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FLAMMABILITY STUDIES OF 3-METHYL PYRIDINE/WATER SYSTEM

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In industrial processes, information on the safety property of chemicals is essentially crucial for safe handling during unit operations. Ensuring the safe use of combustible or flammable substances in processes is unlikely without detailed investigations of their flammability characteristics and related hazards. We studied 3-methyl pyridine (3-picoline), e.g., flammability limits (LFL/UFL), maximum explosion pressure (P_{max}), maximum explosion pressure rise $(dP/dt)_{max}$, minimum oxygen concentration (MOC), vapor deflagration index (K_g), and characterized the influence of inert steam (H_2O) on critical parameters for 3-picoline/water mixtures at 270°C, 1 atm, various oxygen concentrations, and vapor mixing ratios (100/0, 30/70, 10/90 and 5/95 vol.%) with a 20-L-Apparatus in simulated conditions, respectively.

The results showed that the flammability characteristics of 3-picoline$_{(aq)}$ all increased with the oxygen concentration. However, as the composition of inert steam increased, the flammability parameters and the degree of fire and explosion hazards were significantly reduced, instead. This study elucidated the flammability properties of 3-picoline mixed with inert steam. The conclusions could be applied to proactively prevent the relevant processes from incurring fire and explosion accidents.

Keywords: *fire and explosion hazards, flammability characteristics, inert steam, 20-L-Apparatus, 3-methyl pyridine (3-picoline)*

Introduction

Chemicals are frequently and widely used not only in modern petrochemical plants but in our daily lives as well. However, under various abnormal scenarios these substances may create seriously contingent situations. The three most common chemical plant accidents are fires, explosions, and toxic releases, in that order [1]. The history of the chemical process industries is replete with major accidents [2], as has been confirmed by a series of accidents that have occurred around the world specifically involving fires and explosions [3]. Ensuring the fire and explosion safety of combustible or flammable substances used in processes is unlikely without detailed investigations of their flammability characteristics and pertinent hazards. Many studies which emphasize fire and explosion hazard evaluation and analysis [4–16] have been presented, and problems can often be traced to an insufficient knowledge of the hazardous properties of the substances used. If determined carefully and applied properly, safety-related properties will provide information on the reaction behaviors and fire and explosion hazards of the specific substance [17].

3-Methyl pyridine (3-picoline) is the main material for producing vitamin B_3 and also is an important chemical for various medical and agricultural applications in industry. The annual throughput of 3-picoline in Taiwan is 3500 tons mainly produced by Chang Chun Petrochemical Co., Ltd. [18], in Taiwan. Global production is approximately 20000 tons per year, so the demand is quite substantial. In general, 3-picoline is used under 250–350°C in processes where its liquid and vapor is highly flammable and quite easily spread. Thus, serious fires and/or explosions may occur if an ignition source is incidentally encountered in the process [18]. Prevention of explosions has normally been based upon the recognition of material hazards [19], and knowledge of material safety properties is essential for safe handling during unit operations [17]. Nevertheless, to date, safety information for 3-picoline has generally been insufficient, with no basic understanding of its safety-related properties tested in various simulated conditions based on a real process. Such information is critically important, specifically for the effect of inert steam (H_2O) and variations in flammability characteristics from different concentrations, and oxygen concentrations for a specific substance.

Therefore, according to the course of operation, this study started with an initial temperature of 270°C, initial pressure of 1 atm and various oxygen concentrations, together with four typical 3-picoline vapor mix-

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ing ratios (100/0, 30/70, 10/90 and 5/95 vol.%) produced in process measurements by a 20-L-Apparatus in series to obtain the flammability properties of lower flammability limit (LFL), upper flammability limit (UFL), maximum explosion pressure (P_{max}), rate of maximum explosion pressure rise ($(dP/dt)_{max}$), vapor deflagration index (K_g), and minimum oxygen concentration (MOC). We attempted to investigate the influence of inert steam for a 3-picoline/water system and define those fire and explosion properties in various cases. The aim was to study the effect between several initial operating conditions for critical flammability characteristics and thereby identify the degree of hazards within the 3-picoline application processes. With the experimental results, we could provide specific information for related industries and help them prevent unexpected accidents in the future.

Experimental

Materials

Reagent (3-picoline)

3-Picoline, or so-called 3-methyl pyridine, which has the chemical formula of C_6H_7N, is reacted by formaldehyde, acetic aldehyde, and ammonia by the following reaction [18]:

$$2CH_3CHO+2HCHO+NH_3 \rightarrow C_6H_7N+4H_2O \quad (1)$$

3-Picoline is a yellow, liquid. Figure 1 displays its chemical structure. The basic physical and chemical properties of 3-picoline are given in Table 1.

