

Evaluation of thermal hazard for lauroyl peroxide by VSP2 and TAM III

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Abstract When above certain temperature limits, lauroyl peroxide is an unstable material. If the thermal source cannot be properly governed during any stage in the preparation, manufacturing process, storage or transport, runaway reactions may inevitably be induced immediately. In this study, the influence of runaway reactions on its basic thermal characteristic was assessed by evaluating thermokinetic parameters, such as activation energy (E_a) and frequency factor (A) by thermal activity monitor III (TAM III). This was achieved under five isothermal conditions of 50, 60, 70, 80, and 90 °C. Vent sizing package 2 (VSP2) was employed to determine the maximum pressure (P_{\max}), maximum temperature (T_{\max}), maximum self-heating rate ($((dT dt^{-1})_{\max})$), maximum pressure rise rate ($((dP dt^{-1})_{\max})$), and isothermal time to maximum rate ($(TMR)_{\text{iso}}$) under the

worst case. Results of this study will be provided to relevant plants for adopting best practices in emergency response or accident control.

Keywords Accident control · Lauroyl peroxide (LPO) · Runaway reactions · Thermal activity monitor III (TAM III) · Vent sizing package 2 (VSP2)

List of symbols

A	Pre-exponential factor/ $\text{m}^3 \text{mol}^{-1} \text{s}^{-1}$
C_v	Heat capacity under constant volume/ J kg^{-1}
E_a	Activation energy/ kJ mol^{-1}
$f(\alpha)$	Kinetic function depends on conversions/ dimensionless
k	Reaction rate constant/variables
k_0	Frequency factor/ $(\text{l/mol}) \text{n}1 \times (1/\text{s})$
m	Total mass of reactant/g
n	Reaction order/dimensionless
P	Thermal power or heat production rate/ $W = \text{J s}^{-1}$
Q	Heat/J
\dot{Q}	Heat flow/ J g^{-1}
SADT	Self-accelerating decomposition temperature/°C
T	Absolute temperature/K
T_{\max}	Maximum temperature during overall reaction/°C
T_0	Exothermic onset temperature/°C
T_f	Final temperature/°C
T_{NR}	Temperature of no return/°C
t	Time/sec
TMR_{iso}	Isothermal time to maximum rate/min, hr, or day
ΔH_d	Heat of decomposition/ J g^{-1}

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ΔH_{iso}	Heat of decomposition under isothermal condition/ J g^{-1}
$d\alpha dt^{-1}$	Reaction rate/ s^{-1}
$(dP dt^{-1})_{\text{max}}$	Maximum pressure rise rate/ bar min^{-1}
$(dT dt^{-1})_{\text{max}}$	Maximum self-heating rate/ $^{\circ}\text{C min}^{-1}$
Φ	Thermal inertial/dimensionless

Introduction

In the chemical industry, many production processes involve exothermic reactions. Thermal runaway may result from exothermic side reactions that begin at higher temperatures, following an initial accidental overheating of the reaction mixture. Numerous runaway incidents are thermally initiated. It is well recognized that an exothermic runaway reaction can occur if the heat generated by the reaction exceeds that removed by the surroundings. Therefore, thermodynamic data, kinetic parameters, and physical properties of the reactants together with the reactor conditions are required for assessing runaway reaction hazards. For storage and transport, the control and monitoring of critical parameters of unstable substances must be made available. Considerable mystique and partial information or even misinformation has surrounded organic peroxides (OPs), particularly as applied to process safety. Thus, there is a need for a better understanding of OPs.

Lauroyl peroxide (LPO) which is a strong free radical source containing more than 4.0% of active oxygen, is used as a polymerization initiator, catalyst, and vulcanizing agent. LPO generates the radicals $\text{CH}_3\cdot[\text{CH}_2]_{10}\cdot\text{CO}\cdot\text{O}\cdot$ quickly, so that the OP can be regarded as a source of the carbon-centered $\text{CH}_3\cdot[\text{CH}_2]_{10}$ radical [1, 2]. Thermal decomposition of lauroyl, decanoyl, and octanoyl peroxides occurs between 50 and 250 °C in a high molecular weight hydrocarbon solution [3]. During thermal cracking of polyolefin, these peroxides are convenient sources of C-11, C-9, and C-7 straight-chain aliphatic free radicals, whose reactions may be considered as models for the higher molecular weight radicals formed. Decomposition of such OPs indicates a relatively straightforward mechanism by which the diacyl peroxide breaks down by scission of the oxygen–oxygen bond to render acyloxy radicals.

