

# Calorimetric studies on the thermal hazard of methyl ethyl ketone peroxide with incompatible substances

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## Abstract

In Taiwan, Japan, and China, methyl ethyl ketone peroxide (MEKPO) has caused many severe thermal explosions owing to its thermal instability and reactivity originating from the complexity of its structure. This study focused on the incompatible features of MEKPO as detected by calorimetry. The thermal decomposition and runaway behaviors of MEKPO with about 10 wt.% incompatibilities, such as H<sub>2</sub>SO<sub>4</sub>, HCl, NaOH, KOH, FeCl<sub>3</sub>, and FeSO<sub>4</sub>, were analyzed by dynamic calorimeter, differential scanning calorimetry (DSC) and adiabatic calorimeter, vent sizing package 2 (VSP2). Thermokinetic data, such as onset temperature, heat of decomposition, adiabatic temperature rise, and self-heat rate, were obtained and assessed. Experimental data were used for determining the incompatibility rating on hazards.

From the thermal curves of MEKPO with assumed incompatible substances detected by DSC, all the onset temperatures in the other tests occurring earlier advanced, especially with alkaline or ferric materials. In some tests, significant incompatible reactions were found. Adiabatic runaway behaviors for simulating the worst case scenario were performed by using VSP2. These calorimetric data led to the same results that the alkaline or ferric solution was the most incompatible with MEKPO.

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**Keywords:** Methyl ethyl ketone peroxide (MEKPO); Thermal instability; Thermal decomposition; Incompatible reactions; Adiabatic runaway behaviors

## 1. Introduction

In the last century, many thermal explosions in Taiwan, Japan, and China were caused by methyl ethyl ketone peroxide (MEKPO) being subjected to external fires and other igniting sources. Yeh et al. [1] used differential scanning calorimetry (DSC) and vent sizing package 2 (VSP2) with the techniques of thermal analysis to assess the thermal hazards and thermokinetic data. The exothermic onset temperature of MEKPO is determined to be about 110 °C. Tseng et al. [2] indicated that MEKPO is highly hazardous when mixed with H<sub>2</sub>SO<sub>4</sub>, NaOH, Fe<sub>2</sub>O<sub>3</sub>, in corroborating the thermal decomposition and runaway behaviors of MEKPO with specific incompatibilities. We used about 10 wt.% contaminant, such as H<sub>2</sub>SO<sub>4</sub>, HCl, NaOH, KOH,

FeCl<sub>3</sub>, and FeSO<sub>4</sub>, to investigate the incompatible hazards of MEKPO with mixed materials.

Table 1 gathers statistical accidents caused by MEKPO in Taiwan, Japan, and China. In Taiwan, five selected accidents killed 55 and injured 156 people; one of the most catastrophic accidents was the Yung-Hsin plant explosion, which killed 10 people and injured 47 during the fire fighting [3]. The fire was originally caused by a runaway oxidation reaction in a reactor unit that then spread to the tank yard, thus resulting in a disaster. In Japan, from 1953 to 1978, MEKPO was involved in 14 accidents. In particular, in 1964 in Tokyo, about 3600 kg of MEKPO exploded, killing 19 and wounding 114 people [4]. In recent years in China, there have also been several severe accidents resulting from MEKPO.

MEKPO, which is widely used in polymerization in the plastic industry, is produced by the reaction of methyl ethyl ketone (MEK) and hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) with various catalysts [5]. In general, MEKPO has seven different structures; for

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**Nomenclature**

$\Delta H$	heat of reaction ( $\text{J g}^{-1}$ )
$\Delta H_d$	heat of decomposition ( $\text{J g}^{-1}$ )
$P_{\text{final}}$	final pressure (psig)
$P_{\text{max}}$	maximum pressure (psig)
$(dP dt^{-1})_{\text{max}}$	maximum pressure-rise rate ( $\text{psi min}^{-1}$ )
$t$	time (min)
$T$	temperature ( $^{\circ}\text{C}$ )
$dT dt^{-1}$	self-heat rate ( $^{\circ}\text{C min}^{-1}$ )
$(dT dt^{-1})_{\text{max}}$	maximum self-heat rate ( $^{\circ}\text{C min}^{-1}$ )
$T_0$	exothermic onset temperature ( $^{\circ}\text{C}$ )
$\Delta T_{\text{ad}}$	adiabatic temperature rises ( $^{\circ}\text{C}$ )
$T_{\text{max}}$	maximum temperature ( $^{\circ}\text{C}$ )