C_6H_7N

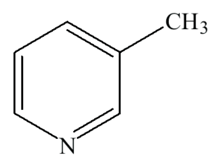

Fig. 1 3-Picoline's chemical structure

Table 1 Basic chemistry and physical properties of 3-picoline

Caracteristic	3-Picoline
Formula	C_6H_7N
Molecular mass/g mol^{-1}	93.1
Flash point/°C	40
Boiling point (1 atm)/°C	143–144
Vapor pressure (25°C)/atm	0.008
Specific gravity (H_2O=1)	0.982
Flammability limits/vol.%	1.3–8.7
CAS No.	108-99-6

Sample preparation

In this study, pure 3-picoline (100 vol.%) was directly acquired from Chang Chun Petrochemical Co., Ltd. in Taiwan for preparing four 3-picoline aqueous mixtures with different concentrations as experimental samples (100, 70, 30 and 5 vol.%). We deliberately mixed the pure 3-picoline with water to simulate various vapor mixing ratios (100/0, 30/70, 10/90 and 5/95 vol.%) produced in a practical process.

Initial conditions

Initial pressure and temperature of 1 atm and 270°C, along with four designed samples and various oxygen concentrations were studied to evaluate the fire and explosion hazards under various testing conditions.

Concerned about the initial temperature, we chose 270°C referring to the normal operating temperature between 250–350°C in process. Because the combustion of liquid fuels takes place in the vapor phase [20], we set the initial temperature as 270°C in order to exceed 3-picoline's normal boiling point (143–144°C, according to Table 1) forming total flammable vapors and ensuring the test was in a good mixing state in the vapor phase.

Methods

20-liter spherical explosion vessel (20-L-Apparatus)

Investigations were carried out on the experimental set-up presented in Fig. 2 [16]. A 20-L-Apparatus (20-liter spherical explosion vessel) was purchased from Adolf Kühner AG and was available for this study. The flammability of the 3-picoline/water mixtures was examined in this device and its control system. In general, the main structure of the 20-L-Apparatus can be roughly separated into four parts: spherical explosion vessel, heating and circulation device, pressure setting system, and transmission computer interface, as illustrated in

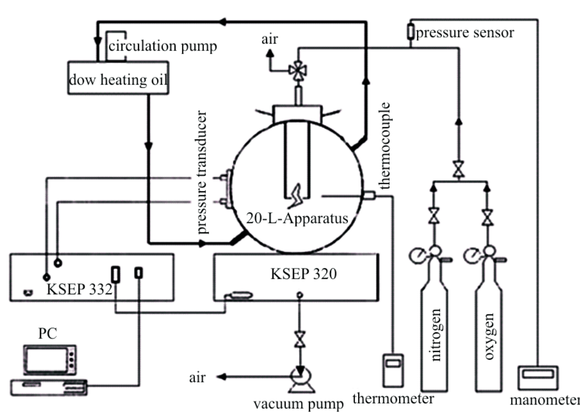

Fig. 2 Schematic diagram of the experimental set-up and its control system [16]

Fig. 2. The test chamber is a stainless steel hollow sphere with a personal computer interface. The top of the cover contains holes for the lead wires to the ignition system. The opening provides for ignition by a condenser discharging with an auxiliary spark gap, which is controlled by the KSEP 320 unit of the 20-L-Apparatus [16]. The KSEP 332 unit uses two 'Kistler' piezoelectric pressure sensors on the flange to measure the pressure as function of time [21]. A comprehensive software package KSEP 6.0 is available, which allows safe operation of the test equipment and an optimum evaluation of the explosion test results [20].

In the past, various types of test apparatus for fire and explosion characteristics have been proposed. Among these devices, the 20-L-Apparatus has the highest reliability because of its standard spherical shape [22, 23]. The test system is able to determine a material's inherent safety properties in accordance with internationally recognized test procedures, e.g., ASTM 1226 (American Society for Testing and Materials, USA) and VDI 2263 (Verein Deutscher Ingenieure, Germany). Essentially, it is suitable for measuring explosion behaviors of combustible materials, such as solvent vapors, flammable gases, or combustible dusts and deriving the flammability properties of LFL, UFL, P_{max}, $(dP/dt)_{max}$, K_g and MOC in the series of test procedures. We could consider normal operation and set various simulating conditions for preventive measures against fire and explosion hazards by investigating the flammability safety-related properties with this equipment.

