomposed suddenly in initial reaction and produced an endothermic reaction at high temperature.

The reaction mechanism of NaOH/H_2O_2 was described in Eqs. 6–8.

$$H_2O_2 \rightarrow H^+ + HO_2^- \quad (6)$$

$$NaOH \rightarrow Na^+ + OH^- \quad (7)$$

Table 1 Thermokinetics of 31 mass% H_2O_2 mixing with various chemicals by DSC under 4 °C min^{-1} of heating rate

Material	Mass/mg	T_0/°C	T_{max}/°C	$-\Delta H_d$/J g^{-1}	ΔH_{endo}/J g^{-1}	E_a/kJ mol^{-1}	A/s^{-1}
H_2O_2	2.1	40	75	963	NA	336	115
$H_2O_2 + Fe_2O_3$ (3:1)	3.4	R	80	991	942	81	22
$H_2O_2 + FeSO_4$ (3:1)	4.8	R	NA	NA	82	NA	NA
H_2O_2 + 6 N NaOH (3:1)	11.1	R	50	420	1,532	325	118
H_2O_2 + 6 N KOH (3:1)	6.2	R	NA	NA	580	NA	NA
H_2O_2 + 6 N LiOH (3:1)	7.7	R	NA	NA	119	NA	NA
H_2O_2 + 6 N H_2SO_4 (3:1)	9.1	60	125	473	935	100	25
H_2O_2 + 6 N HCl (3:1)	5.7	40	110	512	1,534[a]	88	23
H_2O_2 + 6 N HNO_3 (3:1)	8.3	60	125	565	1,291	74	17
H_2O_2 + 6 N H_3PO_4 (3:1)	11.5	60	125	404	1,211	70	16
H_2O_2 + propanone (3:1)	5.0	R	75	523	994[a]	122	36
TATP	5.0	50	110	990	782	95	25

[a] Exothermic reaction

R room temperature, *NA* not applicable

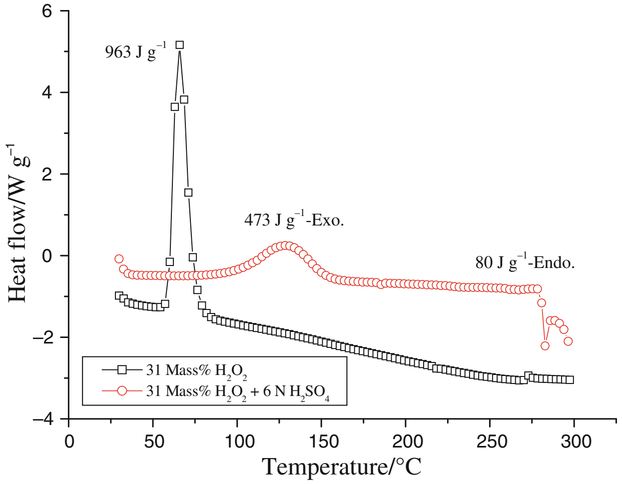

Fig. 2 Reactive hazard evaluation of H_2O_2 compared with H_2SO_4 by DSC at 4 °C min^{-1} of heating rate

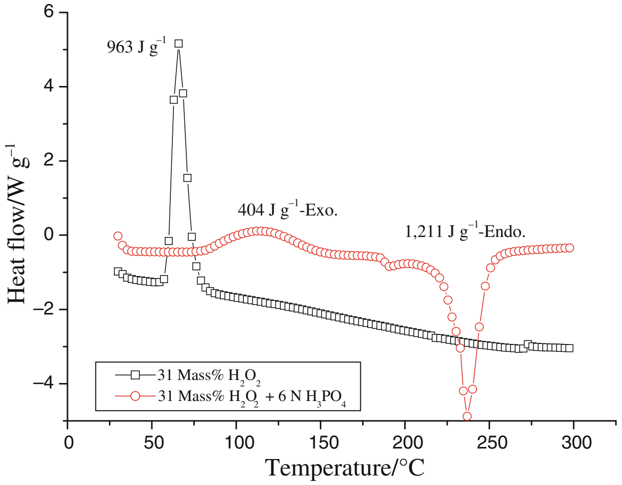

Fig. 3 Thermal decomposition analysis of H_2O_2 compared with H_3PO_4 by DSC at 4 °C min^{-1} of heating rate

$$H_2O_2 + NaOH \rightarrow NaHO_2 + H_2O. \quad (8)$$

The reaction mechanism of KOH/H_2O_2 was illustrated in Eqs. 9–11.

$$H_2O_2 \rightarrow H^+ + HO_2^- \quad (9)$$

$$KOH \rightarrow K^+ + OH^- \quad (10)$$

$$H_2O_2 + KOH \rightarrow KHO_2 + H_2O. \quad (11)$$

The reaction mechanism of LiOH/H_2O_2 was expressed in Eqs. 12–14.