*Greek symbols*

$\Phi$	thermal inertial
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commercial purposes, the supplier often delivers the monomer and dimer structures. Due to the inherently reactive hazards of MEKPO, many organizations have established regulations during the preparation, manufacturing, storage, and transportation stages, such as OSHA 1910.119 [6], National Fire Protection Association (NFPA) 43B [7], Department of Transportation (DOT) [8], and HSE of Control of Industrial Major Accident Hazards Regulations [9], to name a few.

Regarding the accidents that have occurred in Taiwan, there are still several reasons, which cannot be confirmed. Generally, the accidents were considered as fire-induced thermal explosions. Yeh et al. [1] conducted calorimetry tests and found the onset temperature of MEKPO is about  $110^{\circ}\text{C}$ , which could be interpreted as the hidden reason for the thermal explosions from an initial fire accident. However, if incompatibilities exist, these mixtures may result in exothermic phenomena under lower temperature, and then entail ensuing thermal hazards, thermal runaways or even thermal explosions. This is the exact reason that incompatible substances should be taken into account with respect to the influence of the thermal runaway reaction of MEKPO. These contaminants are possibly being encountered in process, storage, and transportation units.

Numerous studies of induced hazards by organic peroxides have been performed worldwide. The United Nations has even suggested that an organic peroxide supplier must make a precise test of self-accelerating decomposition temperature (SADT) in any specific commercial package [10]. TNO in the Netherlands has devoted considerable efforts to the testing and classifica-

tion of organic peroxides. MEKPO has been recognized as a flammable type or class III or IV by the code of NFPA 43B. The members of Design Institute for Emergency Relief Systems (DIERS) have emphasized research on the characteristics of pressure relief for organic peroxides [11,12]. The exothermic threshold temperature of many organic peroxides is usually around  $50\text{--}120^{\circ}\text{C}$ . However, for some runaway incidents caused by MEKPO, the reaction or storage temperatures have even been as low as ambient temperature [1]. To date, the reactive and incompatible hazards of MEKPO have not been clearly identified, and more efforts are needed for the study of its hazardous properties.

The emergency relief area of a safety relief valve or rupture disk of a reactor or storage tank is nearly proportional to the adiabatic self-heat rate at the blowdown conditions using DIERS methodology [11,12]. Most thermal runaway reactions caused by organic peroxides will be accompanied by violent heat-releasing rates and thermal explosions. The maximum self-heat rate is larger than  $100^{\circ}\text{C min}^{-1}$  for many runaway reactions of organic peroxides. The self-heat rate or thermokinetics are affected by temperature, pH value, and metal of containers, ions, and other impurities. The aim of this research was to identify the incompatible characteristics of mixtures with MEKPO. Both DSC and VSP2 or other adiabatic calorimetries were used for thermal analyses in order to acquire thermal runaway data. Data, such as exothermic onset temperature ( $T_0$ ), adiabatic time to maximum heat rate ( $\text{TMR}_{\text{ad}}$ ), adiabatic temperature rise ( $\Delta T_{\text{ad}}$ ), and self-heat rate ( $dT dt^{-1}$ ), etc., were employed for evaluating the hazard rating of incompatibility [13,14]. In summary, this study addressed the following objectives: identification and assessment of the effect of incompatibilities on the liability or stability MEKPO and the applicability of calorimetry to incompatibilities of hazardous materials.

## 2. Experimental

### 2.1. Samples

MEKPO of 31 wt.% diluted in dimethyl phthalate (DMP) was purchased directly from the Aldrich Co., and then stored in a refrigerator at  $4^{\circ}\text{C}$ . Incompatible substances, such as  $\text{H}_2\text{SO}_4$ ,  $\text{HCl}$ ,  $\text{NaOH}$ ,  $\text{KOH}$ ,  $\text{FeCl}_3$ , and  $\text{FeSO}_4$ , which could be encountered in process or storage conditions, were deliberately selected to be prepared at approximately 5 and 10 wt.% relative to MEKPO in both DSC and VSP2 experiments, respectively.