According to the international transport regulations, LPO is classified as a solid OP (UN 3106). For storage and transport, the control and monitoring of critical parameters of unstable substances must be available and accessible. Kotoyori's study on LPO indicated that the phenomenon in which the exothermic decomposition reaction takes place parallel with the endothermic melting belongs to the quasi-AC type [4].

In this study, vent sizing package 2 (VSP2) and thermal activity monitor III (TAM III) were used to analyze the thermokinetic parameters and safety indices. VSP2 is an adiabatic calorimeter that can handle various stirring rates, volume of reactants, concentration of reactants, material of test cells, dosing rates, relief rates etc., to determine the effect of thermal runaway and explosion. VSP2 was employed to evaluate rates of increase for temperature and pressure in decompositions [5]. VSP2 and differential scanning calorimetry (DSC) was used to evaluate the self-accelerating decomposition temperature (SADT) of MEKPO in various storage vessels [6]. The intent of the analyses was to facilitate the use of various auto-alarm devices to detect over-pressure, over-temperature, and hazardous materials' leaks for a wide spectrum of operations. Results indicated that LPO decomposition is detected at temperatures 50–60 °C, and the rate of decomposition was shown to exponentially increase with temperature and pressure. Determining time to maximum rate (TMR), SADT, maximum temperature (T_{max}), exothermic onset temperature (T_0), and heat of decomposition (ΔH_d) is essential for identifying early stage runaway reactions effectively for process industries. The aim of establishing the thermokinetic parameters of this study was to establish an emergency response process to effect loss prevention. An emergency response plan is mandatory and necessary to cope with reactive chemicals under upset scenarios during plant operations.

Experimental method

Standard sample

95 mass% LPO was directly purchased from the Fluka Co., and both density and concentration were measured. Then, LPO was stored in a refrigerator at 4 °C.

Isothermal tests by TAM III

TAM III was used to investigate the runaway reaction at 50, 60, 70, 80, and 90 °C. Absolute temperature can be adjusted to within 0.02 K, while operating in isothermal mode; the bath mean temperature fluctuations were within 10^{-5} K. A maximum scanning rate is $\pm 2 \text{ K h}^{-1}$ for chemical and physical equilibrium. We applied the software of TAM III assistant to govern the thermostat. The thermostat liquid is mineral oil with a total volume of 22 L, and the temperature range of the thermostat is 15–150 °C when mineral oil is employed.

The thermal power is determined by Eqs. 1–3 [7].

$$P = \frac{d\alpha}{dt} \Delta H_d \quad (1)$$

$$\frac{d\alpha}{dt} = kf(\alpha) \quad (2)$$

$$P = \Delta H_d kf(\alpha) \quad (3)$$

Equation 2 indicates the function of reaction rate, and then $d\alpha dt^{-1}$ is the reaction rate (s^{-1}); k is the constant of reaction rate; $f(\alpha)$ is the kinetic function depending on conversions; P is the thermal power or heat production rate ($W = J s^{-1}$); ΔH_d is the enthalpy change or heat of decomposition ($J g^{-1}$).

Adiabatic tests by VSP2

VSP2 manufactured by Fauske & Associates, Inc. [8, 9] was applied to obtain thermokinetic and thermal hazard data, such as temperature and pressure traces versus time. A PC-controlled adiabatic calorimeter (VSP2), the well-known turn-on heat-wait-search (H-W-S) mode for detecting the self-heating rate, was adopted for VSP2. Under heating conditions, the main heater will turn-on to heat the sample to the pre-set temperature, then on a guard heater is turned on to sustain an adiabatic environment. Under waiting conditions, stirring will make the sample temperature more uniform. Under searching conditions, it is considered that the reaction has started into the self decomposition reaction as the sample temperature rise rate is more than apparatus sensitivity ($0.05 \text{ }^\circ\text{C min}^{-1}$). The adiabatic operation allows direct application of the temperature and pressure rise rate data to large-scale process vessels. The data are useful for determining the adiabatic heat of reaction, as well as the adiabatic reaction rate used for pressure relief system design [10].