LFL and UFL for gas and solvent vapors

Flammability limits include the LFL and UFL. The flammability range is from LFL to UFL of a specific substance. Vapor-air mixtures will ignite and burn only over a well-specified range of compositions [1]. The LFL/UFL of a gas or vapor is the lowest/highest concentration at which a gas or vapor explosion is not detected in three successive tests [21]. The 3-picoline/water mixtures were evaporated from liquid to vapor phase at 270°C for testing in this work.

P_{max}, $(dP/dt)_{max}$ and K_g

The explosion indices, P_{max} and $(dP/dt)_{max}$, are defined as the mean values of the maximum values of all three series. Subsequently, the vapor deflagration index (K_g) is calculated from $(dP/dt)_{max}$ by means of the Cubic law [23]:

$$V^{1/3}(dP/dt)_{max}=K_g \quad (2)$$

where K_g and V are the maximum gas explosion constant specific to the gas and the volume of test apparatus (i.e., 0.02 m^3), respectively.

As there are many gas products and industrial practices, it is appropriate to assign this maximum constant to one of several explosion classes (St), as given in Table 2, and to use these as a basis for sizing explosive relief [17, 21, 23].

Table 2 K_g and explosion classes (St) [17, 21, 23]

K_g/mbar s^{-1}	Explosion classes (St)
<1	St-0
1–200	St-1
201–300	St-2
>300	St-3

Minimum oxygen concentration (MOC)

MOC, or the so-called limiting oxygen concentration (LOC), is an especially useful parameter, because explosions and fires are preventable by reducing the oxygen concentration regardless of the concentration of the fuel. This concept is the basis for the common procedure called inerting [1, 24]. When oxygen concentration is less than the MOC, the reaction cannot generate enough energy to heat the entire gas mixtures (including the inert) to the extent required for the self-propagation of the flame [1] under this circumstance; oxygen is the key ingredient and a MOC is required to propagate a flame.

After the first test series in normal air (O_2=21 vol.%), the second series run in oxygen concentration will be about 17 vol.% in total O_2+N_2 (that means $[O_2]/([N_2]+[O_2])$=17 vol.%), for determining the P_{max} and K_g explosion indices. The tests, in turn, have to be continued by systematic reduction, say 3 to 4 vol.% each time of the oxygen concentration in nitrogen (that means $[O_2]/([N_2]+[O_2])$) until gas explosions are no longer possible [17] and this value is detected at least three series in a row [21].

Results and discussion

Different concentrations (vapor mixing ratios)

In this study, four concentrations of 3-picoline$_{(aq)}$ were measured in compliance with their typical 3-picoline/water mixing ratios (100/0, 30/70, 10/90 and 5/95 vol.%) for a flammability test. In a normal air condition of 21 vol.% O_2 and 1 atm, these derived flammability characteristics are also presented in Table 3, indicating their relationship: the highest UFL was 8.00 vol.% measured from the 100/0 vol.% 3-picoline/water mixture; whereas the lowest UFL was 2.12 vol.% measured from the 5/95 vol.% 3-picoline/water mixture. The 100/0 vol.% 3-picoline/water mixture had the widest flammability range from 1.40 vol.% (LFL) to 8.00 vol.% (UFL), and the

Table 3 Flammability characteristics for different 3-picoline/water mixing ratios at 270°C, 21 vol.% O_2, and 1 atm

Mixing ratio 3-picoline/water/ vol.%	LFL/ vol.%	UFL/ vol.%	Flammability range/ vol.%	P_{max}/ bar	$(dP/dt)_{max}$/ bar s^{-1}	K_g/ mbar s^{-1}	Explosion class (St)
100/0	1.40	8.00	6.60	2.50	181.00	49.13	St-1
30/70	1.10	5.00	3.90	2.30	130.00	35.29	St-1
10/90	1.09	3.10	2.01	1.80	53.00	14.39	St-1
5/95	1.13	2.12	0.99	0.70	6.00	1.63	St-1

5/95 vol.% 3-picoline/water mixtures had the narrowest one from 1.13 vol.% (LFL) to 2.12 vol.% (UFL) at 21 vol.% O_2, 1 atm. Table 4 added various oxygen concentrations up for indicating these safety-related properties completely. Test results displayed in Table 5 illustrate the variations of flammability range, UFL, P_{max}, $(dP/dt)_{max}$ and K_g compared with 3-picoline/water 100/0 and 5/95 vol.%, emphasizing the influence of inert steam for flammability characteristics in 3-picoline/water mixing process conditions both in the conditions of 270°C, 21 vol.% O_2 and 1 atm.