$$H_2O_2 \rightarrow H^+ + HO_2^- \quad (12)$$

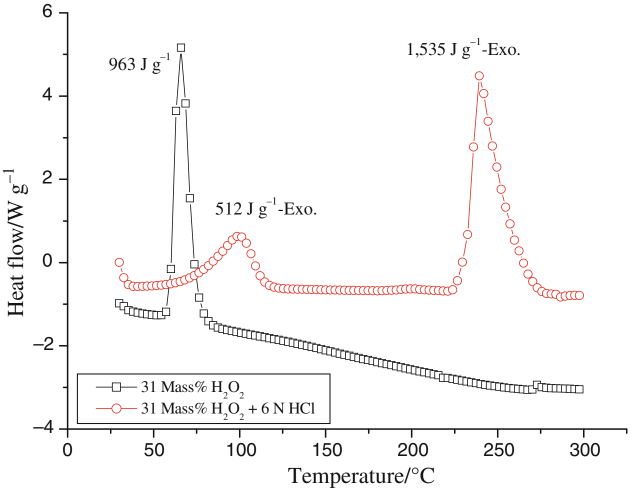

Fig. 4 Reactive hazard investigation of H_2O_2 compared with HCl by DSC at 4 °C min^{-1} of heating rate

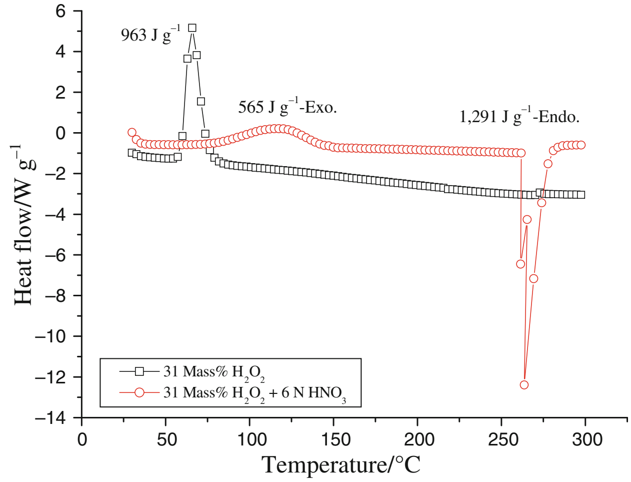

Fig. 5 Reactive hazard investigation of H_2O_2 compared with HNO_3 by DSC at 4 °C min^{-1} of heating rate

$$LiOH \rightarrow Li^+ + OH^- \quad (13)$$

$$H_2O_2 + LiOH \rightarrow LiHO_2 + H_2O. \quad (14)$$

Thermal reaction evaluation of H_2O_2 mixed with Fe^{2+} and Fe^{3+}

The chemistry of the Fe^{2+}/H_2O_2 system is well documented in the literature [8, 31]. H_2O_2 catalytic decomposition occurs through the formation of highly reactive species; of these free radicals such as hydroxyl ($HO^{\bullet}$) and hydroperoxyl ($HO_2^{\bullet}$) radicals are generally believed to be the dominant ones through which H_2O_2 decomposition proceeds as follows:

The reaction mechanism of H_2O_2 with Fe^{2+} was denoted in Eqs. 15–19 [8, 31].

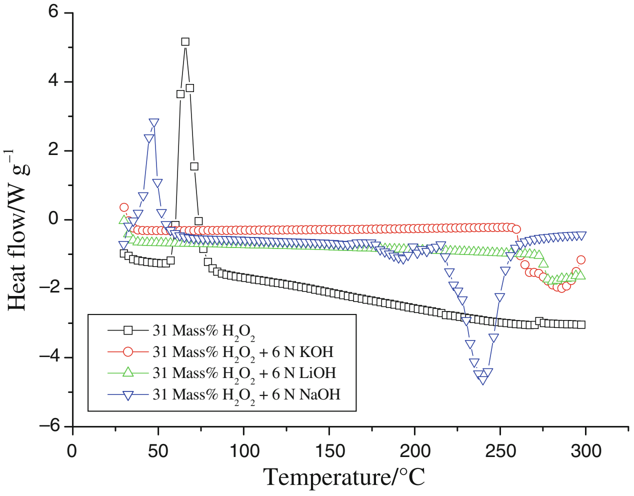

Fig. 6 Reactive hazard investigation of H_2O_2 mixed with various bases by DSC at 4 °C min^{-1} of heating rate

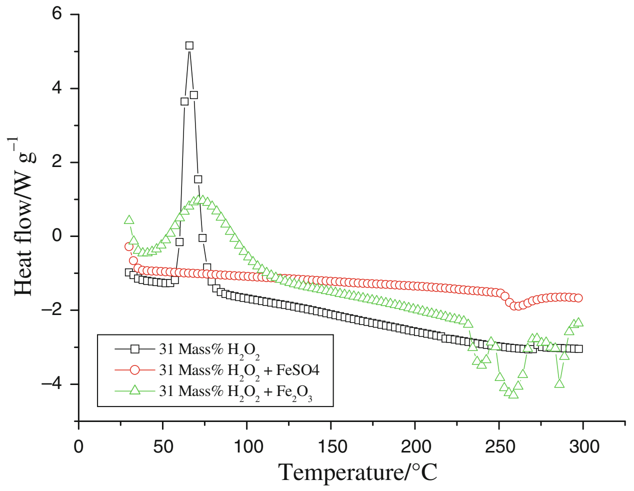

Fig. 7 Incompatible reaction evaluation of H_2O_2 joined Fe^{2+} and Fe^{3+} by DSC at 4 °C min^{-1} of heating rate