The low heat capacity of the cell guaranteed that almost all the reaction heat that was released remained within the tested sample. Thermokinetic and pressure behaviors in the same test cell (112 mL) usually could be tested, without any difficult extrapolation to the process scale due to a low thermal inertia factor (Φ) of about 1.05 and 1.32. The low Φ permits bench scale simulation of the worst credible case, such as incorrect dosing, cooling failure, or external fire conditions. In addition, to avoid bursting the test cell and losing all the exothermic data, the VSP2 tests were run at low concentration or smaller amount of reactants. Accordingly, VSP2 was used to evaluate the essential thermokinetics for LPO.

Result and discussion

Thermal decomposition analysis for TAM III

The reaction curves of LPO under the isothermal conditions were acquired at several temperatures: 50, 60, 70, 80,

and 90 $^\circ\text{C}$ in TAM III. The reaction occurred in solid state, since no endothermic effect was observed at 50 $^\circ\text{C}$. On the 50 $^\circ\text{C}$ reaction curve, the obtained data are plotted as shown in Fig. 1; at first heat flow increased, and after reaching a summit it decreased. The shape of the reaction curves is characteristic of an autocatalytic reaction. These are comprised of two main constituents in the thermal decomposition of OPs: (1) homolysis of the O–O bond and (2) radical-induced decomposition [11, 12].

At 50 $^\circ\text{C}$ constant temperature, thermal decomposition of LPO an intermediate radical [$C_{11}H_{23}COO\cdot$] was produced, and thus it triggered off the rate of reaction, as well as heat flow, to increase gradually until reaching the summit. During this stage the intermediate radical behaves as product, the concentration of which is increasing in value, though its generation and consumption are simultaneously happening all the time. After the summit, the consumption of the intermediate product tends to be dominant and the heat flow decreases with the time. Thus, the conversion ratio of the intermediate product [$C_{11}H_{23}COO\cdot$] mandates the rate of reaction.

The thermal decomposition of the LPO at the 60, 70, 80, and 90 $^\circ\text{C}$ demonstrates the differences in reactivity, the shape of reaction curve as shown in Figs. 2, 3, 4, and 5. Decompositions of LPO at 60, 70, 80, and 90 $^\circ\text{C}$ are controlled by the thermal homolysis in which decarboxylation takes place to a very high extent. After the samples were almost melted, heat flow suddenly leaped to the maximum and then declined exponentially, exhibiting the shape of reactions. It implies that the reactions of LPO closely follow nth-order because of the exponential decays of heat flows on the curves [13]. In contrast, it is more likely controlled by the induced decomposition and autocatalytic reaction at 50 $^\circ\text{C}$.

We have used isothermal time to maximum rate (TMR_{iso}) as a safety index to assess the degree of hazard. Results by TAM III tests are indicated in Table 1. TAM III

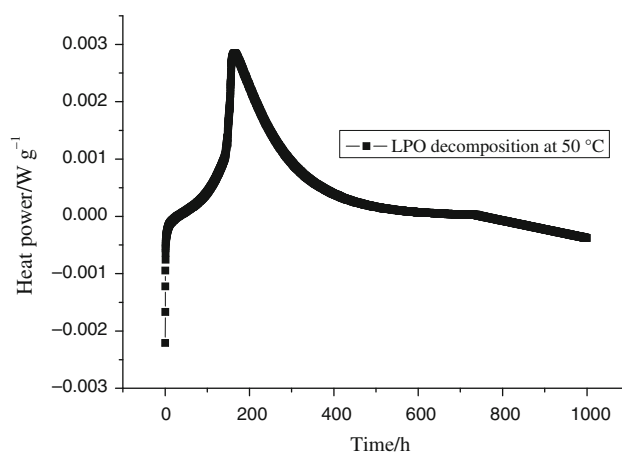


Fig. 1 Heat flow versus time for the thermal decomposition of 95 mass% LPO at 50 $^\circ\text{C}$ by TAM III test

