Journal of Innovative Technology, Vol. 2, No. 1, pp. 41-47, March 2020
http://doi.org/10.29424/JIT.202003_2(1).0006

ACQUISITION OF EXPLOSION-PROOF PARAMETERS OF POLYVINYL ALCOHOL

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ABSTRACT

Polyvinyl alcohol (PVA), a combustible chemical, is a polymer compound through polyvinyl acetate and saponification process. It is mainly used to manufacture polyvinyl acetate, and serves as resistance to gasoline pipelines and polyvinyl alcohol fibers. With the development of modern industrial technology, a wide variety of chemical materials have been used worldwide. As a result, combustible powder explosion has been found to occur frequently. To meet with this problem, caution needs to be taken for each step of the manufacturing process of combustible powders. In this study, the 20-L apparatus, minimum ignition energy apparatus (MIEA), minimum ignition temperature for dust cloud (MIT-C), and minimum ignition temperature for dust layer (MIT-L) were used to establish dust explosion parameters measurement of the polyvinyl acetate manufacturing process. According to the 20-L apparatus explosion vessel tests, maximum dust explosion pressure rise rate (P_{max}) was 6.9 bar and maximum dust explosion pressure rise rate $(dP\ dt^{-1})_{max}$ was 137 bar s^{-1}. The K_{st} value of PVA was St^{-1}, categorized as a weak explosion. In order to prevent loss and incidental mitigation resulting from dust explosion, it is vital to fully survey the dust explosion parameters of PVA.

Keywords: Combustible powders, 20-L apparatus, Minimum ignition energy apparatus, K_{st} value, Dust explosive parameters.

1. INTRODUCTION

Polyvinyl alcohol (PVA) is a highly crystalline, odorless, translucent, nontoxic, water-soluble polymer that has high hydrophilic properties and sound film-forming. The structure of PVA, which has the idealized formula $[CH_2CH(OH)]_n$, is shown in Fig.1. It has a normal melting point of 22°C and a flash point of 78.4°C. It degrades slowly at temperatures > 100°C and decomposes rapidly at temperatures > 200°C (Eliassaf 1972; Saxena 2013). PVA was first prepared in 1924 by hydrolyzing polyvinyl acetate in ethanol with potassium hydroxide. Its physical characteristics and specific functional usefulness are highly dependent on the degree of polymerization and the degree of hydrolysis. There are two species of PVA, including partially hydrolyzed and fully hydrolyzed (Eliassaf 1972; Saxena 2013).

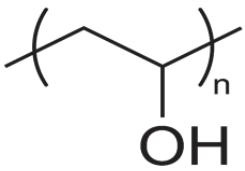

Fig. 1 Structure of PVA

PVA has been used since 1930 in a wide range of industrial, medical, and commercial applications, including resins, lacquers, surgical threads, biomass immobilization, food supplement tablets, and moisture barrier film. PVA, which is also a material for water-soluble fiber 3D printing, is used mainly for printing the support structure (El-Naas *et al*. 2013; Peng and Kong 2007).

Dust explosions have long been the most dominant source of fires and explosions. Combustible dusts or powders may explode due to crushing, grinding, conveying, classifying, and storage (Amyotte *et al*. 2009; Vijayaraghavan 2011; Firefighting precautions at facilities with combustible dust, 2013). Materials which can cause dust explosions are found to exist in a variety of industries, including natural organic materials (feed, flour, grain, linen, and starch), synthetic organic materials (furniture, pesticides, plastics, textiles, rubber, and so forth), pharmaceuticals, dyes, coal, metals (*e.g*., aluminum, chromium, iron, magnesium, zinc, etc.), additive manufacturing, 3D printing, and fossil fuel power generation (El-Naas *et al*. 2013; Peng and Kong 2007).