Effects on inert steam

Different mixing ratios of 3-picoline/water mixtures represented their self-component of inert steam and were utilized to confirm the influence of inert steam on fire and explosion characteristics for 3-picoline/water system within the process. The results in Fig. 3 reveal that as the component of inert steam increased, not only did the flammability range of 3-picoline become narrow, but also the degree of explosion damage, e.g., maximum explosion pressure (P_{max}), was reduced. Thus, it is clear that, through the influence of inert steam in the figure, the flammability range *vs*. P_{max} curves was getting narrower and narrower as long as the component of inert steam was raised. From Figs 4–7, we used three-dimensional schemes with three coordinates of P_{max}, oxygen concentration and 3-picoline/water mixing concentration marked with flammability experimental-derived data of this study displayed the relationships to P_{max} *vs*. 100, 30, 10, 5 vol.% 3-picoline/water mixtures with various oxygen concentrations at 270°C, 1 atm. It is clearly shown that the degrees of fire and explosion hazard of P_{max} and the flammability range had narrowed down over test series significantly and progressively. Flammability characteristics in all conditions are presented in Tables 3 and 4.

Table 4 Fire and explosion characteristics for different of 3-picoline/water mixing ratios at various vol.% O_2, 270°C and 1 atm

$O_2/(N_2+O_2)$/ vol.%	$O_{2\,LFL}/O_{2\,UFL}$/ vol.%	LFL/ vol.%	UFL/ vol.%	P_{max}/ bar	$(dP/dt)_{max}$/ bar s^{-1}	K_g/ mbar s^{-1}	Explosion class (St)
3-picoline/water (100/0 vol.%)							
11	–	–	–	–	–	–	–
12	11.7/11.6	1.84	3.29	0.60	2.00	0.54	St-0
14	13.8/13.4	1.32	4.30	1.90	28.00	7.60	St-1
17	16.7/16.1	1.57	5.13	2.20	94.00	25.51	St-1
21	20.7/19.4	1.40	8.00	2.50	181.00	49.13	St-1
3-picoline/water (30/70 vol.%)							
11	–	–	–	–	–	–	–
12	11.5/10.9	1.38	2.68	0.70	2.00	0.54	St-0
14	13.4/12.7	1.26	3.00	1.40	19.00	5.16	St-1
17	16.3/14.6	1.26	4.06	1.90	51.00	13.84	St-1
21	20.3/17.5	1.10	5.00	2.30	130.00	35.29	St-1
3-picoline/water (10/90 vol.%)							
14	–	–	–	–	–	–	–
14.5	12.8/11.9	1.21	1.86	0.80	2.00	0.54	St-0
15	13.2/12.0	1.23	2.00	1.00	2.00	0.54	St-0
16	14.1/12.7	1.21	2.05	1.10	2.00	0.54	St-0
17	14.9/12.8	1.22	2.47	1.10	6.00	1.63	St-1
21	18.7/14.5	1.09	3.10	1.80	53.00	14.39	St-1
3-picoline/water (5/95 vol.%)							
17	16.2/12.1	–	–	–	–	–	–
21	15.9/12.5	1.13	2.12	0.70	6.00	1.63	St-1

–: not detectable

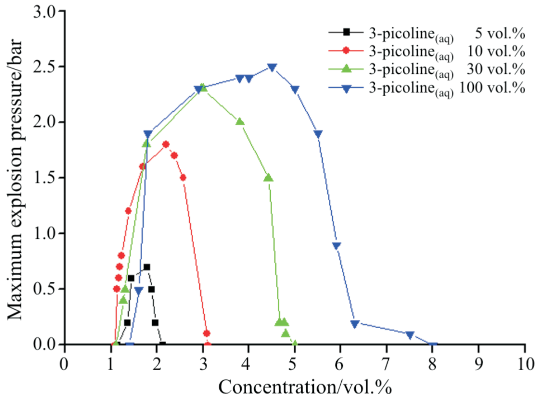

Fig. 3 P_{max} *vs*. 3-picoline$_{(aq)}$ with four vapor mixing ratios at 21 vol.% O_2, 270°C and 1 atm