### 2.2. DSC

Temperature-programmed screening experiments were performed on a Mettler TA8000 system DSC821 $^{\circ}$ ; the test cell (Mettler ME-26732) could withstand high pressure until about 100 bar. The scanning rate selected for the temperature-programmed ramp was  $4^{\circ}\text{C min}^{-1}$  to maintain better thermal equilibrium [15]. About 5 mg of MEKPO was used for testing samples, followed by adding different incompatible substances. The test cell was sealed manually, and the dynamic scanning test was conducted by using DSC.

Table 1  
Major accidents caused by MEKPO in Taiwan, Japan and China

Era	Country	Number of cases	Injuries	Fatalities
1979–2001	Taiwan	5	156	55
1953–1978	Japan	14	115	23
1980–2003	China	13	6	9

### 2.3. VSP2

VSP2, a PC-controlled adiabatic calorimeter system, was manufactured by Fauske & Associates Inc. It can acquire temperature and pressure traces versus time. In theory, an adiabatic calorimeter with the low heat capacity of the test cell ensures essentially all the reaction heat released remains within the tested sample. The thermal inertia ( $\Phi$ ) of the small test cells (116 ml) is about from 1.05 to 1.32, which can assess the thermokinetics and thermal hazards, then directly extrapolate to the process conditions [16]. Chang et al. [17,18] adopted VSP2 to conduct a decomposition reaction of 15 wt.% MEKPO, indicating a rapid pressure change, transcending the limit of the test cell and the subsequent bursting of the test cell. To adequately protect the normal operation of this apparatus and avoid bursting the test cell and missing the end of exothermic data, 10 wt.% of MEKPO was prudently chosen for the experiments of VSP2.

First, we injected 50 g 10 wt.% MEKPO into the test cell, and then used vacuum conditions to draw the incompatible substances within 2.5 g from the injector into the test cell. Then we adopted the standard heat-wait-search (HWS) procedure to execute the adiabatic runaway test. However, some incompatible reactions may occur instantaneously during the mixing with incompatible substances. To avoid heating the system and interfering with the onset condition, after injecting the incompatible samples, we just opened the guard heater to keep the test system at the desired adiabatic environment and waited for a few pre-set minutes. If there was any prominent temperature or pressure-rise, we then terminated the HWS step and switched off the main heater for closely tracking a runaway reaction [17].

## 3. Results

### 3.1. Thermal analysis by DSC for MEKPO with incompatible substances

By using programmed scanning, we readily acquired the onset temperature and heat of decomposition of MEKPO with incompatible substances from the thermal curves. MEKPO itself inherently revealed two conspicuous exothermic reaction regions, and the onset temperature of the first reaction region detected by the DSC, which was defined as the intersection point between the tangent of the heat flow versus temperature curve and the baseline, was ca. 96 °C. The peak areas of the 31 wt.% MEKPO sample gave  $951 \pm 13 \text{ J g}^{-1}$ , and this value was extrapolated to be ca.  $3100 \text{ J g}^{-1}$  for a 100 wt.% MEKPO sample.

Fig. 1 depicts the typical heat flow curves versus temperature and the thermal decomposition of 31 wt.% MEKPO with acidic substances. By mixing with alkaline solution hydroxide ion, the temperature and heat flow curves are shown in Fig. 2. In addition, Figs. 3 and 4 illustrate the incompatible effect on MEKPO of the solutions containing ferric, ferrous, and chloride ions, respectively.

Table 2 summarizes the effects of various contaminants on the hazards of incompatibility detected by DSC.