The Occupational Safety and Health Administration (OSHA) of the United States defines combustible dust as "a solid material composed of distinct particles or pieces," regardless of size, shape, or chemical composition, which presents a fire or deflagration hazard when suspended in air or some other oxidizing medium over a specific range of concentrations (Firefighting precautions at facilities with combustible dust, 2013; ASTM 2010; NFPA 2013; Eckhoff 2005).

Incompatibility of PVA may include various substances, such as oxidizing agents, metals, acids, and alkalis (Vijayaraghavan 2011). On the early morning of June 1, 2011, the Miaoli factory I of Chang Chun Petrochemical Corporation erupted gas explosion, followed immediately by enormous fire. The inhabitants in the neighborhood were shocked by the thundering explosion. The fire engulfed the 324-square meter factory. Fortunately, there was no report of casualty. According to the inves-

Manuscript received January 21, 2020; revised February 21, 2020; accepted February 25, 2020.
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tigation jointly conducted by the Miaoli County Bureau of environmental Conservation and the factory, the production line of PVC was to blame for the explosion. The exhaust gas recycle construction, which was under improvement, might have caused the explosion. The fine dust of PVA, which is dispersed in air in sufficient concentrations, and which is in the presence of an ignition source, is a potential dust explosion hazard. To remove and avoid thermal hazards from PVA under operation, it is necessary to determine the explosion parameters, such as minimum ignition energy (MIE), maximum pressure (P_{max}), maximum pressure rise rate ($dP\ dt^{-1}$)max, minimum ignition temperature (MIT), lower explosion limit (LEL), and deflagration index (K_{st}), thus obtaining proactively safer process design and manufacturing (Vijayaraghavan 2011; OSHA 2013; ASTM 2010; NFPA 2013; Eckhoff 2005; Eckhoff 2003; Blair 2007).

2. EXPERIMENT AND METHODS

2.1 Samples

Eighty μm PVA was purchased directly from the Chang Chun Chemical Corp. (Taiwan). PVA was stored at room temperature. It was first put it into an oven overnight at 50.0°C before conducting any tests.

2.2 Thermogravimetry (TG)

Material was burned in the air. Nickel and iron were used for temperature correction by TG (model Pyris 9.0). Sample tray was made of platinum disc. Software Pyris 9.0 was used for data analysis (Silvera 2013).

Test method: To determine combuste temperature by different heating rates for the substance, the sample mass was 3.0 ~ 6.0 mg, with a range of 30 ~ 800°C min^{-1}, and heating rates being at 1.0, 2.0, 4.0, and 8.0°C min^{-1}. These conditions were applied to understand whether, in case of cracking phenomenon due to the drying process, the powder has undergone any specific thermal behaviors.

2.3 Minimum ignition energy apparatus (MIEA)

The 1.2 L minimum ignition energy Hartmann tube tester was manufactured by Kühner in Switzerland. The body of the container is 1.2 L cylindrical glass. The powder was poured into a glass cylinder by a compressed air machine pressurized to 8.0 bar. The compressed air-driven powder, which was discharged from bottom to top, formed a cloud of dust in the wall, and then a high voltage electrode was applied to induce spark dust cloud explosion.

The experiment was implemented by adjusting the dust concentration to identify the MIE. The optional electrode ignition energy test had seven species: 1, 3, 10, 30, 100, 300, and 1,000 mJ, respectively. Ten experiments were conducted to ignite. The one which effectively ignited was judged to have ignition phenomenon (Wu *et al*. 2009; Janes *et al*. 2008; Martin 2003; Randeberg and Eckhoff 2007; ASTM 2009).

2.4 20-L apparatus

The 20-L apparatus was used to measure the P_{max}, $(dP\ dt^{-1})_{max}$, LEL, LOC, and other parameters. This apparatus conformed to the testing chamber of fire and explosion characteristics, which, according to the National Fire Protection Association (NFPA) regulation, NFPA 68 (NFPA 1993), needed to be "spherical" type and with more than 5-liter (L) volume. The 20-L apparatus was in practice easy to operate and maintain. The derived K_{st} value was fairly consistent and was as good as the 1 m^3 testing vessel, which was a substitute for the ISO standard vessel.