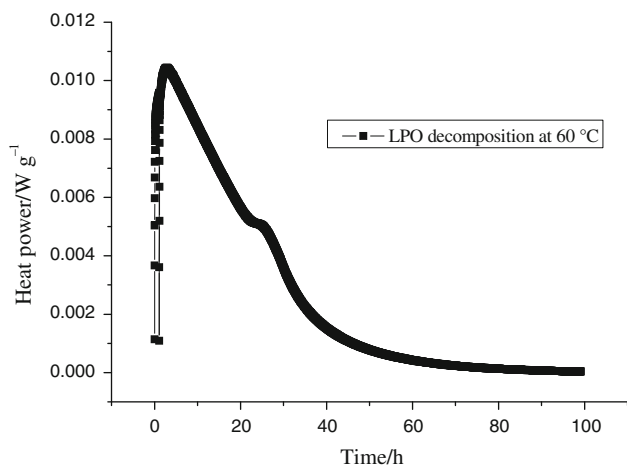


Fig. 2 Heat flow versus time for the thermal decomposition of 95 mass% LPO at 60 °C by TAM III test

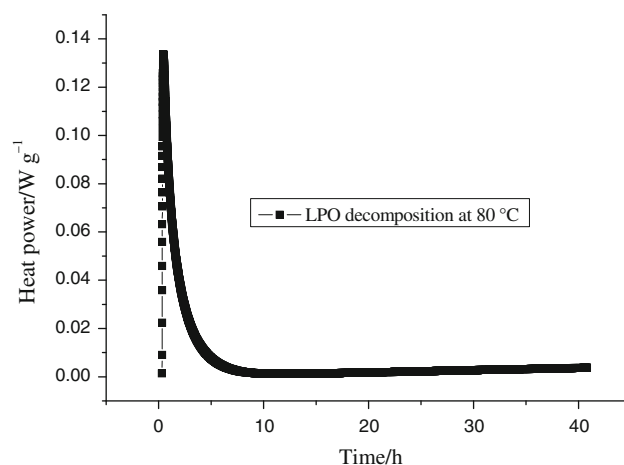


Fig. 4 Heat flow versus time for the thermal decomposition of 95 mass% LPO at 80 °C by TAM III test

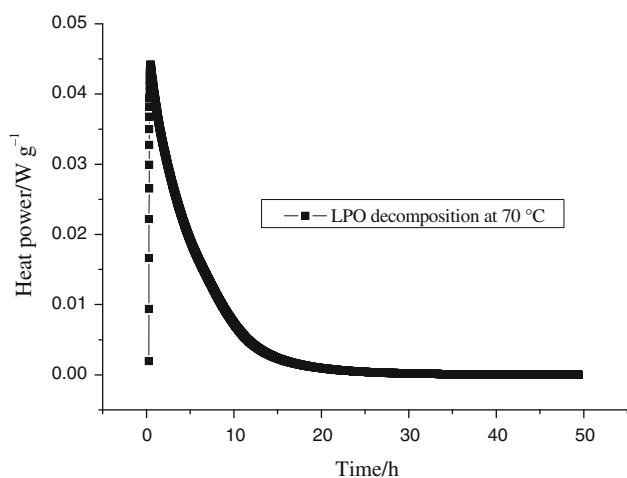


Fig. 3 Heat flow versus time for the thermal decomposition of 95 mass% LPO at 70 °C by TAM III test

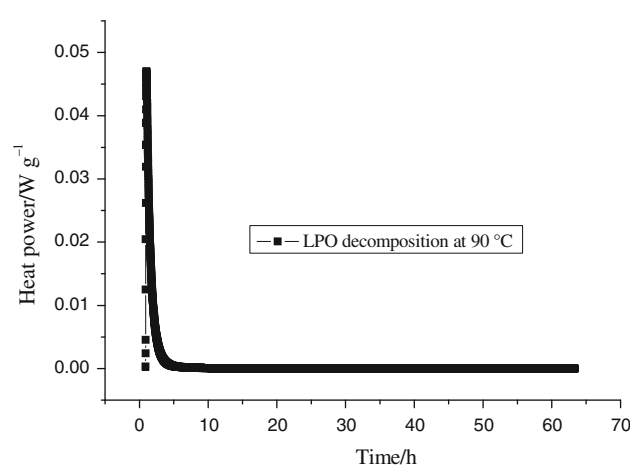


Fig. 5 Heat flow versus time for the thermal decomposition of 95 mass% LPO at 90 °C by TAM III test

isothermal results showed the TMR_{iso} of LPO mixed with for 50, 60, 70, 80, and 90 °C were about 164, 2.3, 0.52, 0.142, and 0.05 h, respectively. By these experimental data, we could significantly realize a tendency that the TMR_{iso} clearly increased with lower isothermal temperature. We found only 30 min to response situation when the temperature reached 70 °C; there was only 3 min to respond to any situation when the temperature reached 90 °C.