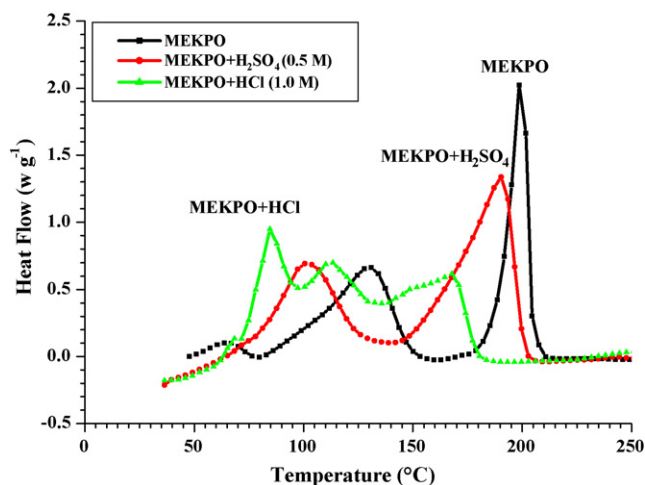


Fig. 1. Thermal curves of 31 wt.% MEKPO mixed with inorganic acid scanned by DSC tests.

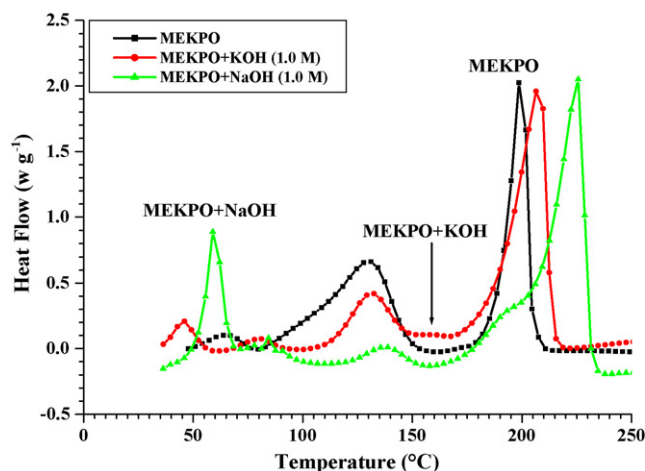


Fig. 2. Thermal curves of 31 wt.% MEKPO mixed with hydroxide ion scanned by DSC tests.

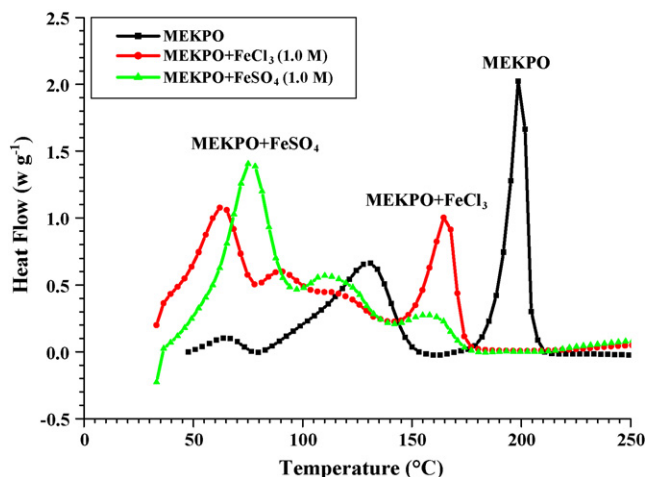


Fig. 3. Thermal curves of 31 wt.% MEKPO mixed with ferric and ferrous substances scanned by DSC tests.

Table 2  
DSC test results for MEKPO mixed with various incompatibilities

Sample, 31 wt.% MEKPO (mg)	Incompatibility		Heating rate ( $^{\circ}\text{C min}^{-1}$ )	Exothermic parameters <sup>a</sup>		
	Substance	Weight (mg)		$T_0$ ( $^{\circ}\text{C}$ )	$T_{\text{max}}$ ( $^{\circ}\text{C}$ )	$\Delta H$ ( $\text{J g}^{-1}$ )
5.5	–	–	4/	96.22	199.84	951.61
4.8	$\text{H}_2\text{SO}_4$ (0.5 M)	0.5	4	79.33	190.75	1186.99
4.2	HCl (1.0 M)	0.4	4	69.18	90.77	1119.45
4.5	NaOH (1.0 M)	0.5	4	53.40	255.49	989.12
4.5	KOH (1.0 M)	0.5	4	38.70	208.07	794.36
4.8	NaCl (1.0 M)	0.5	4	79.25	119.44	964.25
4.9	$\text{FeCl}_3$ (1.0 M)	0.5	4	48.37	165.94	792.18
4.8	$\text{FeSO}_4$ (1.0 M)	0.7	4	57.49	76.34	963.49

<sup>a</sup> The temperature data were estimated with the errors about  $\pm 1.5^{\circ}\text{C}$ , and the heat of thermal decomposition were estimated with the errors about  $\pm 13 \text{ J g}^{-1}$ .