The testing vessel was a sphere made of stainless steel with about 20 Lin volume, and its sustainable maximum explosion pressure was 39 bara. In addition, there was, outside the sphere, a 1.5 L jacket whose primary function was to reach the experimentally set temperature through silicon oil or heating coal oil circulation. It not only maintained the experimentally required temperature condition but also instantly removed the heat produced by the deflagration or detonation. The experiment could thus be carried out smoothly (Kühner 2013).

In order to determine the maximum explosion pressure and rate of pressure rise of the explosion, the PVA density was set at 60, 125, 250, 500, 750, 1,000, 1,250, and 1,500 g m^{-3}, respectively. The ignition sources used for the 20-L apparatus were manufactured by Fr. Sobbedin, Germany (Shu and Wen 2002).

2.5 Minimum ignition temperature for dust cloud (MIT-C) and minimum ignition temperature for dust layer (MIT-L)

The minimum ignition temperature for dust cloud (MIT-C) was examined on a hot surface. It caused dust cloud to ignite and propagated flame with the lowest temperature. Approximately 0.1 g of combustible dust was placed on top of the dust retained in a temperature-controlled oven. Dust was dispersed by compressed air flowing downward past the hot surface of the furnace to explore whether there was ignition and flame generation. If the dust did not ignite, the furnace temperature was tested and repeated until the dust ignitions occurred (BS 2006; Randeberg and Eckhoff 2006).

2.6 Results and discussion

TG was employed to detect the composition of materials, which contained water, solvents, plasticizers, polymer additives, pyrolysis temperature, ash, inorganic additives, and other contents. TG could also be used to detect solid, powder, gel, and liquid material at cracking temperature, thermal stability, composition analysis. Fig. 2 shows PVA by TG analysis, which had three sections of solutions column. The column solution temperatures fell within the range of 200 ~ 450°C. PVA was placed in an oven and dried prior to the experiment, with its physical characteristics remaining unchanged. During the process, one of the important hazard elements from the environment was the sensitivity of a combustion dust-air atmosphere being readily ignited. The energy content of the spark was gradually reduced until the dust no longer ignited (Kühner 2013). Fig. 3 demonstrates PVA at different levels of mass, which was used at the instrument maximum energy 1,000 mJ ignition tests and was found to be free from detonation. The MIE of PVA was greater than 1,000 mJ, which was less susceptible to static electricity caused by the fire and explosion hazards.

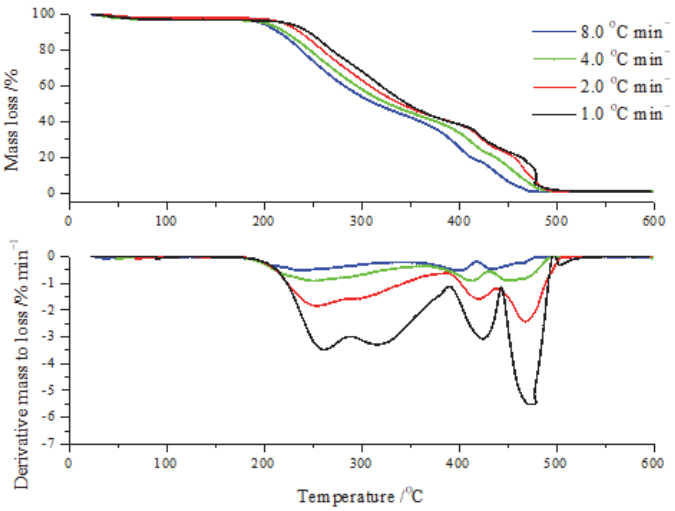

Fig. 2 Thermal analysis of PVA powder under temperature rising rate at 1.0, 2.0, 4.0, and8.0°C min^{-1} by TG