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^{-} + HO\cdot \quad (15)$$

$$H_2O_2 + HO^{\bullet} \rightarrow H_2O + HO_2 \quad (16)$$

$$H_2O_2 + HO_2 \rightarrow O_2 + H_2O + HO^{\bullet} \quad (17)$$

$$Fe^{2+} + HO_2 \rightarrow Fe^{3+} + HO_2^{-} \quad (18)$$

$$Fe^{2+} + OH^{\bullet} \rightarrow Fe^{3+} + OH^{-}. \quad (19)$$

The reaction mechanism of H_2O_2 with Fe^{3+} was noted in Eqs. 20–24.

$$H_2O_2 \rightarrow H^{+} + HO_2^{-} \quad (20)$$

$$Fe^{3+} + HO_2^{-} \rightarrow Fe^{2+} + HO_2 \quad (21)$$

$$H_2O_2 + HO_2 \rightarrow O_2 + H_2O + HO^{\bullet} \quad (22)$$

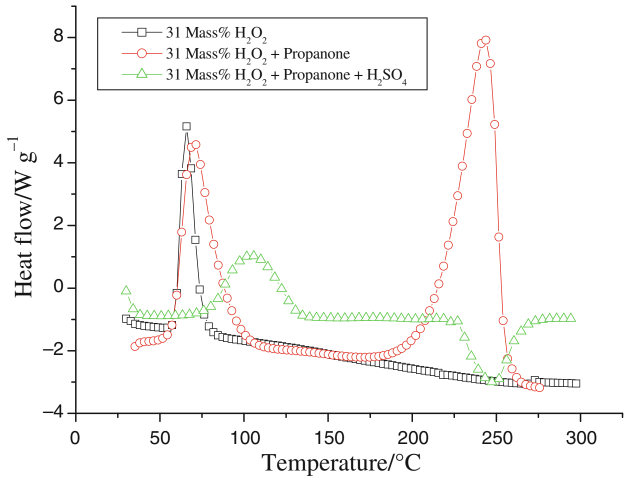

Fig. 8 Thermal hazard characteristic analysis of H_2O_2 mixed with propanone by DSC at 4 °C min^{-1} of heating rate

$$Fe^{2+} + OH^{\bullet} \rightarrow Fe^{3+} + OH^{-} \quad (23)$$

$$OH^{-} + H^{+} \rightarrow H_2O. \quad (24)$$

Storage and handling of H_2O_2 must concerned containing iron (Fe^{2+} or Fe^{3+}) in the chemical industries or manufacturing process. Thermal decomposition reaction of H_2O_2 compared with Fe_2O_3 and $FeSO_4$ are shown in Fig. 7.

Hydrogen peroxide mixed with propanone

Normally, propanone is used as cleaner in university laboratory, chemical industries, and so on. In practice, H_2O_2 and propanone are employed to synthesize TATP or DADP catalyzed by H_2SO_4 is in terrorist attacks. According to the description in Fig. 8, H_2O_2 decomposed at 40 °C (lower temperature) and released 963 J g^{-1} of energy. At the same time, H_2O_2 mixing with propanone was determined the T_0 at 40 °C. The first peak of H_2O_2 mixing with propanone was H_2O_2 decomposition result. T_0 of TATP was evaluated at 50 °C.

Conclusions

H_2O_2 (31 mass%) decomposed at lower temperature (<50 °C) and released numerous pressures in the reactor. E_a of H_2O_2 was calculated about 336 kJ mol^{-1}. However, E_a of H_2O_2 mixed with alkali, acid, propanone, iron, etc., were lower than pure H_2O_2 alone. Lower E_a led to reaction rate change fast even uncontrolled condition. In view of loss prevention, H_2O_2 mixed with HCl must be concerned because of HClO decomposition (1,532 J g^{-1}) occurred at high temperature. H_2O_2 mixing with propanone was determined the T_0 at 40 °C. The first peak of H_2O_2 mixing with propanone was H_2O_2 decomposition result. T_0 of TATP was evaluated at 50 °C.

To protect thermal runaway reaction of acid catalyst in manufacturing process, alkali including NaOH, KOH, and LiOH was widely used as a neutralizer with H^+ at emergency situation that generated H_2O. Incompatible reaction analysis was employed to prevent fire, explosion, or toxic release from occurring. This study used three bases mixed with H_2O_2. Strong alkali released hydroxyl (OH^-) and metal positive ion (M^+).

According to explosion and fire accident occurring theory, product of high temperature decomposition is unintended results. When H_2O_2 mixed with propanone, two exothermic decomposition reaction producing. Hazard of first peak for H_2O_2/propanone mixtures is well known in fundamental process, but second one usually causes severe damage and explosion.

Acknowledgements The authors would like to thank Dr. Kuo-Ming Luo for valuable suggestions on experiments and the measurements of a runaway reaction.

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