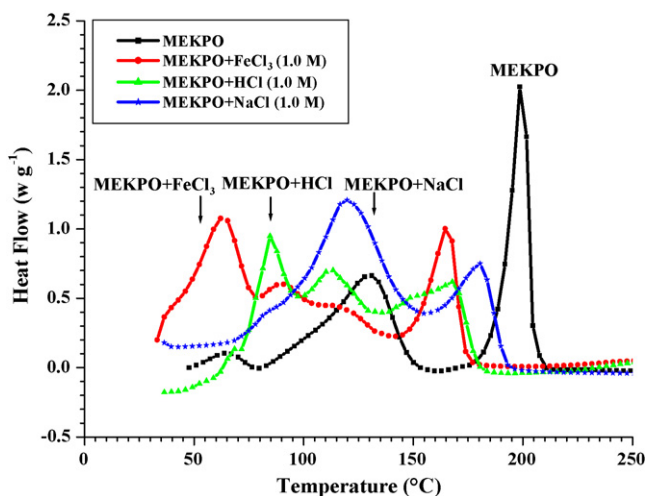


Fig. 4. Thermal curves of 31 wt.% MEKPO mixed with various materials containing chloride ion scanned by DSC tests.

### 3.2. Hazard analysis by VSP2 for MEKPO with incompatible substances

Characteristic temperature and pressure versus time curves for 10 wt.% MEKPO VSP2 tests are recorded in Fig. 5. Fig. 6 demonstrates the temperature versus self-heat rate and pressure-

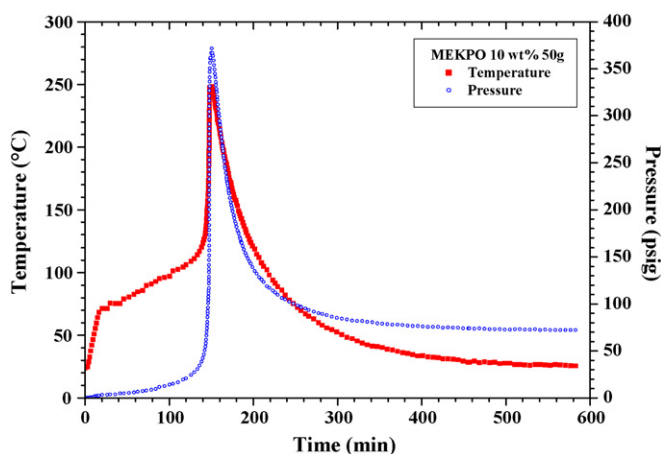


Fig. 5. Behaviors of thermal runaway for the decomposition reaction of 10 wt.% MEKPO scanned by VSP2 tests.

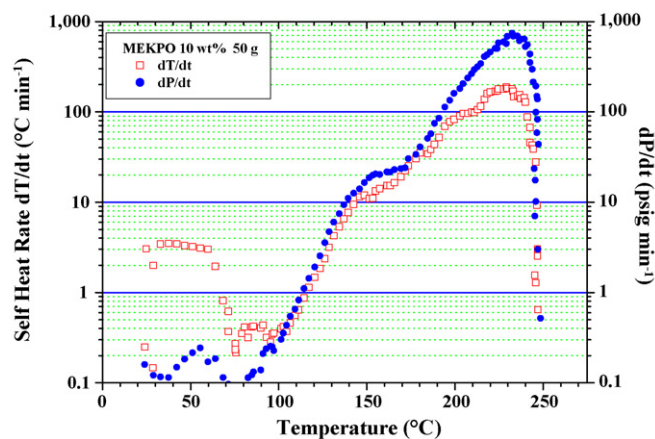


Fig. 6. Self-heat rate and pressure-rise rate vs. temperature for the decomposition reaction of 10 wt.% MEKPO scanned by VSP2 tests.

rise rate for the decomposition reaction of 10 wt.% MEKPO. These two figures were the background data for comparing the hazard rating of incompatible behaviors under adiabatic conditions.