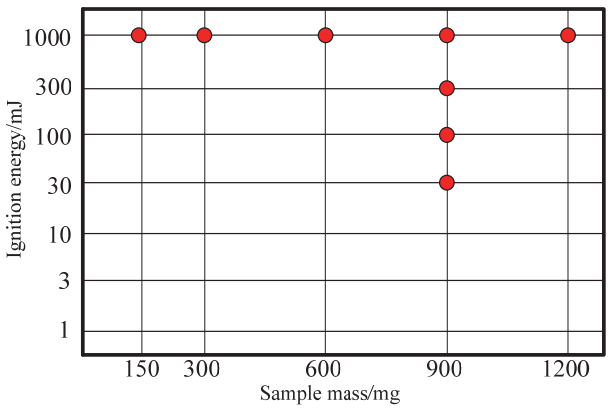

Fig. 3 Tests of minimum ignition energy for PVA at 25.0°C and 1.0 atm

One important conclusion is that conservative experimental determination of MIEA of dust clouds required the use of electric sparks with sufficiently long discharge time to prevent substantial disturbance of the dust cloud by the shock wave generated by the spark discharge (Eckhoff 2003; Blair 2007; Silvera 2013; Wu *et al*. 2009; Janes *et al*. 2008; Martin 2003; Randeberg and Eckhoff, 2007; ASTM 2009). However, it might yield false negative for dusts which were difficult to ignite with a spark but were ignitable by stronger ignition sources; even the MIE of PVA, which was greater than 1,000 mJ, did not ignite (Randeberg and Eckhoff 2006).

Two instruments, including 20-L and 1 m^3 explosion containers, were used in the test dust explosion parameters. The 20-L apparatus was established and operated mainly according to ASTM (American Society for Testing and Materials, USA) 1226 (ASTM 2010; NFPA 1993), and VDI (Verein Deutscher Ingenieure, Germany) 2263 testing criteria associated with the testing and analysis for flammable characteristics of materials. Flammability parameters of upper explosion limit (UEL), LEL, LOC, P_{max}, $(dP\ dt^{-1})_{max}$, and K_{st} (Kenneth 2000) can be obtained by the dust experiment, flammability gases, and fugitive dusts. In this study, the maximum explosion pressure, the Pmax, and the LEL were determined in a standard test apparatus with a content of 20-L (Kühner 2013; Shu and Wen 2002). The dust sample is dispersed into the explosion chamber with compressed air from a storage container via a special distribution system. Two pyrotechnic igniters were manipulated to determine the lower explosion limit by 1.0 kJ, the maximum explosion pressure, and the maximum rate of pressure rise.

Following the specification of standard operating procedures, the tests were measured at different concentrations in different sequences. The results were listed in Table 1. When the concentration was gradually increased upward, pressure generated by the blast and rising rates rose, accordingly. The data obtained from 20-L apparatus were changed into Eqs. (1) and (2). By way of statistical average, Pmax and $dP\ dt^{-1}$ maximum of PVA were 6.9 bar and 137 bar s^{-1}, respectively. Based on the above-mentioned parameters, we can see clearly from Figs. 4 and 5 the trend direction.

$$P_{max} = \frac{P_m[Ser1] + P_m[Ser2] + P_m[Ser3]}{3} \quad (1)$$

$$\left(\frac{dP}{dt}\right)_{max} = \frac{\frac{dP}{dt}[Ser1] + \frac{dP}{dt}[Ser2] + \frac{dP}{dt}[Ser3]}{3} \quad (2)$$