The calculation of thermokinetic parameters

The results of E_a for LPO and with inorganic acid and base by employing the Arrhenius equation are as shown in Eq. 4 [14–16]:

$$k(T) = A \exp\left(\frac{-E_a}{R T}\right) \quad (4)$$

where A , E_a , R , and T represent frequency factor, activation energy, gas constant ($R = 8.314 \text{ J mol}^{-1} \text{ K}^{-1}$), and absolute temperature, respectively.

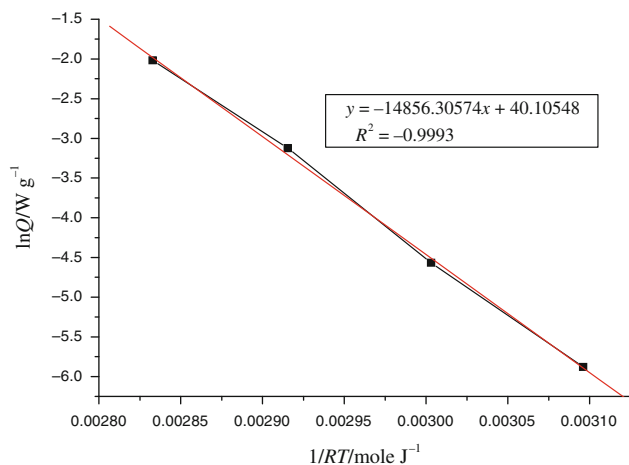
It has been known, i.e., Eq. 5:

$$\ln k = \ln A - \frac{E_a}{R T} \quad (5)$$

The plot of $\ln k$ versus $1/T$ is expected to be a straight line. One of the Arrhenius kinetic parameters, E_a , can be calculated from the plot accordingly (Fig. 6). Calculated from Arrhenius equation, the value of E_a is $123.5 \text{ kJ mol}^{-1}$ at 50, 60, 70, and 80 °C heat powers for 95 mass% LPO. From TAM III tests, we investigated LPO at 90 °C, but under higher isothermal conditions because the thermal

Table 1 Experimental data by TAM III tests for 95 mass% LPO

Sample	Temperature/°C	Mass/mg	Cell	TMR _{iso} /hr	Heat power/W g ⁻¹	ΔH _d /J g ⁻¹
LPO	50	68.65	Glass	164.000	0.0028	744.8
	60	59.40	Glass	2.300	0.0104	899.2
	70	65.86	Glass	0.520	0.0440	887.3
	80	60.80	Glass	0.142	0.1330	839.6
	90	103.30	Glass	0.050	0.0500	105.3

**Fig. 6** Determination of E_a (activation energy) from the slope of $\ln Q$ versus $1/RT$

reaction started before the samples reached thermal equilibrium. From this viewpoint, as calculated from the Arrhenius equation of the value of E_a , we did not consider change of heat flow at 90 °C.

Thermal decomposition analysis for VSP2

The analysis of thermokinetic parameters

VSP2 was used to acquire various thermokinetic parameters [17–20]. Related parameters, such as self-heating rate (°C min⁻¹), pressure rise rate (bar min⁻¹), and exothermic onset temperature (T_0) can be accurately obtained on an adiabatic system. In view of the clear changes of temperature and pressure, the degree of hazard can be defined explicitly. We used LPO for measuring the runaway phenomenon on adiabatic conditions. The adopted function of VSP2 was the H–W–S mode.

Table 2 indicates T_0 , T_{\max} , P_{\max} , $(dT dt^{-1})_{\max}$, and $(dP dt^{-1})_{\max}$ for LPO. The VSP2 time versus pressure plot for thermal decomposition LPO is indicated in Fig. 7. The VSP2 time vs temperature plot for thermal decomposition LPO is in Fig. 8. The LPO is very dangerous because it began self-heating at 50 °C and T_{\max} reached 196 °C. From the VSP2 tests, the VSP2 pressure rise rate vs temperature plot for thermal decomposition LPO is in Fig. 9. Self-heating rate vs temperature from VSP2 experimental data for the thermal decomposition 95 mass% LPO is shown in Fig. 10.