Fig. 7 shows the temperature versus time curves for the decomposition reaction of 10 wt.% MEKPO with sulfuric acid and hydrochloric acid. The temperature versus time curves

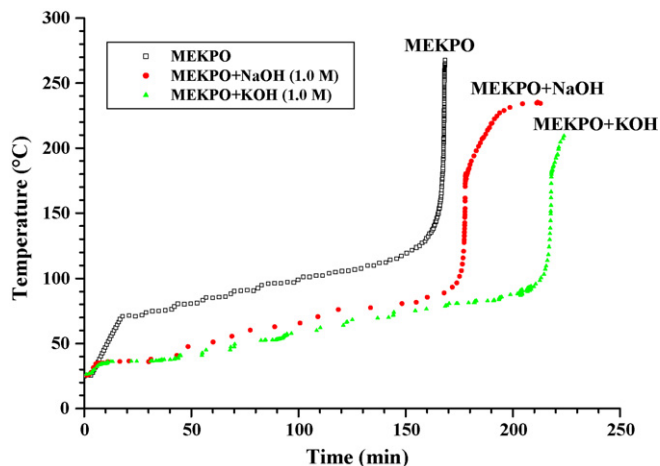


Fig. 7. Adiabatic runaway curves for the decomposition reaction of 10 wt.% MEKPO with inorganic acids scanned by VSP2 tests.



Table 3  
VSP2 test results for MEKPO mixed with various incompatibilities

Substance	$T_0$ (°C)	$T_{max}$ (°C)	$P_{max}$ (psig)	$(dT dr^{-1})_{max}$ (°C min <sup>-1</sup> )	$(dP dr^{-1})_{max}$ (psig min <sup>-1</sup> )	$P_{final}$ (psig)
MEKPO 10 wt.% (50 ml)	105.00	275.24	506.43	261.56	1481.55	94.22
MEKPO 10 wt.% (50 g)	101.37	267.73	371.63	188.65	745.78	88.91
MEKPO 10 wt.% (50 g) + H <sub>2</sub> SO <sub>4</sub> (0.5 M, 2.5 g)	71.84	216.65	343.79	221.82	707.57	45.38
MEKPO 10 wt.% (50 g) + HCl (1.0 M, 2.5 g)	43.26	201.75	275.41	42.44	50.88	17.54
MEKPO 10 wt.% (50 g) + NaOH (1.0 M, 2.5 g)	38.48/76.11	235.26	425.35	206.80	151.02	55.15
MEKPO 10 wt.% (50 g) + KOH (1.0 M, 2.5 g)	34.34/80.18	209.43	310.58	103.81	139.00	54.17
MEKPO 10 wt.% (50 g) + FeSO <sub>4</sub> (1.0 M, 2.5 g)	31.98	237.94	455.15	179.34	624.40	68.82
MEKPO 10 wt.% (50 g) + FeCl <sub>3</sub> (1.0 M, 2.5 g)	29.14	189.35	237.31	121.35	76.28	42.44
MEKPO 10 wt.% (50 g) + NaCl (1.0 M, 2.5 g)	61.86	200.86	299.83	121.39	184.45	53.68

The temperature data were estimated with the errors about  $\pm 1.5$  °C, and the pressure data were estimated with the errors about  $\pm 1.9$  psig. The thermal inertia ( $\Phi$ ) in these experiments was about 1.19.