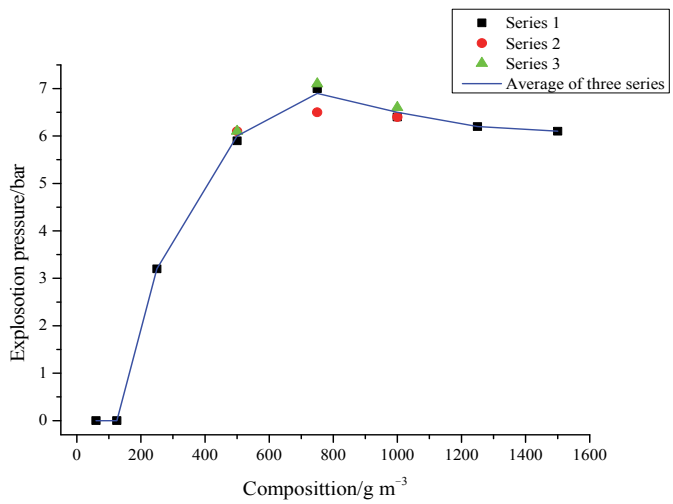

Fig. 4 Maximum dust explosion pressure rise rate for PVA at 25.0°C and 1.0 atm by 20-L apparatus measurements

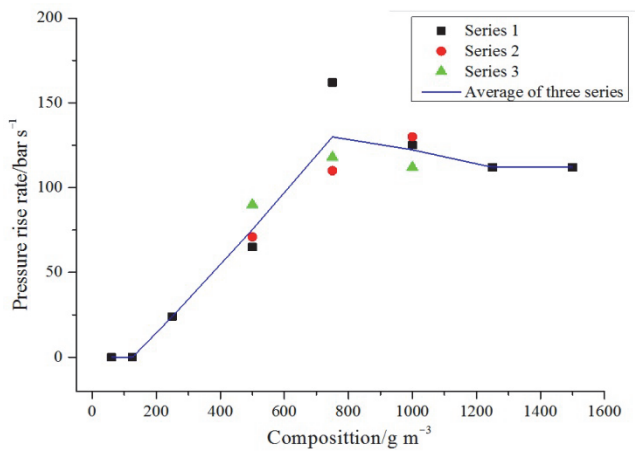

Fig. 5 Maximum dust explosion pressures for different compositions of PVA at 25.0°C and 1.0 atm

Table 1 Different concentrations on explosion pressure of PVA in series by 20-L apparatus measurements

Test	Series	PVA composition/g m^{-3}	P_m/bara	$dP\ dt^{-1}$/bara s^{-1}	t_v eff/ms
1	1	60	0	0	60
2	1	125	0	0	60
3	1	250	3.2	24	60
4	1	500	5.9	65	60
5	1	750	7	162	60
6	1	1000	6.4	125	60
7	1	1250	6.2	112	60
8	1	1500	6.1	112	60
9	2	500	6.1	71	60
10	2	750	6.5	110	61
11	2	1000	6.4	130	60
12	3	750	7.1	118	60
13	3	500	6.1	90	60
14	3	1000	6.6	112	60

t_v eff: Iginition delay time (ms)

In dust processing environments, a very serious risk is the ignition of a dust cloud within an enclosed volume. If not relieved or suppressed properly in a timely manner, the developing deflagration will result in a rapid rise of pressure and an ensuing explosion. The LOC is the highest concentration of oxygen that prevents a fugitive dust from forming an explosive atmosphere. The test was carried out in the 20-L apparatus. 750 g m^{-3} of combustible dust was placed in the dust container and the explosion chamber was filled with normal atmosphere (Siwek and Cesana 1995; Joseph 2007; Pilão 2006; Cashdollar 2000).

Once ignition was effected at a known oxygen concentration, the level of oxygen was reduced until a point at which no ignition occurred. The quantity of injected dust was adjusted to find the most explosive concentration in a reduced oxygen atmosphere (which was generally at lower dust concentrations than at normal atmospheric conditions). Subsequent tests were carried out with this mass of dust, and the oxygen concentration was gradually lessened until no ignition was found in three consecutive tests. The maximum explosion pressure at (or above) 0.2 bar was listed where an ignition was considered to have occurred. The information was summarized in Table 2. While test work is normally undertaken with nitrogen as the inert gas, it may also be carried out using carbon dioxide or argon. In actual applications, it is common to include a safety margin when applying the test results to plant design in terms of prudent loss prevention. The results, based on Table 2, were shown in Table 3, where the LOC was found to be 17 vol%. The safety margin should be in response to practical size of the plant operating and its control system for redressing oxygen increases. An oxygen monitoring sensor was installed to accurately detect oxygen concentration as well as the response time. The safety margin was unlikely to be less than 2%. The PVA dust explosion testing determined the LOC to be 17 vol%; the maximum-permitted oxygen concentration (MPOC) allowed on plant could be set at 15 vol%. To alleviate the oxygen concentration to the point where dust cloud deflagrations could not happen, a crucial strategy was used to prevent and mitigate explosions in process systems. An inert gas could be added during the procedure (*e.g.*, argon, carbon dioxide, nitrogen, or helium) to determine the lowest oxygen concentration to prevent combustion of a dust cloud from occurring.