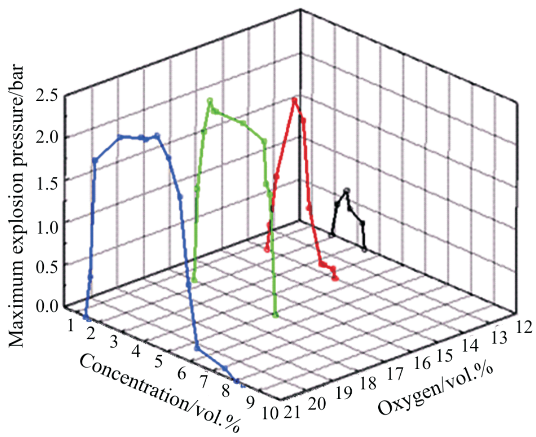

Fig. 4 P_{max} *vs*. 100 vol.% 3-picoline$_{(aq)}$ with various oxygen concentrations at 270°C and 1 atm

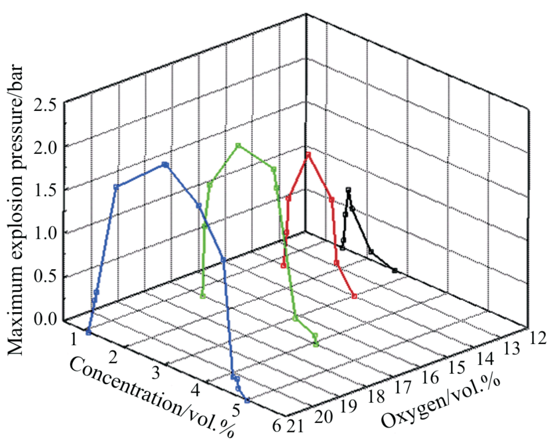

Fig. 5 P_{max} *vs*. 30 vol.% 3-picoline$_{(aq)}$ with various oxygen concentrations at 270°C and 1 atm

Nitrogen (N_2), carbon dioxide (CO_2) and steam are regarded as the typical inert vapor widely used in industrial processes. The combustible concentration of gaseous fuel may vary with the addition of inert vapor; the inert vapor here will conspicuously reduce the degree of mixing of fuel and air to restrain its combustion hazard. We deliberately tried to confirm

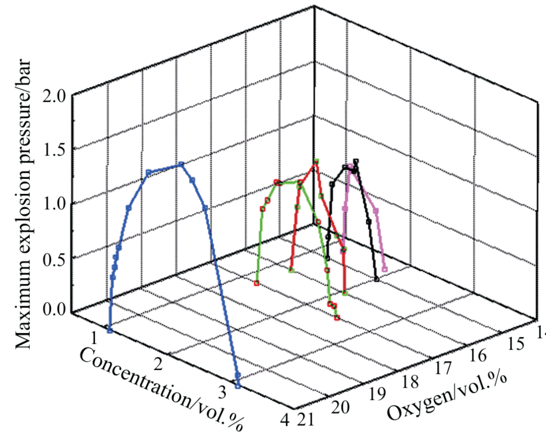

Fig. 6 P_{max} *vs*. 10 vol.% 3-picoline$_{(aq)}$ with various oxygen concentrations at 270°C and 1 atm

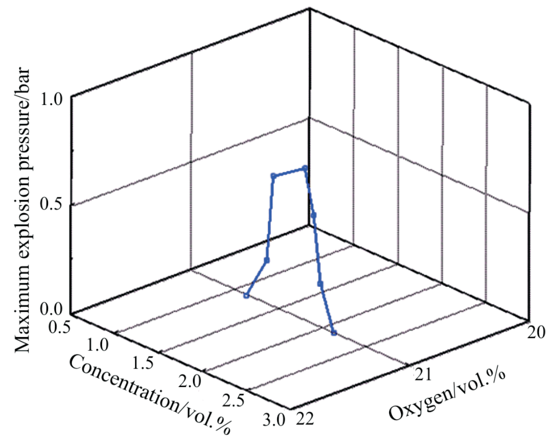

Fig. 7 P_{max} *vs*. 5 vol.% 3-picoline$_{(aq)}$ with various oxygen concentrations at 270°C and 1 atm

the characteristics of inert steam and this influence on the flammability property of 3-picoline/water mixtures. According to experimental results, we found that when the component of inert steam is more than 97 vol.% (3-picoline/water, 3/97 vol.%), the vapor of 3-picoline will no longer be ignited.

For the case of 100 and 5 vol.% 3-picoline, by comparison of the explosion parameters of increasing the water mixing ratio from 0 to 95 vol.% of 3-picoline under 270°C, 21 vol.% oxygen concentration, and 1 atm and their variation are displayed in Table 5.