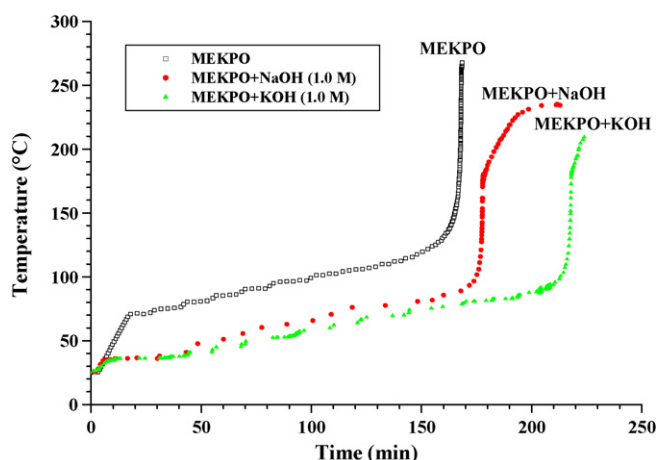


Fig. 8. Adiabatic runaway curves for the decomposition reaction of 10 wt.% MEKPO mixed with alkaline solutions or hydroxide ions scanned by VSP2 tests.

of 10 wt.% MEKPO mixed with alkaline solutions containing sodium hydroxide and potassium hydroxide are diagrammed in Fig. 8. In addition, Figs. 9 and 10 depict the incompatible reaction experiments on MEKPO with solutions containing ferrous sulfate and ferric chloride, respectively.

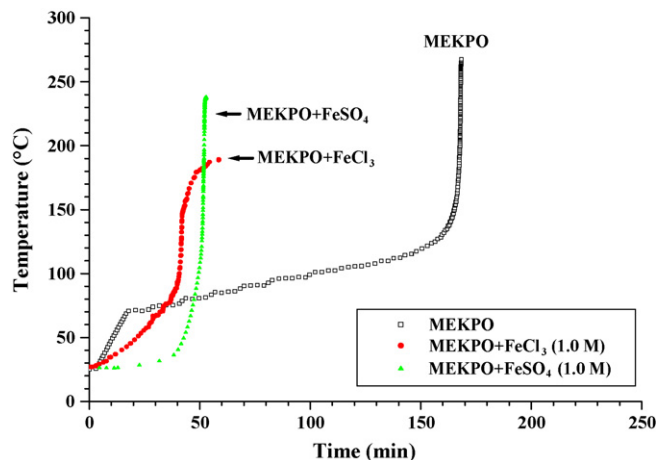


Fig. 9. Adiabatic runaway curves for the decomposition reaction of 10 wt.% MEKPO mixed with ferrous and ferric solutions scanned by VSP2 tests.

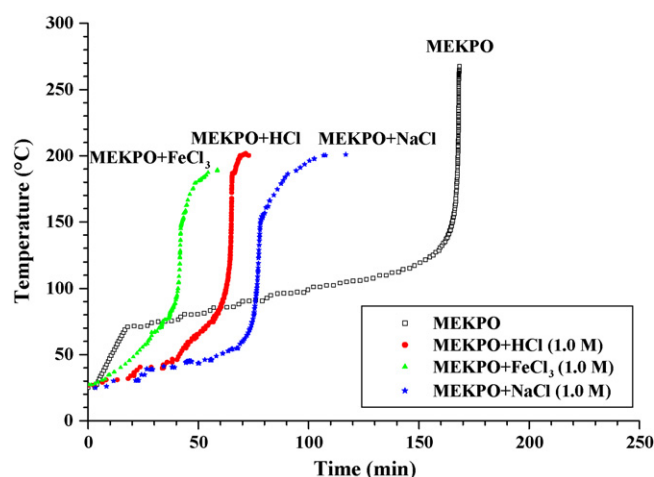


Fig. 10. Adiabatic runaway curves for the decomposition reaction of 10 wt.% MEKPO mixed with various solutions containing chloride ions scanned by VSP2 tests.

The experimental data obtained from the tests of 10 wt.% MEKPO mixed with incompatible materials are summarized in Table 3. The exothermic onset temperature of 10 wt.% MEKPO was  $101 \pm 1.5$  °C with  $\Phi$  of 1.19. The onset temperature is here defined at the temperature at which a self-heat rate exceeds  $0.1$  °C min<sup>-1</sup> detected in the VSP2 calorimeter.