Table 2 Tests of LOC of PVA by 20-L apparatus measurements

PVA compotation/g m^{-3}	Oxygen concentration/vol%	P_{max}/bara	$(dP\ dt^{-1})_{max}$/bara	Time/s	Temperature/°C	Relative humidity/%
750	21	6.5	110	173	24	58
	19	5.9	62	219	21	62
	17	4.8	38	325	21	60
	16	0.1	0	170	22	64
	15	0	0	62	21	60
	15	0	0	40	21	60

Table 3 Criterion for judging explosion phenomenon

IE* = 10 kJ	P_{ex}/bar	P_m/bar	Phenomenon
Explosion limits test	< 1.5 bar	< 0.2 bara	No explosive phenomenon
	≥ 1.5 bar	≥ 0.2 bara	Explosive phenomenon

*IE: Ignition energy (J)

The explosion behaviors of a combustible dust in a closed system are described by the K_{st} value and the P_{max}. The explosion indices, P_{max} and $(dP\ dt^{-1})_{max}$, are defined as the mean values of the maximum values of the three tests. As to the Kst value, it can be calculated from $(dP\ dt^{-1})_{max}$, and is shown in Eq. (3):

$$K_{st} = \left(\frac{dP}{dt}\right)_{max} \times V^{1/3} \tag{3}$$

where V is the volume of reaction container, K_{st} value is calculated by Eq. (3), the explosion class (St) can be determined according to Table 4. We calculated the K_{st} value of PVA as K_{st} = 37 barm s^{-1}. The K_{st} value of PVA is St^{-1}, classified as a weak explosion. This K_{st} value, as expressed as Eq. (4), can be used to calculate the maximum rate of pressure rise for other volumes (Randeberg and Eckhoff 2006; Kenneth 2000; Siwek and Cesana 1995; Joseph 2007; Pilão 2006).

$$K_{st} = \left[\frac{dP(t)}{dt}\right]_{max} V_0^{1/3} = 4.84\left(\frac{P_{max}}{P_0} - 1\right) P_{max} S_u \tag{4}$$

K_{st} is the size-normalized maximum rate of pressure rise. Being size-normalized, this derivation of the cubic law is based on the idealized condition, where the vessel size is large compared with either the dust flame thickness or the igniter flame volume (NFPA 1993; Kühner 2013). The K_{st} value was used in the practical design of venting systems of NFPA in 1998 (Randeberg and Eckhoff 2006; Kenneth 2000). From Eq. (4), where V_0 is the chamber volume, P_0 is the initial absolute pressure, S_u is the burning velocity. The burning velocity is the rate of flame propagation relative to the unburned gas ahead of it. The K_{st}, whose value was 0.217 m s^{-1} can be used to calculate.

The concentration of a compound in air was below LEL, thus preventing the mixture from catching fire. If the atmosphere is less than LEL, an explosion cannot occur even if there exists a source of ignition. The LEL of PVA is listed in Table 5. We can apply physical or mechanical ventilation to control explosion hazard reduction and limit the concentration of flammable gases or vapors to a maximum level of 25 vol% , which is their lower explosive or combustible limit (Cashdollar 2000; Going *et al*. 2000; Dahoe *et al*. 2001; Garcia-Agreda *et al*. 2011; Bouillarda *et al*. 2010).