Effects on oxygen concentrations

The flammability ranges of different 3-picoline/water mixtures all narrowed down diminishingly with the decrease of oxygen concentrations at the same pressure. Other safety-related properties summarized in Tables 3 and 4 were also on the down side. The variations of fire and explosion characteristics between different oxygen concentrations of 21 and 12 vol.% are shown in Table 6. It demonstrates the effect of ox-

Table 5 Influence of inert steam on flammability characteristics between different 3-picoline/water ratios of 100/0 and 5/95 vol.% in 21 vol.% O_2, 270°C and 1 atm

3-Picoline/water	100/0 vol.%	5/95 vol.%	Variation
Flammability range/vol.%	6.60	0.99	85.0% decreased
UFL/vol.%	8.00	2.12	73.5% decreased
P_{max}/bar	2.50	0.70	72.0% decreased
$(dP/dt)_{max}$/bar s^{-1}	181.00	6.00	96.7% decreased
K_g/mbar s^{-1}	49.13	1.63	96.7% decreased

Table 6 Influence of oxygen concentration on flammability characteristics between different oxygen concentrations of 21 and 12 vol.%

Oxygen concentration	O_2=21 vol.%	O_2=12 vol.%	Variation
Flammability range/vol.%	3.90	1.30	66.7% decreased
UFL/vol.%	5.00	2.68	46.4% decreased
P_{max}/bar	2.30	0.70	69.6% decreased
$(dP/dt)_{max}$/bar s^{-1}	130.00	2.00	98.5% decreased
K_g/mbar s^{-1}	35.29	0.54	98.5% decreased
Explosion class (St)	St-1	St-0	decreased 1 class to St-0

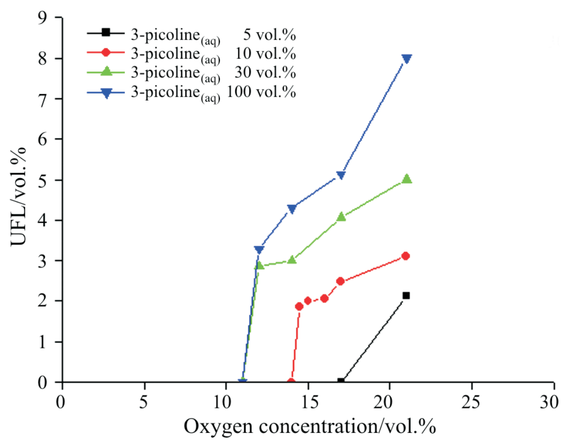

Fig. 8 UFL *vs*. oxygen concentration with four vapor mixing ratios at 270°C and 1 atm

ygen concentration on most flammability characteristics; thus, it is evident that the UFL and the degree of fire and explosions will be alleviated gradually as long as the oxygen concentration is reduced.

In Fig. 8, no matter what the concentration of 3-picoline/water was, the UFL dropped off suddenly and significantly while the oxygen concentration was dropping gradually in the test. In industrial processes, if the flammability level of the combustible or fuel can be suppressed to a safe range, their fire and explosion hazards will be correspondingly reduced to an acceptable level. Once the oxygen concentration is below the MOC, an explosion is no longer possible [17]. Therefore, we could practically dictate the oxygen concentration so as to prevent fires and explosions.

In the case of 3-picoline/water (30/70 vol.%), Table 6 shows the explosion parameters by reducing the oxygen concentration from 21 to 12 vol.% under 270°C and 1 atm and their variations.

Conclusions

Under the experimental conditions of simulated situations in a real one, we reached the following conclusions:

- As the component of inert steam increased, not only did the flammable range of 3-picoline become narrow, but the degree of explosion damage also diminish.
- As the inert steam content was more than 97 vol.% in 3-picoline/water mixture, the vapor mixture will no longer be ignited.
- This study elucidated the flammability properties of 3-picoline mixed with inert steam. The results could be applied to prevent relevant manufacturing processes from fire and explosions.

Nomenclature

K_g	gas or vapor explosion constant [mbar s^{-1}]
LFL	lower flammability limit [vol.%]
MOC	minimum oxygen concentration [vol.%]
P_{max}	maximum explosion pressure [bar]
P	initial pressure [atm]
St	explosion class, dimensionless
T	initial temperature [°C]
UFL	upper flammability limit [vol.%]
V	the volume of test apparatus [m^3; L]
$(dP/dt)_{max}$	maximum rate of explosion pressure rise [bar s^{-1}]

subscript

max maximum value of experimental property

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