According to Figs. 9 and 10, the properties of LPO of solid decomposed to liquid and were transferred to vapor suddenly. The phenomenon could be explained under the isothermal storage conditions of TAM III. First, the exothermic decomposition reaction of LPO began in the liquid, while the liquid and the solid phases coexisted. Second, the heat flux from the exothermically decomposing liquid was applied to the endothermic melting of the solid. Moreover, the system kept being under the quasi-isothermal conditions till the melting was completed [8, 9]. From VSP2 data, the temperature stayed between 48–50 °C about 12 min; the system was maintained under quasi-isothermal conditions and then the system remained at self-heating 2 °C min⁻¹ until 60 °C; the system started to self-heat abruptly and ran away rapidly [4, 13]. In this connection, T_0 and temperature of no return (T_{NR}) were 50 and 60 °C, respectively.

The evaluation of thermokinetic parameters by VSP2

Thermal hazard investigations are widely used for early stage of prevention. This study should provide warning information of OPs' tests by various calorimeters. If the sensible heat is absorbed by a vessel, it cannot be negligible. The energy balance equation of the adiabatic reaction system is described as Eq. 6:

Table 2 Calorimetric data from the dynamic scanning experimental of 95 mass% LPO by VSP2

Sample	Sample mass	Φ	$T_{NR}/^{\circ}\text{C}$	$T_{\max}/^{\circ}\text{C}$	P_{\max}/psi	$(dT dt^{-1})_{\max}/^{\circ}\text{C min}^{-1}$	$(dP dt^{-1})_{\max}/\text{bar min}^{-1}$	$E_a/\text{kJ mol}^{-1}$
LPO	3.37 g	1.1	60	196.44	6.36	6648.38	495.16	84.42

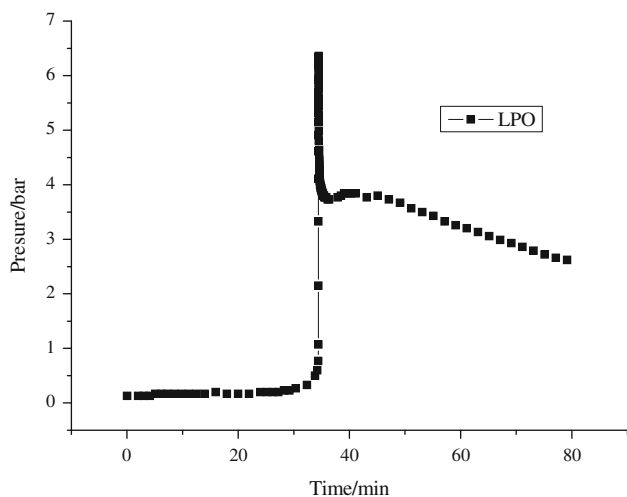


Fig. 7 Time versus pressure by the VSP2 experiment for thermal decomposition of 95 mass% LPO

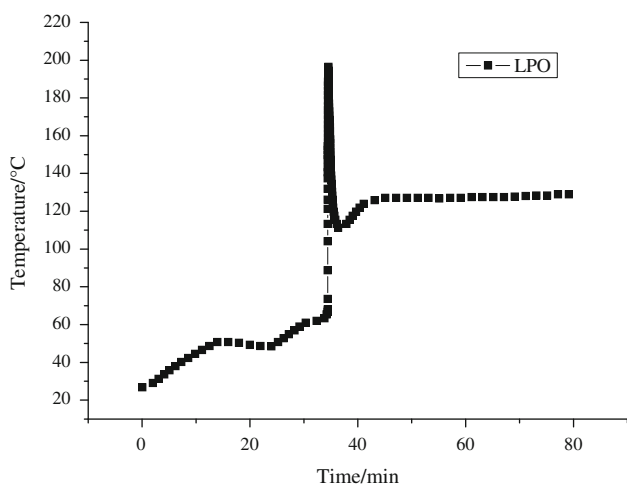


Fig. 8 Time versus temperature by the VSP2 experiment for thermal decomposition of 95 mass% LPO

$$\Phi m C_v \frac{dT}{dt} = (-\Delta H)rV \quad (6)$$

The reaction rate equation is expressed as Eqs. 7 and 8:

$$r = -\frac{d\alpha}{dt} \quad (7)$$

$$\ln k = \ln k_0 - \frac{E_a}{RT} = \ln \frac{\frac{dT}{dt}}{C_0^{n-1} \left(\frac{T_f - T}{T_f - T_0} \right)^n (T_f - T_0)} \quad (8)$$