A fugitive dust, once comes to contact with a heated environment, hot surfaces, electrical devices, and friction sparks, will inevitably cause a dust explosion. The MIT is an important value. When the environment temperature is below the MIT, a dust explosion can be avoided. The MIT-C determines the lowest temperature capable of igniting a dust dispersed in the form of a cloud. Table 6 demonstrates the use of different nozzle pressures and different PVA masses of dust cloud. The dust temperature of PVA can be ignited at temperatures ranging between 360 and 420°C. The UK laboratory for explosion test about minimum ignition temperature of a dust cloud postulates a maximum operating temperature in the drying process of 300°C, giving a safety margin of 150°C (UK laboratory for explosion testing, 2016). Table 6 indicates the operating environment of PVA must be 210°C or less, but in the MIT-L, neither fire nor explosion phenomenon was found (Gummer and Lunn, 2003; Polka *et al*. 2012).

Table 5 Tests of LEL of PVA by 20-L apparatus measurements

PVA concentration/gm^{-3}	Oxygen concentration/vol%	P_{max}/bara	$(dPdt^{-1})_{max}$/bara	Temperature/°C	Relative humidity/%
150	21	0	0	21	62
180	21	0.1	0	21	61
200	21	0.2	15	21	60
220	21	1.0	15	21	62
240	21	2.5	13	21	60

Table 4 Classification and description of explosion classes (St)

K_{st} value/baramsec^{-1}	Explosion class (St)	Description
0	St–0	No explosive phenomenon
1–200	St–1	Weak explosions
201–300	St–2	Strong explosions
> 300	St–3	Particularly large explosions

Table 6 PVA tests at different pressures and mass by MIT-C

Nozzle pressure/bar	0.2	0.5	0.8	1.1
Mass/g	Temperature/°C			
40	410–420	390–400	380–390	360–370
60	400–410	410–420	370–380	360–370
80	380–390	390–400	380–390	370–380
100	380–390	390–400	390–400	370–380

3. CONCLUSIONS

To prevent dust explosions, it is mandatory to understand the intrinsic safety of a material the present study considered explosion-proof parameters of PVA, maximum explosion pressure, maximum rate of pressure rise, MIE, and LEL, all of them being determined by a standard test apparatus. The PVA dust explosion test determined the LOC to be 17 vol%, and the maximum-permitted oxygen concentration (MPOC) allowed on plant might be set to 15 vol%. Operating environment of PVA must be 210 °C or less，the Kst value of PVA was St–1, which was regarded as weak explosions.

Various conditions may affect the need to reach an explosive concentration of combustible dust with a threshold amount. As a result, plant operators should make a comprehensive hazard analysis for each facility in advance. The importance of scientific and technical knowledge on dust education and personnel training system deserves emphasis, and the intrinsically safer process design should also be emphasized and implemented.

ACKNOWLEDGEMENTS

Financial support for this study was graciously provided by Chang Chun Group (CCPG) and Ministry of Education, Taiwan, ROC.

NOMENCLATURE

$dP\ dt^{-1}$	pressure rise rate (bara min^{-1})
$(dP\ dt^{-1})_{max}$	maximum rate of explosion pressure rise (bara s^{-1})
IE	ignition energy (mJ)
K_{st}	deflagration index (m bara s^{-1})
LEL	lower explosion limit (g m^{-3})
LOC	limiting oxygen concentration (%)
MIE	minimum ignition energy (J)
MIT	minimum ignition temperature (°C)
MPOC	maximum permitted oxygen concentration (vol%)
P_0	initial absolute pressure (bar)
P_{ex}	over pressure (bar)
P_{max}	maximum explosion pressure (bara)
St	explosion class (dimensionless)
S_u	burning velocity (m s^{-1})
t_v	ignition delay time (ms)
V_o	chamber volume (m^3)
UEL	upper explosion limit (g m^{-3})

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