#### 4. Discussion

By inspecting the thermal curve, one can see that the first decomposition region of MEKPO detected by the DSC is at ca. 96 °C. However, while the MEKPO mixed with incompatible substances, these onset temperatures were reduced by 20 °C and even as much as 57 °C compared to that of pure MEKPO. These observations indicate that the incompatibility could lead the thermal decomposition reaction of MEKPO to being increased and the probability of thermal hazards being enhanced as well. From Fig. 1 and Table 2, while MEKPO was mixed with inorganic acid, the first onset temperature was slightly decreased, and the exothermic curves demonstrated that the incompatible substances squeezed the second reaction peak into the first one. In Fig. 2, by mixing with alkaline solutions containing hydroxide

ions, the first peak of the reaction in terms of area was obviously decreased. On the other hand, in Table 2, the strong alkaline led the onset temperature to be significantly reduced even to being impending near the ambient conditions.

Figs. 3 and 4 show that ferric chloride and ferrous sulfate caused variations in thermal curves, also exhibiting incompatibility phenomena. These curves have two apparent exothermic peaks, indicating that the decomposition of MEKPO is also affected by ionic compounds. This indicates that the incompatible mixtures make the reaction mechanism more complicated than the one associated with the MEKPO alone.

Adiabatic calorimetry can always simulate the worst case scenario in bench scale under experimental design. MEKPO and its mixtures were thoroughly studied for the purpose of comparison. Thermal decompositions of 10 wt.% MEKPO under adiabatic conditions are displayed in Figs. 5 and 6. Adiabatic runaway data detected by VSP2, such as onset temperatures ( $T_0$ ), heats of decomposition ( $\Delta H_d$ ), adiabatic temperature rises ( $\Delta T_{ad}$ ), and self-heat rates ( $dT/dt$ ), etc., could be clearly compared to each other among these incompatible mixtures. The adiabatic curve only demonstrated one continuous runaway curve, which was different from the DSC thermal curve associated with several peaks. The trajectory appearing for the one curve for the adiabatic calorimeter (VSP2) lay in whether or not the first exothermic peak had released enough heat and raised the temperature to initiate the second reaction.

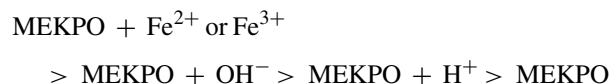
The adiabatic runaway onset temperature was at about 90 °C, for which a self-heat rate of 0.1 °C min<sup>-1</sup> existed as detected by VSP2. The maximum self-heat rate was at about 200 °C min<sup>-1</sup>. This told that the adiabatic runaway of MEKPO might lead to a thermal explosion thereafter. The effects of inorganic acids on MEKPO are presented in Fig. 7. Inorganic acids catalyze the decomposition of MEKPO justified from the earlier adiabatic runaway behaviors or from the lower onset temperatures. Hydrochloric acid is the most catalytic. Alkaline components also have similar influences as inorganic acids. Mixtures of MEKPO with alkaline solutions decompose much earlier than those of acidic solutions. Fig. 8 reveals the characteristics of the adiabatic runaway reaction of MEKPO mixed with alkaline solutions, which were compared to that of MEKPO itself.

Ferric chloride and ferrous sulfate were capable of simulating the impurities that polluted the MEKPO in a container made of iron or its alloys. Fig. 9 depicts the influences of these compounds and compared to the MEKPO itself. Ferric chloride seems to decompose the MEKPO at the instant of addition without any hot environment. The different effects of hydrochloric acid and sodium chloride on the catalytic decomposition are displayed in Fig. 10. From the adiabatic traces of runaway reactions, we can tell that hydrochloric acid is more incompatible with MEKPO than sodium chloride even in spite of containing the same chloride ion. Table 3 summarizes those incompatible behaviors detected by using VSP2 in adiabatic conditions.

## 5. Conclusions

By comparing the data of thermal analyses and adiabatic runaway reactions detected by using either the DSC or VSP2

calorimetries, we assessed the incompatibility rating of hazards on MEKPO contaminated with impurities. In summary, the labile or unstable sequences were proposed as follows:



According to the calorimetric observations of this study, MEKPO is a very hazardous, even dangerous, compound itself or mixed with other incompatible substances, especially with hydroxide ion or ferric materials. To actively and prudently prevent a system from any accident caused by MEKPO, it is imperative not only to clearly understand the hazardous characteristics of MEKPO, but also to avoid unexpected factors during processing, transportation, storage, or even disposal.

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