Equation 3 can be used to determine the E_a at the same time [21, 22]. The calculated E_a are shown in Table 2. When LPO is dissolved completely, it causes instantaneous runaway and generates huge pressure and huge exothermic heat. We employed VSP2 to derive the E_a value. VSP2 is very suitable for the material with slowly-decomposed and

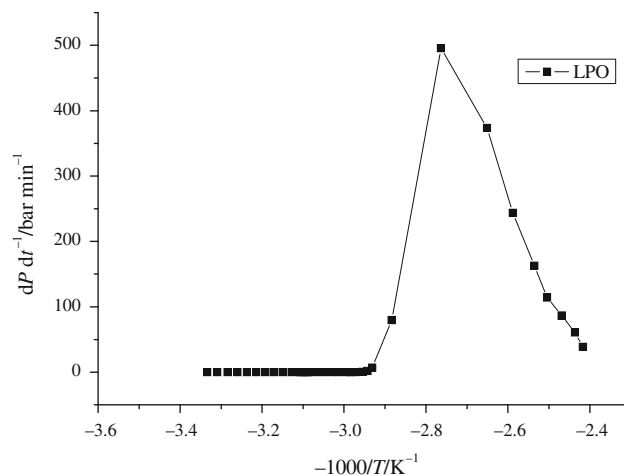


Fig. 9 Pressure rise rate versus temperature from VSP2 experimental data for the thermal decomposition 95 mass% LPO

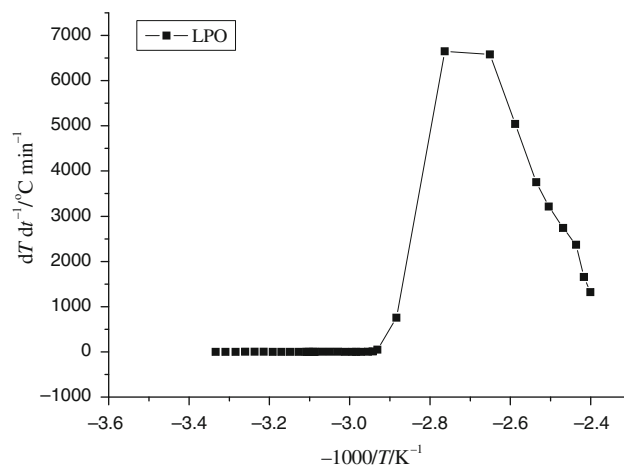


Fig. 10 Self-heating rate versus temperature from VSP2 experimental data for the thermal decomposition 95 mass% LPO

sluggishly exothermic property. Therefore, we concluded that the E_a value from VSP2 might not be useful for the situation.

Conclusions

According to the data and evidence collected in this study, under isothermal storage conditions, the exothermic decomposition reaction of LPO begins in the liquid phase, while the liquid and the solid phase coexist. The heat flux from the exothermically decomposing liquid is, in turn, utilized for the endothermic melting of the solid, so that the system is maintained under quasi-isothermal conditions until the melting is completed. Under isothermal conditions, there was only 3 min to respond when the

temperature was at 90 °C. In this connection with VSP2, LPO was subjected to an isothermal storage test; it started to self-heat abruptly and rapidly, as the maximum self-heating rate $(dT dt^{-1})_{\max}$, and maximum pressure rise rate $(dP dt^{-1})_{\max}$ were 6648 °C min⁻¹ and 495 bar min⁻¹, respectively.

LPO will not initiate any prominent runaway reaction unless it undergoes through the melting stage under higher temperature, so it must be properly maintained at low temperature during transport and storage. In practice, these data are necessary for safe application, storage, and transport of a reactive chemical of interest. In addition, if a bulk quantity LPO were heated to 40–50 °C, it would slowly self-heat until the melt occurred and then engage in a swift thermal runaway.

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